Calorimeter for a top-loading dilution refrigerator in high magnetic fields

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Abstract

We have developed a relaxation calorimeter for specific-heat measurements at millikelvin temperatures. The special features of the calorimeter are compatibility with high magnetic fields and stability at temperatures above the normal operating range of dilution refrigerators. The calorimeter has been successfully used to measure the specific heat of milligram samples at temperatures between 34 mK and 3 K and in magnetic fields up to 18 T. The samples can be cooled from room temperature to the lowest temperature in just five hours. As an example, we give an experimental result on high-purity copper at around 0.5 K in a 18 T field.

Key words: calorimeter; high magnetic field; millikelvin temperatures

Currently, there is much need for small-sample calorimeters for specific-heat measurements at millikelvin temperatures in high magnetic fields. One research area that will particularly benefit from such instrument is low-dimensional qauntum magnetism, where field-assisted long-range order often occurs at temperatures below 1 K. Another area is heavy fermions, in which a novel behavior in specific heat sometimes emerges at low temperatures that are beyond the reach of a ^{3}He refrigerator.

In this paper, we present the design of a relaxation calorimeter for a top-loading dilution refrigerator [1] on the 20 T superconducting magnet at the National High Magnetic Field Laboratory. The calorimeter is capable of cooling milligram samples from room temperature to 34 mK in five hours and measuring specific heat over two decades in temperature, between 34 mK and 3 K, without disrupting a stable operation of the dilution refrigerator.

Figure 1 shows the main components of the calorimeter that attaches to the bottom of the top-loading probe that is directly inserted in the plastic tail of the mixing chamber. An essential feature of the design is the vacuum can that thermally isolates the calorimeter from the cold liquid helium in the mixing chamber. 24.6 mm in diameter and 61 mm in height, the can is made of brass to block thermal radiation and to reduce eddy-current heating in the calorimeter and the sample. A grease joint with a taper of 7° provides a superfluid-

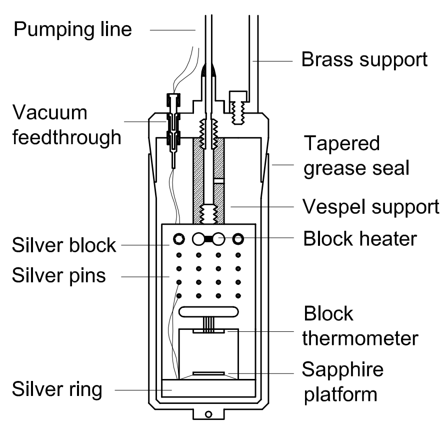

Fig. 1. Schematic cross section of the calorimeter cell.

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tight vacuum seal [2]. The can is evacuated at room temperature via a 1.3 mm-diameter CuNi tube, which runs along the length of the top-loading probe up to the 1 K pot, where the diameter increases to 3.2 mm.

The vacuum feedthrough for the electrical leads consists of two rows of six gold-plated socket pins taken from a single in-line pin (SIP) socket [3] and epoxied with Stycast 2850FT [4] into individual holes drilled on the lid of the vacuum can. The original spacing of the pins of the SIP socket is maintained in the feedthrough, so that a pair of SIP sockets for the external leads and another pair for the internal leads will directly plug to it from above and below, respectively.

In order to keep its time constant in the magnetic field reasonably short, the thermal reservior is entirely made of silver, which has one of the smallest nuclear heat capacities. The reservoir consists of a 6.3 mm-thick silver block, which attaches to the lid of the vacuum can via a Vespel SP-22 [5] rod, and a silver ring welded to the bottom of the block. A vertical gap has been cut in the ring to reduce eddy-current heating.

The thermal link between the silver reservoir and the cold liquid helium outside the vacuum can is provided by twelve 79 μm-diameter silver leads that run from the silver block to the feedthrough. On the silver block, the end of each lead is tightly wrapped around a 0.64 mm-diameter silver pin and covered with EPO-TEK 417 silver epoxy [6]. The pins have been cut from a Formvar-insulated wire and glued with Stycast 2850FT [4] into tight holes drilled in the silver block.

A 220 Ω Speer carbon resistor that has been ground to a 0.5 mm thickness is used as the reservior thermometer. A Karma-alloy (Ni 74%, Cr 20%, Al 3%, and Fe 3%) wire of a 79 μm diameter and a 35 cm length is wound on the block as a heater.

The sample platform is a sapphire disk, 6.4 mm in diameter and 0.13 mm in thickness. A small slice of a 220 Ω Speer resistor attached to the platform with EPO-TEK H31LV silver epoxy [6] is used as a thermometer. The platform heater is a thin evaporated layer of a Cr-7% Ti alloy. The electrical leads for the thermometer and the heater are 76 μm-diameter Pt-10% Rh wire, which also serves as mechanical supports and a weak thermal link for the platform. Each lead is soft-soldered to a heat sink made of a small piece of 0.1 mm-thick silver foil that has been glued with Stycast 2850FT to the ring.

To test the performance of the calorimeter, we have measured the heat capacity of 284.34 mg of high-purity copper in a 18 T field. The sample was glued to the platform with a thin layer of Wakefield 120 silicon compound [7]. The temperature difference ΔT across the weak link between the platform and the silver ring was produced by the Cr-Ti heater, and its relaxation after the heater was turned off was measured by a lock-in amplifier as a null detector of a Wheatstone bridge. A relaxation curve at 0.505 K is shown in Fig. 2. Whereas a single-exponential decay was observed at zero field, the curve at 18 T showed a double-exponential decay due to the heat capacity of the nuclear magnetic moments in the magnetic field [8]. The heat capacity was obtained from a separately-measured thermal conductance of the weak link and a non-linear least-squares fit of the relaxation data to a double-exponential form. After subtraction of the addenda from the total heat capacity, a specific heat of 4.25 mJ/K mol was obtained for the copper sample. The value agrees within 4% with the sum of a calculated nuclear specific heat, 4.05 mJ/K mol, and the zero-field specific heat of 0.36 mJ/K mol for copper [9] at this temperature.

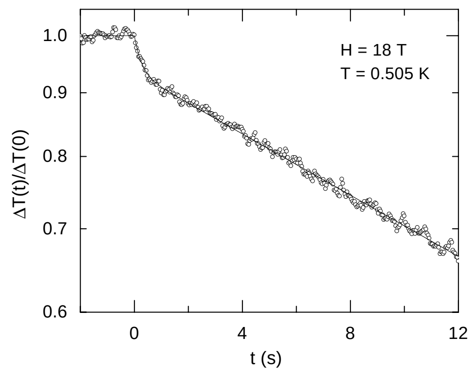

Fig. 2. Temperature relaxation of the calorimeter with a copper sample in 18 T at 0.505 K. The solid line is a non-linear least-squares fit of the data to a double-exponential form.

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