Journal of Physics: Conference Series

OPEN ACCESS

The Vienna Nuclear Demagnetization Refrigerator

The Vienna Nuclear Demagnetization Refrigerator

D.H. Nguyen,[*] A. Sidorenko, M. Müller, and S. Paschen

Institute of Solid State Physics, Vienna University of Technology, Wiedner Hauptstr. 8 - 10, 1040 Vienna, Austria

A. Waard and G. Frossati
Leiden Cryogenic BV., Kenauweg 11, 2331 BA Leiden, The Netherlands

[*]E-mail: duyha.nguyen@ifp.tuwien.ac.at

Abstract. A new nuclear demagnetization system coupled to a powerful dilution refrigerator and a vector magnet was successfully built and operated. Our aim was to construct a versatile, modular cryostat, with a large experimental space providing an excellent platform for various types of ultralow temperature measurements. A powerful dilution unit allows us to cool the mixing chamber down to 3 mK and to precool a massive copper (~90 mol) nuclear stage in a field of 9 T to 8 mK in 100 h. After demagnetization the lowest temperature of the copper stage measured by a Pt thermometer was 50.9 µK in a field of 20 mT. The cryostat is integrated with a 8 T - 4 T vector magnet system. The refrigerator is provided with a 50 mm central clear shot tube allowing the insertion of a top-loading probe to cool down samples for measurements inside the vector magnet bore in a reasonably short time of about 4 hours. The system will be used to study quantum critical behavior of heavy fermion compounds.

1. Introduction

At present the method of choice for producing temperatures in the microkelvin regime is a combination of two basic ideas: (1) adiabatic demagnetization cooling first shown by Kurti[1,2] and (2) the dilution of ^{3}He in ^{4}He suggested by London[3] and in another version improved by London et *al*.[4]. A major step forward in the development of the nuclear cooling technique was the successful use of a dilution refrigerator and a superconducting magnet to obtain improved starting conditions in the early 1970s. Some of the major achievements in dilution refrigeration and nuclear cooling can be found in the literature.[5-12]

In this paper, we describe the construction and performance of a new nuclear demagnetization refrigerator which was designed for experimental work on condensed matter at microkelvin temperatures.

2. Dilution refrigerator and magnet

The dilution refrigerator used in the described cryostat is a model DRS 1000 (figure 1A) that was designed and manufactured by Leiden Cryogenics BV. Technical details about the design and construction of the 1 K pot, the still, the continuous counter flow heat exchangers (HE), and the mixing chamber (MC) can be found in the literature.[5,6,11,12] This system has very large cooling power: $Q_{120\ \mathrm{mK}}$ = 1300 µW, $Q_{30\ \mathrm{mK}}$ = 100 µW, and $Q_{10\ \mathrm{mK}}$ = 10 µW at the circulation rate of 1.5 mmole/s. Without the copper nuclear stage we reached a base temperature of 3.15 mK.

Journal of Physics: Conference Series **400** (2012) 052024 doi:10.1088/1742-6596/400/5/052024

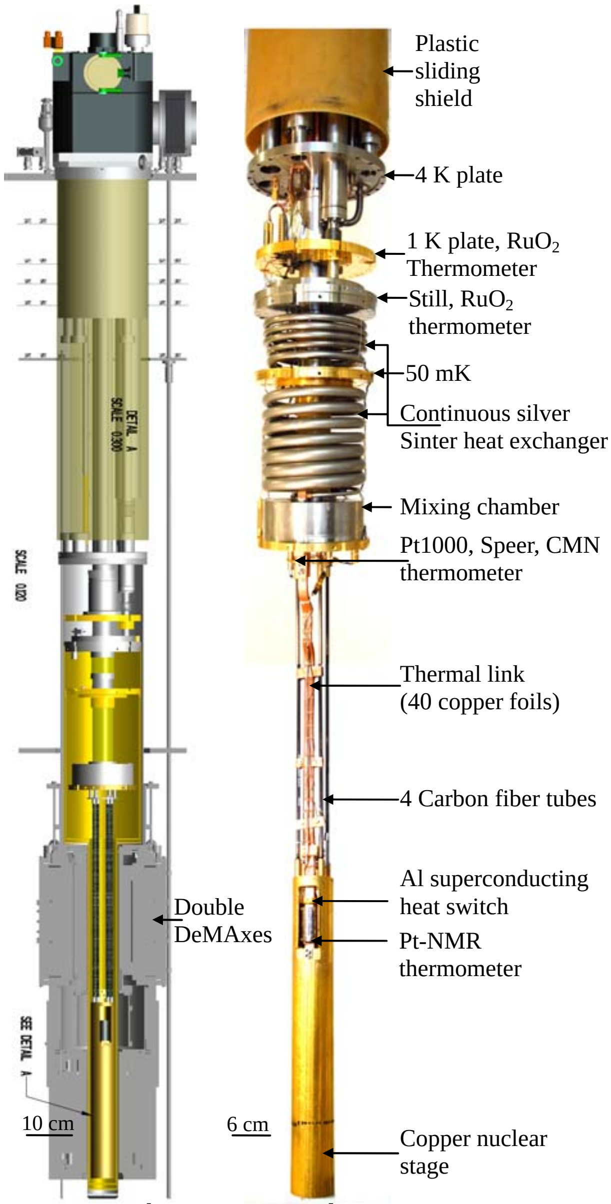

Figure 1. The Vienna Nuclear Demagnetization Refrigerator. A - Schematic vertical cross-section. B - Photograph (from 4 K plate on).

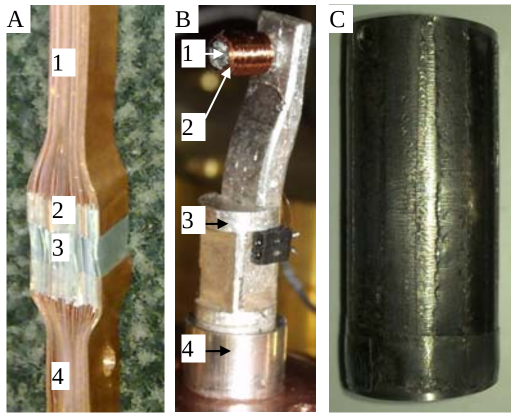

Figure 2. A - Photograph of the heat link and the Al superconducting heat switch made up of (1) 40 copper foils connected to the mixing chamber, (2) diffusion welding contacts, (3) 40 Al foils, (4) 40 Cu foils connect to the nuclear stage. B - Photograph of the heart of Pt-NMR thermometer made up of (1) Pt brush, (2) Cu pick-up coil, (3) silver foot, (4) M5 direct mounting thread to the nuclear stage. C - Multilayer Nb/NbTi/Nb high magnetic field shield.

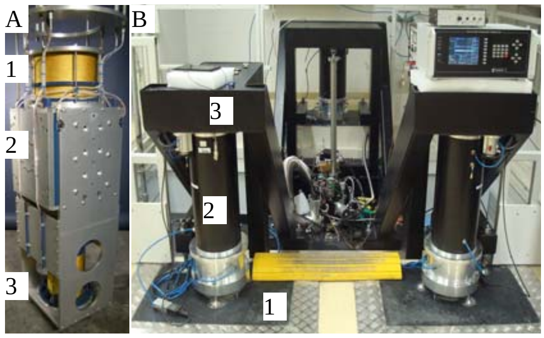

Figure 3. A - Photograph of the Double-DeMAxes™ MX-2 magnet system consisting of (1) upper compensation coil, (2) 2D sample vector magnet (combination of 8 T + 4 T magnet), (3) 9 T demagnetization magnet. B - Photograph of the active electro-pneumatic vibration isolation system consists of (1) passive damping pads, (2) air spring pistons, (3) 2 tons platform.

The gas handling system consists of: (1) control panel, (2) controller for turbomolecular pumps, (3) MaxiGauge vacuum gauge controller, (4) triple current source, (5) pumping system, and (6) 600 liter tank used for storing ^{4}He-rich mixture. Most of the valves of the control panel are integrated into an aluminum block- making the system very compact and reliable. They are furnished in a cabinet made of hollow square stainless steel tubes welded to make a leak-tight reservoir for storing the 176 liters of ^{3}He.

For measuring the temperatures at various places of the cryostat we use two different kinds of thermometry: (1) resistance thermometry (Pt resistance thermometers Pt-1000, RuO_2 thermometers, and sliced Speer carbon resistance thermometers) monitors temperatures above 10 mK and (2) magnetic thermometry (CMN, Pt-NMR thermometers) is used for temperature below 20 mK.

Journal of Physics: Conference Series **400** (2012) 052024 doi:10.1088/1742-6596/400/5/052024

Our superconducting magnet system (Double-DeMAxes, figure 3A) was manufactured by American Magnetics Inc. It consists of 3 superconducting magnets immersed in a helium bath of 150 liters. The system with the magnets at maximum field in persistent mode can work continuously during 4 days until the next transfer of liquid ^{4}He. The vertical field magnet for nuclear demagnetization has a bore of 78 mm and a maximum field at 4.2 K of 9 T, with a homogeneity better than 0.1% over 1 cm diameter spherical volume (DSV). The upper part of the coil has compensation windings which reduce the field to less than 100 G. The second vertical magnet is a solenoid coil with clear bore of 78 mm that generates a maximum field of 8 T at the sample position. The third magnet is a split-coil magnet that generates a maximum field of 4 T perpendicular to the solenoid fields. Both latter magnets have a field homogeneity about 0.5 % over 1 cm (DSV). This magnet system produces a rated field vector of 4 T magnitude in 2D space.

3. Nuclear stage

The nuclear stage is made of a high purity copper rod (4N electrolytic Cu, NOSV from Aurubis, Germany) of 60 mm diameter and 450 mm length with 36 spark-cut radial slits of 0.2 mm width. The total mass of the copper nuclear stage is 10 kg; the effective amount in 9 T field is 90 moles. The copper nuclear stage was annealed in low pressure oxygen for 100 hours at 960°C. The residual resistance ratio reached about 1000. In order to protect the copper nuclear stage against oxidation and to permanently ensure small thermal resistance of the contact surfaces, the entire stage was plated with a high purity gold layer. The nuclear stage was rigidly mounted to the mixing chamber plate by four thermally insulating carbon fiber tubes of 8 mm diameter, 2 mm wall thickness, and 500 mm length (figure 1B). The stage is protected against thermal radiation by gold plated copper shields, fixed at 50 mK and at the still.

4. Thermal link and heat switch

A thermal link (figure 2A) between the mixing chamber plate and the copper nuclear stage consist of 80 copper foils (0.2 mm thickness, 13 mm width, 40 foils of 580 mm length and 40 foils of 70 mm length, 4N electrolytic copper NOSV from Aurubis, Germany), and 40 aluminum foils (0.2 mm thickness, 13 mm width, 30 mm length, 6N Specpure of Johnson Matthey). The Cu and Al foils were pressed using a stainless steel jig with molybdenum bolts and heated under high vacuum (10^{-6} mbar) for 30 minutes at 550°C and for 12 hours at 450°C. The switching field coil was made of 11868 turns of 67 µm diameter NbTi multifilament superconducting wire in Cu matrix on a brass holder, giving a field to current ratio of 3925 G/A. The coil was shielded from fringe fields with a multilayer Nb/NbTi/Nb cylinder of 50 mm length. The current required to switch Al to normal state is enhanced from 27 mA to 72 mA. At 6.45 mK a switching resistance ratio of 10^6 was reached.

5. Pt-NMR thermometer

The heart of our thermometer consists of 4000 5N platinum wires of 20 µm diameter and 8 mm length and an NMR excitation coil being made of 1348 turns of 15 µm diameter copper wire. The bundle of Pt wires of 2.7 mm diameter was pulled through a 4N silver holder and welded to spherical cap. By this, the thermal contact among the Pt wires and the silver foot is established. The bundle of Pt wires and silver foot was annealed at 800°C for 12 h in air to make stress free. This NMR pick-up coil with an inner diameter of 3 mm was placed over the platinum brush and fixed to its foot with a tiny amount of super-glue. The two leads for the coil were twisted together and glued onto the silver foot at several places for thermal anchoring (Fig. 2B). For making the field coil, a brass holder was wound with several Nb/Kapton turns filled stycast 1266 to improve the homogeneity of the magnetic field inside the coil. Then 12696 turns of 67 µm diameter NbTi multifilament superconducting wire in Cu matrix was wound on top. These coils were shielded from fringe fields with a multilayer Nb/NbTi/Nb cylinder of 50 mm length (figure 2C).

6. Vibration isolation

Figure 4. A table showing (left) the lowest temperature measured so far in the Vienna Nuclear Demagnetization Refrigerator by a Pt-NMR thermometer at 158 kHz and (right) the free induction decay signal at 50.9 µK.

Isolation from vibrations is very important because they have two very serious effects: (1) the mechanical response of hysteretic heating as solid part of the devices flex and (2) Joule heating from eddy currents induced as metallic parts move in a magnetic field or vice versa. The latter effect can be particularly serious during precooling, when the nuclear stage is exposed to high magnetic fields of several T, or during transfer of liquid ^{4}He. Our cryostat was hung into the 2 tons platform of an active electro-pneumatic vibration isolation system (Figure 3B) with 3 degrees of freedom with the main advantages of yielding: (1) highly-effective isolation of vibration without resonance amplification, (2) optimal positional precision in both the vertical and horizontal direction, (3) minimal deflection and subsidence times with changing load, (4) highly-effective real-time control. The vibrations of the main pumping line were isolated by double T-pieces and wall-mounting. With this operation, the base temperature of the nuclear stage at 9 T before demagnetization was reduced from 30 mK to 6.45 mK.

7. Performance

In the first run, without the copper nuclear stage mounted, the dilution refrigerator reached the lowest temperature of 3.15 mK. During the second run with the copper nuclear stage mounted but without a magnetic field applied, the base temperature of the nuclear stage was 6.45 mK (measured by the CMN thermometer). Upon magnetization, the temperature of the nuclear stage increased to around 60 mK (depending on the ramp rate of the magnetic field). In general, it takes 24 (100) hours to precool the copper nuclear stage to 15 (8) mK. One of the successful demagnetization processes started with the initial conditions B_i = 9 T and T_i = 6.45 mK and was done as follows. The field was linearly decreased in time using the sequential ramps at 1 T/h from 9 T to 4.5 T, at 0.5 T/h from 4.5 T to 2.25 T, at 0.25 T/h from 2.25 T to 1.125 T, and at 0.1 T/h from 2.25 T to 20 mT. The lowest temperature measured so far by the Pt-NMR thermometer at a frequency of 158 kHz, located on the top of the nuclear stage, was 50.9 µK (figure 4) in a field of 52 mT on 27 March 2011. At this point, the temperature did not decrease further even though the magnetic field was still decreasing. This may be due to an insufficient thermal contact between the silver foot and the copper nuclear stage at temperatures below that point. We decided to keep the field at 50 mT until the temperature of the nuclear stage warmed up to 300 µK.

8. Conclusions and improvements

A new nuclear demagnetization refrigerator was successfully constructed and installed at the Institute of Solid State Physics at Vienna University of Technology – as the heart of the newly founded Vienna Microkelvin Laboratory. In spite of harsh vibration conditions (the laboratory is situated in the centre of Vienna in the 5th to 7th floor) excellent performance (the lowest temperature measured by a Pt-NMR thermometer was 50.9 µK, the system was kept below 100 µK for several hours) could be demonstrated within only two months after delivery of the system.

To optimize the system for the demanding planned experiments, further improvements are planned: (1) better damping of the vibrations of the pumping lines, (2) miniaturization and magnetic shielding of the heat link and the heat switch, (3) improvement of the thermal contact between the silver foot of the Pt-NMR thermometer and the nuclear stage, and of the pick-up coil and the nuclear stage, (4) testing and improving the thermal contact of the top-loading probe, (5) electromagnetic shielding.

9. Acknowledgements

Financial support from the European Research Council under the European Community's Seventh Framework Program (FP7/2007-2013)/ERC grant agreement no. 227378 and from Vienna University of Technology is gratefully acknowledged.

Journal of Physics: Conference Series **400** (2012) 052024 doi:10.1088/1742-6596/400/5/052024

References

[1] N. Kurti, F. Simon, Proc. Roy. Soc. A 149 (1935) 152.
[2] N. Kurti, F. N. Robinson, F. E. Simon, and D. A. Spohr, Nature 178 (1956) 450.
[3] H. London, Proc. Int. Conf. Low Temp. Phys., Oxford (1951).
[4] H. London, G. R. Clarke, and E. Mendonza, Phys. Rev. 128 (1962) 1992.
[5] G. Frossati, H. Godfrin, B. Hebral, G. Schumacher and D. Thouluoze, Proc. Hakone Int. Symp. The Physical Society of Japan (1977) 205.
[6] G. Frossati, J. Phys 39 (1978) C6-1578.
[7] P. M. Berglund, G. J. Ehnholm, R. G. Gylling, O. V. Lounasmaa, and P. R. Sovik, Cryogenics 12 (1972) 297.
[8] O. V. Lounasmaa, *Experimental Principles and Methods Below 1 K*, Academic Press (1974).
[9] W. J. Huiskamp and O. V. Lounasmaa, Rep. Prog. Phys. 36 (1973) 423.
[10] Leiden Cryogenics BV, Kenauweg 11, 2331 BA Leiden, the Netherlands.
[11] P. G. Van de Haar, PhD thesis, Leiden University (1991).
[12] G. A. Vermeulen and G. Frossati, Cryogenics 27 (1987) 139.