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NUCLEAR DEMAGNETIZATION OF PrS AND PrNi₅ (*)

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Résumé. — Nous avons désaimanté les composés PrS et PrNi₅ dont le paramagnétisme de Van Vleck est augmenté par les couplages hyperfins. Des températures finales de 0,72 mK (PrS) et de 0,55 mK (PrNi₅) ont été mesurées au moyen d'un thermomètre à fil de platine NMR. On a mesuré l'entropie nucléaire de PrS dans un champ de 75 mT. On s'attend à des températures finales plus basses pour PrS avec un contact thermique amélioré.

Abstract. — We have demagnetized the hyperfine enhanced Van Vleck paramagnets PrS and PrNi₅. End temperatures of 0.72 mK (PrS), and 0.55 mK (PrNi₅) were measured by a Pt-wire NMR thermometer. The nuclear entropy of PrS in a field of 75 mT has been measured. With improved thermal contact lower end temperatures are expected for PrS.

In the process of testing various hyperfine enhanced Van Vleck paramagnets as nuclear refrigerants for our double-stage nuclear demagnetization apparatus [1], we have investigated PrS and PrNi₅. The latter had already been successfully used for nuclear cooling by Andres and coworkers [2], who have obtained end temperatures at around 0.8 mK, starting from 17 mK and 2.3 T. PrNi₅ has a hyperfine enhancement factor of 14.3 and an anticipated electron-nuclear ordering temperature of 0.57 mK [2]. No ultralow temperature data have been reported for PrS. From magnetic and specific heat measurements at $T > 1.3$ K, an enhancement factor of 5.8 has been determined and a cooperative nuclear ordering at a temperature below 0.3 mK has been predicted [3].

For our experiments, 25.7 g of PrNi₅, and 18.1 g of PrS have been used. The PrNi₅ sample has been prepared as a rod of 0.65 cm diameter by arc melting at Ames Laboratory [4]. Its resistance ratio was 28. For the production of PrS samples, we pre-reacted the appropriate ratio of Pr-metal (from Ames Laboratory) and pure sulphur in a quartz ampoule while heating to 950 °C. The inhomogeneous reaction product was filled under argon atmosphere into a tungsten ampoule which was sealed by electron beam welding. Within 10 minutes the ampoule was brought to 2 440 °C, the melting point of PrS. The melt was overheated

for 1 hour at 2 550 °C, cooled in a temperature gradient within 3 hours to 2 300 °C, and then faster to room temperature. The polycrystalline sample of 1.2 cm diameter had grains up to 0.1 cm³ within the first frozen parts. The PrS exhibits a dark golden colour, is single phase and very brittle. Stoichiometric and metal rich samples do not adhere to the tungsten walls, whereas sulphur rich samples are not removable from the crucible without cracking.

For the demagnetization experiments, the samples have been soldered to 50 strands of 0.03 cm diam. Cu wires linking them to our Pt-wire NMR thermometer [5] and *via* a superconducting heat switch [6] to the pre-cooling mixing chamber. Whereas soft soldering to PrNi₅ is no problem, making thermal contact to PrS proved to be a substantial obstacle. Eventually, PrNi₅ turned out to be a suitable solder for PrS. We soldered the PrS by induction heating in vacuum. The sample itself was used as a crucible by spark cutting a central hole into it. After melting inside this hole, PrNi₅ makes good contact to PrS. Then a second hole was cut into the PrNi₅ (remaining amount of 0.79 g PrNi₅) and to this material we soldered the copper wires.

Starting conditions for the demagnetizations were $H_i = 8$ T, and T_i between 17 mK and 36 mK. For both compounds a linear relation between the final temperature T_f and final fields H_f was observed down to about 3 mK. We consider this as a support of the reliable performance of our Pt thermometer, and of

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the minimal entropy losses during demagnetizations down to 3 mK. Deviations from linearity between H_f and T_f are only expected at lower temperatures due to the onset magnetic ordering below 1 mK [2, 3]. The final temperature observed with PrNi_5 in a field of 0.015 T was 0.55 mK (+ 5 %). This temperature is lower than the minimum temperature of 0.8 mK previously observed for PrNi_5 , but is consistent with a calculated ordering temperature of 0.57 mK [2]. The observed relaxation times of the Pt-Cu- PrNi_5 system were less than 1 min. at $T > 2$ mK; they increased to 5 min. at 1 mK, and to 30 min. at our lowest temperatures; this relaxation results from the low thermal conductivity of PrNi_5 and its high specific heat.

Taking data on PrS was strongly hindered by much longer relaxation times; they increased to 8 h. around 1 mK. We measured the effective thermal coupling of the Cu-wires to the PrS and found a conductance proportional to T , being 2 nW/mK at 3 mK. As the heatleak into the set-up is measured to be 0.5 ± 0.1 nW, the minimum temperature at the Pt-thermometer is expected to be 0.75 mK, if most of the heatleak is absorbed along the thermal path with the mentioned conductance, even if the PrS itself is much colder. Hence the entropy measurements (see Fig. 1) have considerable uncertainty. But we can conclude, that

FIG. 1. — Nuclear entropy of PrS. The curve at 8 T is calculated, the curve at 75 mT measured. Shaded area denotes uncertainty, due to the bad thermal coupling and long internal relaxation. The dotted line shows path during demagnetization. With improved thermal contact minimum temperatures below 0.5 mK are expected.

the nuclear ordering temperature certainly is below 0.8 mK, which makes the material interesting as a refrigerant, if the contact problem can be solved and if thin slices provide short thermal path lengths.

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