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Magnetic transition of solid ^3He observed by polarized neutrons

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Résumé. — On décrit des mesures de transmission de neutrons polarisés sur une cible de ^3He solide faiblement polarisée. Des températures de 0,45 mK sont obtenues. Sur la courbe de fusion, ce résultat permet d'envisager de réaliser des expériences neutroniques en dessous de la température d'ordre magnétique. On estime la résistance de Kapitza.

Abstract. — Measurements of the transmission of polarized neutrons through a weakly polarized solid ^3He target are reported. Temperatures down to 0.45 mK have been reached. This permits experimentation on the melting curve below the temperature of the magnetic transition. The Kapitza resistance is estimated.

A major contribution to the understanding of the magnetic properties of solid ^3He will be the observation of magnetic reflections in a neutron diffraction experiment [1]. The first problem is to cool a ^3He solid target in the presence of a neutron beam, since the neutron absorption cross section (σ) of ^3He nuclei is high : $\sigma \sim 3\,000$ b for a wavelength of 1 Å [2].

In the same spirit as Passel and Schermer [3] who have measured the magnetization of absorbed ^3He nuclei on zeolite by studying transmission of a polarized neutron beam, we have set up an experiment with a weakly polarized solid ^3He target. Since the susceptibility has been measured previously as a function of temperature and of molar volume, these measurements give, *in situ*, the temperature of a ^3He solid target in a neutron beam.

1. **Considerations.** — The basic idea for the experiment is that the neutron capture occurs only for the singlet state built up by the nuclear spin $I = 1/2$ and $s = 1/2$ of ^3He and of the neutron. The neutron capture may occur only for antiparallel spin components of the neutron and ^3He . If, with respect to the spin component of the neutron, n^+ and n^- are the number of ^3He nuclei of a target of an area S of parallel and antiparallel spin component, the transmission of N_0 neutrons is equal to :

$$N = N_0 \exp - \frac{2 n^- \sigma}{S}.$$

[The factor 2 takes into account the fact that the absorption cross section is given for random spin orientation]. Using an absorption parameter α defined by

$$\alpha = A \frac{\sigma x}{V}$$

where A , V and x are respectively Avogadro's number, the molar volume and the thickness of the target, N can be written as a function of the ^3He magnetization M normalized to the saturation M_0

$$N = N_0 \exp - \alpha(1 - M/M_0)$$

α can be determined by simple transmission experiments at higher temperatures. The flipping ratio (R) between the normalized number of transmitted neutrons with up and down polarization is given by :

$$R = \exp 2\alpha M/M_0.$$

This quantity will lead to a magnetization measurement.

For a given target, α is proportional to V^{-1} . The measurement of the neutron transmission at high temperature gives the density of the ^3He solid. By performing experiments under well known conditions, a molar volume gauging can be performed. This has been realized using the pressure *versus* volume relation of the liquid ^3He phase [4] where the pressure of the target can be measured outside at room temperature.

By observing the change in the neutron transmission with V , the molar volume of the solid phases can be determined. In our experiment, the accuracy reaches 0.5 %. It can be increased using higher count rates.

2. Experiment. — Using a dilution refrigerator of our own design in conjunction with a copper nuclear demagnetization stage, we are able to achieve very low temperature ($T \sim 0.3$ mK). The experiment was set up on the neutron diffractometer of the Melusine reactor of CEN laboratory of Grenoble. The target is fashioned with 700 Å powder of silver sintered on two copper plates of respective thicknesses 1 mm and 0.3 mm. The target is squeezed between two aluminium pieces. The 800 Oe polarizing field is achieved by a permanent magnet (Fig. 1).

The curve of the gauging of the molar volume is shown in figure 2. Using these data, the pressure is increased in the target up to the chosen density. The volume is continuously measured during the cooling procedure which leads to the solid phase. After a precooling time of 48 hours down to 10 mK, further cooling is achieved with the demagnetization of copper down a final field of 2 kOe. A lowest temperature of 0.3 mK is reached by the copper lattice. The beam is open after a delay of 12 hours. The number of neutrons falling onto the sample is $N_0 = 1.2 \times 10^4$ n/s. For a counting time of 300 s, the uncertainty on R is 1.5 %.

3. Results. — According to the strength of the magnetic Grüneisen coefficient, $\Omega_m \sim -17$ [5], the Curie-Weiss temperature and the transition temperature decrease with the molar volume. Figure 3 reports, in arbitrary units, the susceptibility data corresponding to the two molar volume measured of 23.1 cm³ and 24.2 cm³.

Figure 4 shows the corresponding flipping ratio as a function of the time of the neutron irradiation.

In the experiment at the molar volume of 23.1 cm³, the maximum flipping ratio was observed immediately after the neutron beam was opened. Thus, this sample did not pass through the magnetic phase transition at 0.45 mK. On the other hand, for the sample at 24.2 cm³, where the phase transition occurs at 1 mK, the initial increase of the flipping ratio and thus of the suscep-

Fig. 1. — View of the low temperature experimental set up. MC, S, CD, CC, H_D , H_P and T are respectively the mixing chamber filled up with silver sintered powders, the superconducting switch, the copper demagnetization stage, the compensating coils, the demagnetization magnet, the polarizing permanent magnet and the target.

tibility shows clearly that experiments below T_N can be maintained during a time of $\Delta t = 50$ min. after the opening of the neutron beam. The factor of 2 observed between the susceptibility measured at $t = 0$ ($T < T_N$) and the susceptibility maximum ($T = T_N$) is in excellent agreement with the thermodynamic measurement of Hata *et al.* [5]. This proves that the temperature of the target is lower than T_N before the opening of the neutron beam.

The observed time variation of M via the measurement of R is related to the available magnetic entropy. The initial linear increase of R can be understood by a slow conversion of the ordered phase to the paramagnetic phase at the ordering temperature. After T_N , the slow decrease of R down to equilibrium is due to the fact that solid ^3He reaches its maximum $R \ln 2$ well above T_N [6].

The heating power $\dot{Q}_{^3\text{He}}$ produced by the neutron capture on ^3He atoms can be estimated from the results obtained on the melting curve. According to the entropy drop $\Delta S \sim 0.4 R \ln 2$

Fig. 2. — Observed check of the linearity of $\text{Log } N_0/N$ versus the inverse of the molar volume V . The full circles are the values measured for the volumes of the liquid phases. The open circle shows how, in the solid phase, the volume is deduced from the measurement of $\text{Log } N_0/N$.

Fig. 3. — Temperature variation of the susceptibility χ . In full and dotted lines respectively for $V = 24.2 \text{ cm}^3$ and 23.1 cm^3 (see reference [5]).

at T_N [6] and the measured time Δt below which the condition $T \leq T_N$ is realized, a value $\dot{Q}(^3\text{He}) = 8 \times 10^{-10} \text{ W}$ is derived through the relation :

$$\dot{Q}(^3\text{He}) = T \frac{\Delta S}{\Delta t}.$$

The energy loss $\dot{Q}$ of the charged particles (proton and triton) produced by the nuclear reaction is $\dot{Q} \sim 1.3 \times 10^{-9} \text{ W}$. Using tables of atomic stopping cross sections [7], 80 % of this energy may be absorbed in silver. The comparable values of $\dot{Q}(^3\text{He})$ and $\dot{Q}$ may show either parasitic

Fig. 4. — Time dependence of the flipping ratio measured for $V = 24.2 \text{ cm}^3$ (full circle) and for $V = 23.1 \text{ cm}^3$ (open circle). The time origin corresponds to the opening of the neutron beam.

heating coming with the beam (γ -rays, fast neutrons) or an overestimate of the role of the silver powder.

Extrapolating to infinite time, an equilibrium value of $T \sim 2 \text{ mK}$ seems to be reached. Thus, the Kapitza resistance R_K between ^3He and the silver powder can be estimated from the temperature difference δT and the heating $\dot{Q}(^3\text{He})$

$$\delta T = \frac{R_K}{S} \dot{Q}(^3\text{He}) \quad (S = 0.3 \text{ m}^2).$$

At $T \sim 2 \text{ mK}$, the derived value of $6 \times 10^5 \text{ m}^2 \text{ K/W}$ is in excellent agreement with the determination of $5 \times 10^5 \text{ m}^2 \text{ K/W}$ recently reported by Mamiya *et al.* [8] which is deduced by indirect relaxation time measurements. The concordance between the two results reinforces the interest of confirming the anomalous temperature dependence of $R_K \sim T^{0.9}$ found above T_N in reference [8]. This poses a basic question concerning the mechanism which is involved.

4. Conclusion. — The reported results prove the feasibility of studying solid ^3He by neutrons at very low temperatures, notably below T_N . Since Bragg diffraction lines have been observed in similar conditions with signal to background ratio of five and good statistics in 10 min., the probability of observing the magnetic structure is high [9].

This experiment shows clearly that solid ^3He can be cooled down below 10 mK in a high magnetic field in order to polarize neutrons.

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