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Observation of a highly polarized liquid ^3He state

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Résumé. — Nous décrivons ici une expérience qui prouve qu'à basse température il est possible de préparer un liquide d'hélium 3 avec une forte polarisation magnétique. Celle-ci est obtenue par fusion rapide d'un solide d'hélium 3, lui-même fortement polarisé. Le temps de relaxation de l'aimantation de ce liquide polarisé est identique au temps de relaxation nucléaire spin-réseau T_1 du ^3He liquide non polarisé : 150 s à 300 mK sous 22 bar.

Abstract. — We describe an experiment which indicates that at low temperature it is possible to prepare a liquid of helium 3 with a large magnetic polarization. The latter is obtained by rapid-melting of highly polarized solid helium 3. The relaxation time of the magnetization of this polarized state is essentially the same as the nuclear spin-lattice relaxation time T_1 of the non-polarized liquid ^3He : 150 s at 300 mK under 22 bar.

B. Castaing and P. Nozières have recently proposed [1] that a highly polarized ^3He liquid phase could be prepared by a rapid adiabatic melting of a highly polarized solid phase of ^3He . They have calculated several thermodynamic properties of such a transition and they have predicted, using the Fermi liquid and solid ^3He properties, that the melting curve $p = f(T)$ would be lowered significantly according to the magnetic polarization of the initial state.

Any polarized liquid thus produced, normally has only a transient existence : the non-polarized liquid state is recovered after a time T_1^* which characterizes the relaxation of the Zeeman energy ; the magnetization decreases down to a value calculated within the framework of Fermi liquid theory and given by

$$M_f \simeq \frac{3}{2} \frac{H}{T_F} \quad (1)$$

where H is the external applied field and T_F the Fermi temperature.

In fact, many relaxation processes could be effective during the melting of the solid, as well as strong relaxations in the cell where the liquid is prepared : for instance, relaxation onto the walls might have played a role and it was not clear whether this simple procedure would work or not, even if there are no objections against the principle of the experiment.

The purpose of the report given below is to give experimental evidence that such a polarized liquid helium phase exists for a time comparable to T_1 , the relaxation time of bulk liquid helium 3 in equilibrium at the same temperature ; it opens the possibility for performing new experiments to test the properties of Fermi-liquids.

1. Principle of the experiment. — The physical idea which sustains the experiment is the following : in the solid phase, the system behaves for our purposes like a Curie paramagnet. At large enough field and

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low enough temperature the polarization can reach a reasonable fraction of the magnetization at saturation :

$$M = \tanh \frac{H}{T} \quad (2)$$

where T is the temperature of the solid. This is much larger than the magnetization obtained with liquid helium 3 in the same conditions (see formula (1)), because the latter is a Fermi-liquid. The enhancement factor is of the order of T_F/T .

To prepare the liquid we have adopted the following procedure : prepare a solid at high pressure, cool the sample to low temperature, apply large external field, wait long enough to reach an equilibrium temperature and then melt the solid rapidly to get the transient polarized liquid state. By melting rapidly, we mean in a time scale short in comparison with the relaxation processes.

The behaviour of the system is observed by measuring the magnetization at each stage of the melting process.

2. Experimental conditions and procedure. —

Figure 1 shows the experimental set up. The ^3He is contained in a cell of constant volume thermally coupled to the mixing chamber of a dilution refrigerator [2]. Its inner diameter (1 cm) is equal to the

Fig. 1. — Low temperature cell : VJ : vacuum jacket ; S : shield at 0.5 K ; MC : mixing chamber ; CW : copper wires ; SM : superconducting magnet ; C : cell ; HE : heat exchanger ; F : filling capillary ; N : NMR coil ; CR : carbon resistor.

height. The heat is transferred from the cell to the mixing chamber by 100 copper wires ($\varnothing = 0.01$ cm) soldered to a heat exchanger made of sintered silver plates. The cell is placed at the centre of a superconducting magnet which gives a field $H_0 = 3.5$ T.

During an experiment (melting of the polarized solid) three parameters can be recorded :

- the ^3He temperature inside the cell is measured by a carbon resistor, the magnetoresistance of which is calibrated using the nuclear susceptibility of the solid ^3He under the same field (Curie law),

- the magnetization of the ^3He is measured by NMR.

The experiments were performed at 114.8 MHz in 3.54 T with a coherent pulsed NMR spectrometer. The transverse coil is almost in a Helmholtz arrangement ; it has a Q factor of the order of 100. We can obtain pulsed rotating magnetic fields of the order of 1 gauss with 33 dBm of electromagnetic power. This is the maximum power admissible in the spectrometer.

In order to avoid the heating of the sample by the RF pulses we observe the free decay with very small angle pulses (0.2° in solid ^3He at the lowest temperatures). The free precession is damped by the inhomogeneity of the applied magnetic field

$$(\approx 3 \times 10^{-5})$$

with a characteristic time T_2^* of the order of $40 \mu\text{s}$. The signal is displayed on a fast digital transient recorder. The signal to noise ratio is better than 256 in solid ^3He at the lowest temperatures.

- The third parameter is the pressure which is measured at room temperature with a fast response gauge as shown in the insert of figure 2.

Fig. 2. — Melting curve of pure ^3He . The full line refers to the non-polarized ^3He and the dotted line is the calculated melting curve at constant magnetization for polarized ^3He (see ref. [1]). In the insert is sketched the decompression circuit.

In order to explain the procedure, we have sketched on figure 2 the melting curve without polarization and a calculated solidification curve with polarization.

The dashed area between these two curves corresponds to a mixture of polarized solid and liquid.

For practicality, the ^3He is first compressed up to 50 bar at a temperature of 1.2 K. The solid He is cooled down to 20 mK (point A on figure 2). A plug of solid ^3He is formed in the filling capillary at a point where the temperature is 0.3 K. This procedure avoids the use of a Pómeranchuk cell and provides a homogeneous temperature in the cell. Then the magnetic field is applied and the decompression is performed by connecting at room temperature the filling capillary to a tank, the volume of which is such that the final pressure of the whole system is 22 bar. A microvalve controls the decrease of the pressure as a function of time.

When the pressure in the capillary is decreased down to 29 bar (the plug is out of the magnetic field and is governed by the usual melting curve) the pressure is released in the cell and after melting the point B is reached; it corresponds to an increase of temperature due to the reverse of the Pomeranchuk effect.

The initial conditions before a decompression are the following : Molar volume of $^3\text{He} = 23.6 \text{ cm}^3/\text{mole}$

$T_i \simeq 20 \text{ mK}$ } corresponding to a polarization of
 $H_0 \simeq 3.5 \text{ T}$ } about 15 %.

The ^3He we used contains about 200 ppm of ^4He impurities.

3. Experiments and results. — One can classify our experiments in 2 types :

— measurements under static conditions in order to establish the equilibrium properties of the system (measurements of T_1 at different pressures and temperatures);

— dynamic measurements of M , P and T during decompression.

3.1 MEASUREMENTS AT EQUILIBRIUM. — In order to define the dynamical properties of the system which are known to vary strongly according to the experimental conditions (geometry, purity of helium, coating of the cell), we have performed measurements of relaxation times in the liquid as well as in the solid, in the cell where the experiment is performed.

The relaxation time in the liquid state is measured by the following procedure : the equilibrium magnetization is first tipped to an angle of about 60° by applying a sequence of 6° pulses of $10 \mu\text{s}$ length and repetition rate of 2 per s; the recovery of the longitudinal magnetization is observed by applying successive 0.2° pulses every 20 s.

The experimental result for T_1 is given in figure 3; the temperature range extends from 50 mK to 1.2 K.

The T_1 relaxation time tends to follow a T^{-2} law in the limit of low temperature and presents a minimum of the order of 130 s at 300 mK. Its general behaviour

Fig. 3. — Relaxation times measured in equilibrium conditions in liquid : [Δ] at 22 bar and in solid at $23.6 \text{ cm}^3/\text{mole}$; [$\circ$]. Below 200 mK the relaxation times in the solid have a different meaning (see text).

is in agreement with other results [3] and reflects the temperature variation of the spin diffusion coefficient D . The magnitude T_1 is slightly reduced, probably due to relaxation of ^3He on the walls and on the copper-wires, which implies D as well.

The values of T_1 for the solid state are given in figure 3; above 200 mK they are in agreement with previous results [4]; the high T part corresponds to the contribution of vacancies to the relaxation at our frequency; around 300 mK, we observe the same plateau, with the right value, which is due to the exchange contribution. Below 200 mK, T_1 is more difficult to interpret. In fact, at these low temperatures and high fields, the magnetic specific heat of the spins is several orders of magnitude larger than the specific heat of the phonons of the solid. So that once the magnetization has been tilted as a result of a sequence of RF pulses, the lattice cannot restore equilibrium by itself; it rises quickly to the temperature of the Zeeman reservoir and is unable to relax the magnetization; on the contrary, the thermalization process takes place by the coupling with the dilution mixing chamber and it is very long (several minutes) at these temperatures; the lattice is the channel through which the energy flows between the Zeeman reservoir and the dilution mixing chamber.

3.2 DECOMPRESSION EXPERIMENTS. — The measurement of the magnetization is done by the same procedure used to observe T_1 , with pulse angles of 0.2° ; the repetition rate of the measuring pulse can be increased up to two per second.

Fig. 4. — Characteristic diagrams for a decompression. The magnetization is measured with a repetition rate of 0.6 s. Notice the two scales for the time. The initial drop of magnetization is characterized by a time constant of 1.8 s. Then the polarization of the liquid relaxes with $T_1^* = 150$ s. For a rapid decompression (Fig. 4a), the initial loss of magnetization is about 35 % while for a slow decompression (Fig. 4b) it reaches 75 %.

We have done two types of experiments; the measured quantities (M , P , T) are registered and reported in figure 4a and b. The first (4a) corresponds to a fast decompression experiment. In such conditions, the magnetization drops suddenly by 35 % at least in a time constant of $\simeq 1.8$ s and then decreases slowly with a relaxation time equal to $T_1^* \simeq 150$ s. The temperature monitored by the carbon resistor increases up to about 290 mK in a few tenths of a second.

We can observe that after the initial jump, the temperature as well as the pressure reach an equilibrium value and stay constant. This seems to indicate that the system reaches the liquid phase in the first second although the magnetization decreases slowly, since any further solid-liquid transition would imply a change of P or T values. None of these effects are observed when the liquid is decompressed at 30 mK from 29 bar to 21 bar.

On the other hand when the decompression is slow (Fig. 4b), the initial drop of the magnetization can be much larger and can even be as large as to restore the magnetization of the Fermi liquid in equilibrium at once. The slowest decompression rate

we did monitor was of the order of about 40 s down to 22.8 bar. In such conditions the initial drop was as large as 75 % and took place with a time constant of 1.8 s. As in the previous case, temperature and pressure remain constant on a long time scale.

4. Discussion of the results. — The main result of these experiments is that the existence of the highly polarized liquid is proved. After decompression the non-equilibrium magnetization is much larger than the magnetization predicted by (1); it can be observed on a time scale $T_1^* = 150$ s. But several remarks must be added :

1) First of all, the temperature T_f of the final state, about 300 mK in our experiments, corresponds to the minimum of the melting curve. Of course T_f is dependent on the initial conditions : not only of P_i and T_i but also of M_i .

In our case the polarization is 15 % but the decrease of entropy due to the magnetic field is only 3 %. Apparently, T_f is not dependent on the speed of the decompression; the sudden jump of the temperature as indicated by the resistor, which happens just after the beginning of the decompression, may be understood as a heating of the phonons reservoir of the solid ^3He by the first droplet of liquid produced at the final temperature during the melting; the specific heat of the phonon reservoir being smaller by orders of magnitude than that of the liquid, it is heated up immediately.

2) Once the equilibrium pressure is established, the magnetization decreases with a relaxation time T_1^* which is the T_1 measured at the same temperature (see curve 3).

The relaxation of the polarization on a long time scale occurs with a single time constant T_1^* . This indicates that a unique relaxation process describes the phenomenon and since T_1^* is equal to T_1 in the liquid, this supports the idea of a direct transfer of polarization during decompression from the solid at T_i to the liquid at T_f .

3) The initial drop of magnetization is the most unexpected observation because it can be as large as 75 % in a slow decompression. A first contribution of the order of 14 % to this drop is simple to explain : when we pass from the highly compressed solid to the liquid at 21 bar, going from $23.6 \text{ cm}^3/\text{mole}$ to $27.3 \text{ cm}^3/\text{mole}$ there is a flow of liquid helium going out of the cell.

But how can we explain 50 % or 75 % ? We are then led to admit that the initial decrease of the magnetization with a time constant of 1.8 s is due to a new relaxation process which takes place at the interface between the solid and the liquid. The longer the melting takes, the larger is the effect of this process. But it is not clear from our experiment whether that relaxation process is due to the movement of the melting front, including for instance a fluctuating

local field, or if it is only due to the coexistence of solid and liquid helium. This is clearly a question to be faced directly.

On the other hand, the existence of polarized liquid phase being proved [5], it remains necessary to increase still further the polarization of the initial solid; it would not be difficult in the near future to start from 8 T, 5 mK; the polarization will be as high as 80 % and many of the effects predicted by

Castaing and Nozières [1] will be enhanced; in particular the melting curve $p(T)$ could be shifted significantly in comparison with the non-polarized case. Many of these effects depend on M^2 and were difficult to detect in our first experiment.

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