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Pulse-Tube Dilution Refrigeration Below 10 mK

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Abstract We report the design, realization and performance of dilution refrigerators using a pulse-tube cooler as a first cryogenic stage. The absence of a Dewar containing cryogenic fluids makes this new type of refrigerators particularly versatile. The system provides relatively high cooling power, and reaches temperatures well below 10 mK.

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1 Introduction

The need of very low temperature studies, well below 1 K, is steadily increasing, both for fundamental and applied research. Present studies cover quantum fluids, electronic and thermal properties of solids (including nanosciences), metrology, highly sensitive instrumentation like bolometers for astrophysics, near-field microscopy, low noise electronics, etc. The best method to reach millikelvin temperatures available today is the dilution refrigerator (DR). Many modern DRs are based on the Grenoble design incorporating sintered silver heat exchangers [1–4], with a large flow rate (a few mmol/s) handled by pumping system using a 60 m^3/h rotary pump and a 500 m^3/h Roots pump. These systems are nowadays a "standard" of conventional dilution refrigerators, using a liquid helium bath.

In recent years pulse-tube (PT) coolers able to produce temperatures of a few kelvins with nearly one watt of cooling power have become available. This motivated

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us to undertake the development of pulse-tube pre-cooled dilution refrigerators (PT-DR), in the framework of a scientific-industrial collaboration. We describe here for the first time the results obtained on two models (PT-DR1 and PT-DR2), developed since 2000, and commercialized since 2001. An excellent work on this type of refrigerators has been performed independently by Uhlig [5, 6]. Note also the pioneering work of Koike et al. [7] on a hybrid refrigerator, P-T and Gifford–McMahon pre-cooled. There exist similarities between these machines, particularly in the general aspect, but also substantial differences in the design. Our objective was to develop a high power (a fraction of a mmol/s flow rate), very low temperature ($T < 10$ mK) refrigerator. We describe in the following the design and the results obtained with this new type of refrigerators.

2 Pulse-Tube Refrigerator

The pulse-tube refrigerators used in the systems described in the present work are commercial units, manufactured by Cryomech.[1] The model PT405 provides at its first stage a cooling power of 25 W at 55 K, and 0.3 W at 3.5 K at its second stage. Contrarily to other machines, like the Gifford–McMahon cryocoolers where the role of strong vibrations was evidenced in pioneering works [8], PT-refrigerators are rather quiet devices.

3 Design of the High Temperature Stage

The general design of the high temperature ($T > 3$ K) part of our cryostat is relatively straightforward. As seen on Fig. 1, it consists of a room temperature flange ($D = 37$ cm), and the body of the cryostat, a cylindrical vessel ($D = 33$ cm, $L = 105$ cm). The total height of the PT-DRs is of the order of 1.3 m, including the PT head. The two stages of the PT refrigerator are each connected to a flange of large diameter, which constitute the main cold stages of the dilution unit above 3 K. They also serve as mechanical supports for the 60 K radiation shield and the 4 K vacuum can, which acts also as a radiation shield.

The absence of cryogenic fluids, as well as the lack of a large gas enthalpy, make thermal grounding a crucial aspect in PT-DR design, and experience acquired in usual systems can be misleading. In the machines described here, designed for large experimental volumes, there is a relatively high radiation power on the heat shields. Thermal gradients must be kept to reasonable values, thus leading to relatively thick mechanical parts (flanges, shields). Since this results in long pre-cooling times, an optimization work is needed in the design. The thermal connection of the PT stages to the cryostat flanges is classical. In order to reduce transmission of vibrations from PT to the DR unit, we used copper braid welded to solid copper parts, which are bolted on the PT stages and to the DR flanges. A similar solution has been used by Uhlig [5, 6]. In our first model we adopted a conservative solution, long copper braid straps

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[1] Cryomech Inc., Syracuse, New York 13211, USA.

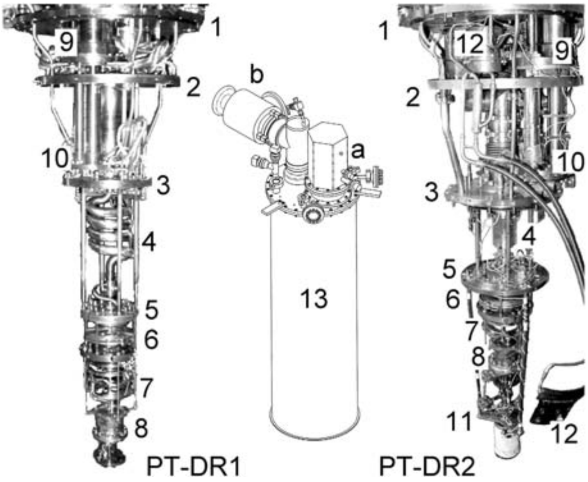

Fig. 1 *1*: 300 K flange, *2*: 60 K flange, *3*: 4 K flange, *4*: vapor (700 mK/4 K) exchanger, *5*: still, *6*: continuous exchanger, *7*: step exchangers, *8*: mixing chamber, *9*: PT1, *10*: PT2, *11*: nuclear demagnetization, *12*: N2 thermal pipe, *13*: global view of PT (*a*) and pumping line (*b*)

were used at the 60 K and 4 K stages. For PT-DR2, we used very short straps without noticeable increase in the vibration nuisances. The 60 K shields in both DRs were made out of copper ($D = 28$ cm, $L = 97$ cm, $t = 1.2$ mm, $m = 10.7$ kg). In PT-DR1 the shield is cooled through the 60 K flange, and cooled directly by the PT thanks to short braids in PT-DR2. We also tested, for PT-DR2, a 1 mm thick aluminum shield. It has a smaller mass, only 3 kg. This design corresponds to the needs of neutron scattering experiments. A nitrogen thermal pipe was used with this shield in order to delocalize the PT cold source to the middle of the shield. It also divides thermal gradients by two, allowing the use of thin aluminum shields with performances similar to those of copper ones. The vacuum can has a dual function, since it also serves as a 4 K thermal shield. Again, the absence of a helium bath generates some interesting cryogenic challenges. Thermal gradients at the 4 K flange must remain small during all the operation procedure of the DR (pre-cooling, condensation and circulation), in order to preserve the efficiency of the machine. For PT-DR1 we used a copper can ($D = 16$ cm, $L = 75$ cm, $t = 2$ mm, $m = 6.8$ kg). The PT-DR2 vacuum can was made out of stainless steel ($D = 20$ cm, $L = 75$ cm, $t = 0.8$ mm, $m = 3.5$ kg). The thermal conduction was ensured by an independent path, through several massive copper plates (total mass 5.2 kg). With 15 layers of super-insulation on both shields, we measured for PT-DR1 a total heat load of 17 W on the first PT stage at 50.2 K; the steel flange was at 70 K. On the second stage we find 0.3 W at 3.6 K; the steel flange was at 4.6 K. For PT-DR2, the total heat load was 22 W on the first PT stage at 58 K; the nitrogen thermal pipe was at 65 K and the shield below 85 K; on the second stage we find 0.25 W at 3.5 K; the steel flange was at 4.3 K and the shield below 6 K.

4 The ^{3}He–^{4}He Dilution Stage

The objective of the work described in this article is to build PT-DRs of high power at very low temperatures, with performance comparable to that of the refrigerators using a liquid helium bath developed in Grenoble and commercialized as “Maxidil” by L’Air Liquide.

The PT-DR, however, requires a careful design of the condensation line, both for its thermal grounding as for the choice of the flow impedances that determine where the heat is transferred. It is essential to “protect” the 4 K stage of the PT, since the efficiency of the incoming ^{3}He pre-cooling will have a strong impact on the DR performance. In the systems described here, the ^{3}He is pressurized up to a few bars in order to reduce its enthalpy at Kelvin temperatures [9]. Different configurations, in addition to the obvious cold plates thermal grounds, have been tested in order to thermally anchor the incoming ^{3}He capillary.

Between the 4 K and 0.7 K stages, the ^{3}He incoming capillary passes through the still pumping line, reaching the main flow impedance. We investigated several versions of this Joule-Thomson exchanger, changing the external geometry (see Fig. 1) and the heat exchanger inside the pumping line to optimize its efficiency.

The impedances along the condensation path were determined using in-situ pressure measurements at several intake points, adapting them to the results of thermodynamics calculations. Typical values for the main impedance are on the order of 10^{12} cm^{-3}, close to the values used by Uhlig [5, 6].

The lower part of the dilution unit (mixing chamber, still, heat exchangers, shields) is a standard Maxidil unit. The mixing chamber, made out of copper, is of large volume. It contains a sintered silver powder heat exchanger of 80 m^2 surface area. The still has a large diameter, and thus a substantial volume. This, together with the mixing chamber big size, makes the refrigerator insensitive to the large pressure variations at the ^{3}He compressor (inlet of the DR), thus allowing a large flow-rate range. A first continuous counter-flow exchanger is used. In the case of PT-DR1, four sintered silver heat exchangers were used, and for PT-DR2, only two. The dilution unit has a copper thermal shield, anchored onto the still (PT-DR1: $D = 12.4$ cm, $L = 50$ cm, $t = 1$ mm; PT-DR2: $D = 18$ cm, $L = 50$ cm, $t = 0.8$ mm).

The mixing chamber temperature was measured by Speer 100 Ω calibrated carbon resistors and a CMN thermometer. 68 Ω and 47 Ω Allen-Bradley carbon resistors were used between 1 K and 30 K, and 100 Ω; platinum resistors were used at higher temperatures.

5 Performance of the PT-DRs

The first aspect of the performance of the PT-DR to be considered is the pre-cooling time. For both models used here, in spite of the relative large size and mass of the units, about 16 hours for PT-DR1 and 12 hours for PT-DR2 were needed to cool the system to the desired temperature, as seen in Fig. 2 for PT-DR2. This compares well with the 12 hours quoted by Uhlig [5, 6].

The mixture condensation time can be rather long for large dilution units. PT-DR2 reached a flow rate of 220 μmol/s which corresponds to a condensation time of 5 hours for this two step-exchangers dilution.

The PT-DR1, with a 600 l/s turbomolecular pump and 40 m^3/h rotary pump, had a flow rate in the range 70–350 μmol/s. The cooling power measured at 120 mK, 0.360 mW, is comparable to that of excellent classical dilution refrigerators. This can be seen on Fig. 3. The measured minimum temperature was 8 mK at about 150 μmol/s. This is somewhat higher than expected, but it must be pointed out

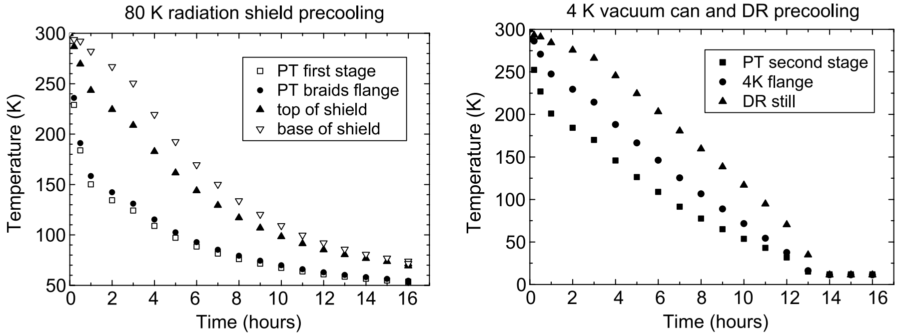

Fig. 2 Typical PT-DR2 pre-cooling curves

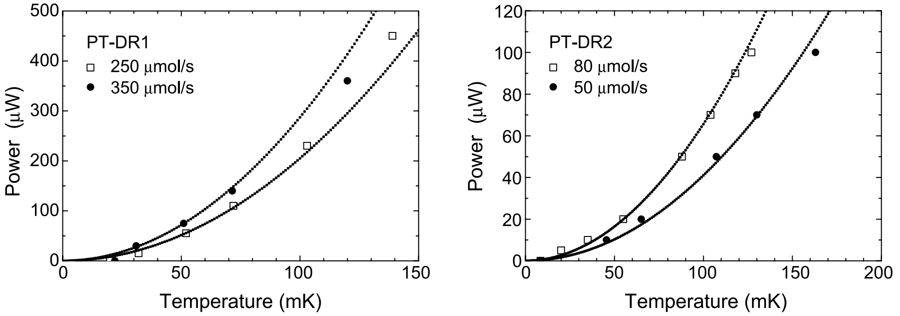

Fig. 3 Cooling power of PT-DR1 and DR2 for different circulation conditions. *Lines*: theoretical values for the ^{3}He flow rates given in the figure

that during these tests the system was NOT specially insulated against vibrations (pumping lines, supports, etc.). The PT-DR2 was tested with a small pumping system (300 l/s turbo-molecular pump and 18 m^3/h rotary pump). We measured a minimum temperature of 8.5 mK, at flow rates of 45 to 75 μmol/s.

6 Conclusions

We have developed very low temperature ($T < 10$ mK), high power (circulation rate larger than 0.3 mmol/s) dilution refrigerators free of cryogenic fluids baths (PT-DR). The pulse-tube refrigerator used in our experiments is a commercial system which delivers sufficient cooling power to pre-cool large-scale dilution refrigerator systems. The dilution units work extremely well, in spite of the differences between cryogenic bath vs. cold plates thermal grounding, and possible vibrations of the pulse-tube cooler. Their performance is basically identical to that of conventional dilution refrigerators. The particular characteristics of the PT-DR make this system particularly adapted to difficult environments. This includes underground laboratories where large cryogenic detectors of cosmic particles are installed, and where helium and nitrogen cannot be used easily, as well as dilution units for neutron scattering, optical, radio-frequency and other measurements which require a short and direct (no cryogenic fluids in the way) access from room temperature to the experimental cell. These advantages, together with the simplicity of operation, will certainly make PT-DR a common laboratory instrument in the next few years.

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