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A continuous ^{3}He cryostat with pulse-tube pre-cooling and optical access

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ABSTRACT

We have designed and constructed a continuously operating ^{3}He cryostat with windows for laser ablation and spectroscopy. A two-stage pulse-tube refrigerator cools two platforms to base temperatures of approximately 40 K and 4 K respectively. The platforms are equipped with heat exchangers that cool separate streams of ^{4}He and ^{3}He. The ^{4}He stream is used to run a thermally isolated evaporative refrigerator with a base temperature of approximately 1.5 K and a cooling power of 20 mW. The ^{4}He refrigerator is used to condense ^{3}He, which is used to run a ^{3}He evaporative refrigerator on the experimental cell. The cryostat runs continuously at temperatures from 10 K to 0.4 K with a cooling power of 1.5 mW at 0.5 K.

1. Introduction

The high cost of liquid helium and the advantages of continuous operation have made closed-cycle cryocoolers an increasingly popular choice for research cryostats [1,2]. Pulse-tube coolers [3–5] have no moving parts and relatively low vibration and have a base temperature of ∼3–4 K. To reach lower temperatures, additional refrigeration stages are required. ^{4}He evaporative refrigerators are effective down to ∼0.9 K. For lower temperatures, either ^{3}He evaporative refrigerators [6] or ^{3}He–^{4}He dilution refrigerators [7–13] are used. The choice between these two technologies depends on the base temperature and cooling power requirements of the application. For temperatures above 0.4 K, the cooling power of a ^{3}He refrigerator greatly exceeds that of a dilution refrigerator [14].

Our laboratory has been engaged in a number of investigations which require temperatures in the range ≈0.4–10 K, optical access, and relatively large heat loads; these include in situ laser ablation [15], ellipsometry [16,17], optical imaging for superfluid flow experiments [18,19], and studies of friction at low temperatures [20,21]. In this paper we describe the design and performance of a home-built cryostat based on a commercial pulse-tube refrigerator (PTR). The PTR maintains two radiation shields at approximately 40 K and 4 K, respectively. Each shield as well as the outer vacuum tank has four large 63.5 mm diameter windows for optical access to the experimental cell. Low temperatures are achieved using a closed cycle ^{3}He evaporative refrigerator. The ^{3}He is liquified in a separate ^{4}He refrigerator which uses gas from a room temperature tank that is passed through the system only once. The cryostat can be operated from 10 K to 0.4 K and may be run continuously for months at a time.

2. Construction

2.1. Cryostat

The cryostat is based on the PT410 PTR purchased from Cryomech [22]; it is a two-stage pulse-tube cooler. The first stage has a cooling power of 40 W at 45 K and the second stage has a cooling power of 1 W at 4.2 K. The base temperature with no heat load is approximately 2.7 K. A schematic diagram of the cryostat is shown in Fig. 1. The PTR is suspended from a 0.66 m diameter stainless steel plate (**a**), which constitutes the top of the vacuum chamber (**b**) that contains the cryostat. The PTR has two copper disks which provide the thermal connection to the upper, 40 K stage, and lower, 4 K stage. Each of these copper disks is bolted to a 12.5 mm thick copper plate of diameter 0.406 m (**c**) and 0.305 m (**e**), respectively, which form the upper flange for the radiation shields. Pumping lines which penetrate the flange include a bellows section to accommodate slight misalignment of the plates and a right-angle to prevent infrared radiation from reaching the cold areas of the cryostat. The cylindrical radiation shields (**d** and **f** in Fig. 1) were bolted to the corresponding flanges. The flange on the inner 4 K shield uses an indium wire O-ring to provide good thermal contact and to form a vacuum seal. The experimental cell hangs from the 4 K flange supported by three stainless steel tubes (0.238 m long, 12.3 mm outer diameter, and 0.5 mm wall thickness). The experimental cell is made of OFHC copper and is 0.203 m high with a 0.152 m outer diameter, and has a mass of 15.6 kg. Because of the low conductivity supports, the cell is essentially thermally isolated from the 4 K flange. During the initial cooldown, a small

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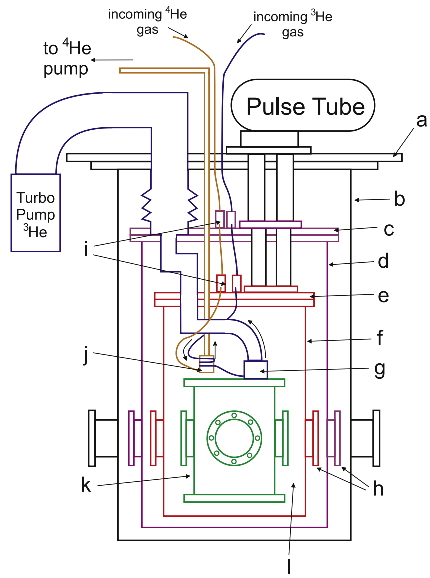

Fig. 1. Schematic of the cryostat. Components are labeled as follows: (a) stainless steel vacuum flange; (b) stainless steel outer vacuum can; (c) 40 K copper plate; (d) 40 K copper radiation shield; (e) 4 K copper plate; (f) 4 K copper radiation shield; (g) ^{3}He pot; (h) IR-absorbing windows; (i) heat sinks for incoming ^{3}He and ^{4}He; (j) ^{4}He pot. Incoming ^{4}He is pre-cooled (40 K) and subsequently liquified (4 K) by two heat sinks, then passed through a capillary and pumped away by a mechanical pump. Incoming ^{3}He is pre-cooled by both stages of the pulse-tube and then liquified (1.6 K) by a heat sink attached to the ^{4}He pot. It then passes through a flow impedance and is pumped away by a molecular turbo pump. The ^{3}He pot is bolted to the experimental cell, and both rest inside the vacuum-tight 4 K copper shield.

amount of helium exchange gas is admitted in to the inner vacuum space. Once the temperature of the cell approaches 4 K, the exchange gas is pumped out and the remainder of the cooldown proceeds using the ^{3}He and ^{4}He refrigerators.

The flanges, radiation shields, and the cell were all made of OFHC copper. The plumbing feed throughs and the flanges for the windows were vacuum-furnace brazed using a nickel alloy at Thermal-Vac [23]. The high-temperatures of the brazing process anneals the copper and makes it very soft. The initial tightening of bolts on the flanges and evacuation of the vacuum vessels strains the copper and rapidly work-hardens it, but care must be taken while the copper is in the soft annealed state to avoid warping the flanges or buckling the walls of the vessel. The outer vacuum can is 0.762 m tall with a 0.457 m outer diameter. The 40 K radiation shield is 0.663 m tall with a 0.358 m outer diameter, and the 4 K radiation shield is 0.444 m tall with a 0.254 m outer diameter. The radiative heat load on the 40 K shield due to the room temperature is 390 W while the radiative heat load on the 4 K radiation shield is 0.058 W. To reduce the radiative heat load on the shields, the 4 K radiation shield was gold plated and the 40 K radiation shield was plated with nickel. There were three types of thermometers used on the cryostat; an RTD Platinum thermometer placed on the 40 K plate of the pulse-tube for temperatures above 40 K, a silicon diode thermometer placed on the 4 K plate of the pulse-tube for temperatures above 4 K, and 4-wire germanium thermometers on the refrigerators and cell for temperatures above 0.3 K.

Six flanges for four 63.5 mm diameter windows and two 23.5 mm diameter windows were brazed into each shield to allow for optical access (**h** in Fig. 1). The original method of making a vacuum seal used an indium wire O-ring pressed between the window and the metal flange [24], but this lead to problems with leaks and to window breakage due to differential contraction and overtightening. Subsequently, we made titanium flanges with a thin (0.25 mm wall thickness) tube extension similar to those described in reference [16]. Titanium is used because it closely matches the thermal expansion coefficient of many types of glass. The windows were epoxied with Stycast 2850 FT into the titanium flanges, which in turn were vacuum-sealed onto the shield flanges using indium wire. The windows were 3.25 mm thick and made of Schott KG1 glass, which strongly absorbs infrared radiation but passes visible light.

The cell also has four 63.5 mm diameter windows and one small 25 mm window located below the plane of the four larger windows. Each window is made of quartz and is sealed with an indium O-ring. The cell is used for isothermal experiments on helium adsorption and hydrodynamics, so it is essential to be able to add helium at low temperatures without causing a heat leak due to superflow and to be able to pump the helium out after the experiment is finished. Helium was admitted through a 0.152 m long nickel capillary with an inner diameter of 0.05 mm. Even with bulk superfluid in the cell, the heat leak through the capillary was negligible. Pumping out the helium requires a connection with a much higher pumping speed. This was accomplished using a Varian UHV all metal valve (part number 9515085) mounted on top of the cell. Although not originally intended for low temperature use, the valve was made cryogenically compatible by replacing all polymeric seals and O-rings with indium with no impact on the functionality of the valve. The valve can be opened and closed by manually turning a rod connected to a rotary feed through (Varian L6691301) that passes through the inner vacuum space and out of the cryostat. The mechanical connection to the valve body was through a slotted screwdriver mechanism that could be parked in a neutral position with no thermal contact from the 4 K flange to the cell.

2.2. Refrigerator

A diagram of the refrigerators can be seen in Figs. 1 and 2. The evaporative ^{3}He and ^{4}He cooling systems were designed as follows. The incoming ^{4}He gas comes from a standard ultra-high purity grade bottle of ^{4}He passing through a liquid nitrogen cold trap. The typical incoming gas pressure is ~266 kPa. The ^{3}He comes from a closed-cycle system (not shown in Fig. 1) which uses an oil-free Varian SH-100 hermetically-sealed scroll pump to recirculate the ^{3}He gas from the cryostat through a liquid nitrogen cold trap, then passes it back into the cryostat at a maximum pressure of 133 kPa. In a traditional ^{3}He cryostat, the incoming ^{3}He and ^{4}He

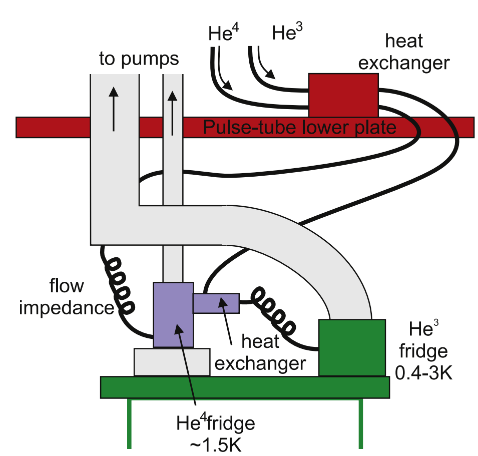

Fig. 2. A diagram of the refrigerator. The ^{3}He and ^{4}He enter a set of wrapped heat sinks. The ^{4}He enters a ^{4}He refrigerator to be pumped away while the ^{3}He passes through a final wrapped heat sink where it is finally cooled and condensed before entering the ^{3}He refrigerator.

gas needed for continuous evaporation are pre-cooled by a bath of liquid ^{4}He at 4.2 K. Because the cryostat is cryogen-free, this is not possible. Instead we cool the incoming ^{3}He and ^{4}He gas streams using several copper heat exchangers (**i** in Fig. 1) bolted to the 40 K and 4 K flanges. The first heat exchanger on the 40 K stage is composed of 6.25 mm diameter tubing soldered to copper blocks. The tubes are filled with copper pellets to increase the heat transfer area. The second heat sink on the 40 K stage consists of approximately 20 wraps of 0.5 mm diameter nickel tubing silver soldered around a copper cylinder 25 mm in diameter. Two similar heat sinks on the 4 K flange are of the same design. The heat exchangers cool both gas streams to ~4 K. The ^{4}He stream is liquified in the heat exchangers on the 4 K flange and passes through a stainless steel capillary flow impedance into a copper pot where it is pumped away through a 6.25 mm diameter pumping tube out of the cryostat and into the atmosphere by a mechanical pump. The ^{4}He pot is thermally isolated from both the 4 K flange and the experimental cell and operates at a typical temperature of 1.5 K. The ^{4}He pot provides a low temperature heat sink for a heat exchanger (see Fig. 2) composed of 10 wraps of 0.5 mm diameter nickel tubing soldered around a 12.5 mm diameter copper post that is used to cool and liquify the ^{3}He stream. The ^{3}He passes through a flow impedance and into a ^{3}He pot which is pumped with a hybrid turbo-molecular drag pump (Pfeiffer TMH 260) backed by the scroll pump. The ^{3}He pumping lines are 25 mm in diameter at the low temperature section and 0.10 m in diameter at room temperature. The ^{3}He gas is recirculated and sent back into the cryostat as described above.

The performance of the refrigerators depends critically on the value of the flow impedances. The impedance Z is defined [14] by:

$$Z = \frac{\Delta P}{\dot{V}\eta} \tag{1}$$

where ΔP is the pressure drop across the impedance in pascal, $\dot{V}$ is the flow rate measured in m^3 s^{-1}, η is the dynamic viscosity of the fluid in Pa s, and Z has units of m^{-3}. The flow impedances were characterized at room temperature by pressurizing a $V_1 = 1$ l volume with ^{4}He gas to a pressure $P_1(t=0) \approx 10$ atm and monitoring $P_1(t)$ as a function of time as the helium leaked out through the capillary into the room at pressure P_{atm}. For flow of a compressible gas, the $\dot{V}$ term in Eq. (1) is not constant along the tube even if the number flow rate $\dot{N}$ is constant because the pressure varies along the tube. Assuming that the characteristic pressure is the average pressure, the relation between $\dot{N}$ and $\dot{V}$ is $\dot{V}(P_1(t) + P_{atm})/2 = \dot{N}kT$. Substituting this expression into Eq. (1) yields an equation for $\dot{P}_1$:

$$V_1\dot{P}_1 = -\frac{1}{2Z\eta}(P_1 + P_{atm})(P_1 - P_{atm}) \tag{2}$$

Fitting numerical solutions of this equation to measured values of $P_1(t)$ yielded values of Z. Initially the flow impedances for the ^{4}He and ^{3}He were constructed using the traditional method [25,26] by placing a wire inside a stainless steel tube. A simple fluid mechanics calculation [27] shows that the impedance for this annular geometry should be proportional to the length of the tube. We found, however, that the measured values of the impedance made by the wire-in-tube technique often deviated from this prediction by more than an order of magnitude and were not reproducible. Large changes in the length sometimes produced no observable change in the value of the impedance, while sometimes the impedance would drop by an order of magnitude. We believe that this stochastic behavior is due to localized high impedance sections caused by imperfections in the tube or wire, and debris in the tube [25].

To avoid these problems, the impedances were made using very small diameter ultra-precision bore tubes purchased from VICI Valco Instruments Co. They are nickel tubes and can have inner diameters as low as 0.025 mm. The tubes are manufactured by electroplating nickel onto a carbon fiber of a certain diameter. The surface of carbon fibers are extremely uniform and smooth therefore provide nearly perfect tubes. The thermal conductivity of nickel is considerably higher than stainless steel. To avoid thermal conduction down the impedance, we always used a section of relatively large bore stainless steel tubing in series to block heat conduction.

The theoretical value of the flow impedance for a cylindrical tube is [27]

$$Z = \frac{8l}{\pi}R^{-4} \tag{3}$$

where R is the radius of the tube and l is the length. The measured impedance of the tubes were found to be within a factor of 2 of the value predicted in Eq. (3) using the manufacturer's value for the tube radius, and the values were systematically linear in the length of the tube. One ^{4}He impedance was made using a nickel tube with an inner diameter of 0.05 mm and an outer diameter of 0.36 mm with a 88 mm length, which had an impedance measured to be 6.65×10^{17} m^{-3}. Two ^{3}He impedances were made using the nickel tubing. The first was made from a tube with an inner diameter of 0.025 mm, an outer diameter of 0.36 mm, a length of 56 mm, with $Z = 5.5 \times 10^{18}$ m^{-3}. The second ^{3}He impedance was made from a nickel tube with an inner diameter of 0.056 mm, an outer diameter of 0.79 mm, and a length of 0.25 m with $Z = 1.63 \times 10^{18}$ m^{-3}.

These impedance values were determined by trial and error to ensure that both refrigerators operated in a regime in which they were "wet" and contained bulk liquid. This was determined by measuring the cooling power which is the steady-state temperature as a function of the heat input. The cooling curves for the ^{3}He refrigerator are shown in Fig. 3, and the cooling curves for the ^{4}He refrigerator are shown in Fig. 4. Resistive heaters were placed on the cell near the ^{3}He refrigerator and on the ^{4}He refrigerator and were used to add heat to the system. To avoid hysteresis, measurements were done from high temperature to low temperatures. Both curves show two distinct regions: at low power levels, the temperature is low and very weakly dependent on the

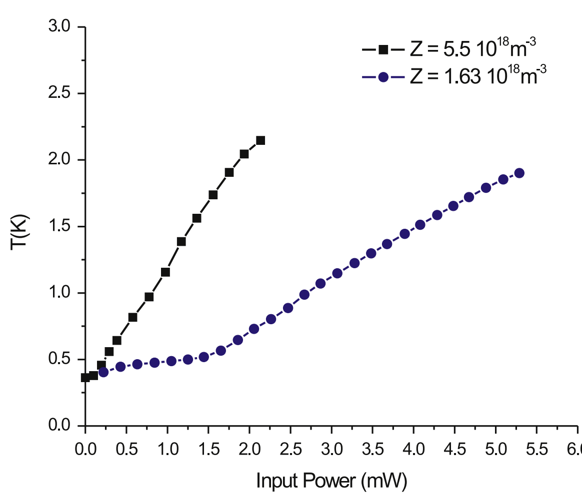

Fig. 3. Temperature as a function of input power for the ^{3}He refrigerator for two different impedances. The cooling curve was measured with an input pressure of 101 kPa and a constant pumping speed. Changing the impedance from $Z = 5.5 \times 10^{18}$ m^{-3} (black squares) to $Z = 1.63 \times 10^{18}$ m^{-3} (blue circles) dramatically changes the performance of the refrigerator. An abrupt change in the slope which occurs at 1.75 mW and 0.5 mW, respectively, signifies dry-out of the refrigerator. For higher power levels the refrigerator runs in a dry state and the temperature is linear in the input power. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

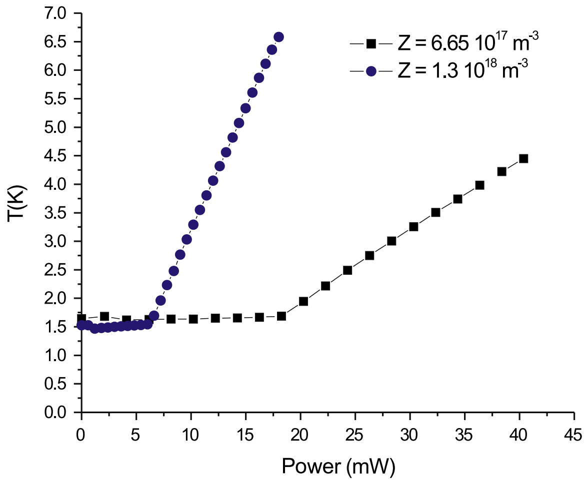

Fig. 4. Temperature as a function of input power for the ^{4}He refrigerator for two different impedances. The cooling curve was measured with a constant inlet pressure of 266 kPa. Two different impedances were measured, one with a value of 6.65×10^{17} m^{-3} represented by squares and another 1.3×10^{17}m^{-3} represented by circles. The discontinuity in the slope represents the drying out of the refrigerator and occurs at 6 mW for the larger impedance and 20 mW for the smaller impedance. For larger power inputs the refrigerator is in a dry state and the the temperature is roughly linear in the applied power. For the optimal operation of the system the ^{4}He refrigerator must be as cold as possible.

input power (characteristic of a wet state), but above a threshold power, the temperature rises approximately linearly (characteristic of a dry state) [14]. The critical power level that will lead to dry-out using the 6.6×10^{17} m^{-3} impedance for the ^{4}He refrigerator is 20 mW, and the critical power is 1.75 mW for the ^{3}He refrigerator using an impedance of 1.63×10^{18} m^{-3}. If the impedance for the ^{4}He is too large, then it will lack the cooling power necessary to liquify the ^{3}He and will run in a dry mode. Because the source of ^{4}He is a bottle instead of a bath at 101 kPa, the inlet pressure to the refrigerator can be varied. Eq. (1) suggests that increasing this pressure will increase the flow rate and therefore increase the cooling power. Although the flow rate in the refrigerator does change, it has been observed that when the ^{4}He is running in a dry mode and the inlet pressure is increased, the cooling power of the refrigerator is not significantly altered.

The flow rates of the ^{3}He and ^{4}He gas streams were measured while the refrigerators were in steady state operation. The flow rate of the ^{3}He gas stream was determined by measuring the pressure drop across a calibrated room temperature flow impedance in the ^{3}He gas loop. We found that the flow rate of ^{3}He is a linear function of the pressure drop as long as the temperature of the ^{4}He refrigerator is held constant. For a pressure drop of ~101 kPa, the flow through the impedance is 1.5 mmol/s. The heat load on the ^{4}He refrigerator to cool this flow rate of ^{3}He from 3 K to 1.5 K is approximately 18.2 mW. The flow rate of the ^{4}He gas stream was measured by measuring the flow output of the pump on the ^{4}He refrigerator and was found to be 0.35 mmol/s at an input pressure of 225 kPa. Changing the input pressure of the ^{4}He refrigerator did not create a proportional linear response in the flow rate due to two phase fluid flow through the impedance and other pressure drops in the system.

3. Operation and performance

Cooldown was initiated by establishing the flows of ^{3}He and ^{4}He gas through the systems with the apparatus at room temperature. Even with a sealed scroll pump in the ^{3}He system and the use of high purity ^{4}He gas from a tank, we found that we would occasionally plug the capillaries if we did not pass the gas through liquid nitrogen temperature charcoal traps. Before the initial cooldown, a few kilopascals of ^{4}He is added to the 4 K vacuum space to provide a thermal link to the experimental cell. Once the PTR is turned on, it takes 16 h to cool the cell from 300 K to 4 K. If high heat loads are required for brief periods, for example during laser ablation or activation of motors, the exchange gas can be used to cool the cell. With exchange gas in the inner vacuum space, we found that the cell temperature remains below 10 K even with heat loads of approximately 0.5 W. The exchange gas can be removed by raising the inner shield temperature to approximately 6 K and pumping with a diffusion pump. Once the exchange gas is removed, approximately 1 h is required for the ^{3}He refrigerator to cool our relatively massive cell to below 1 K.

Table 1
Temperature of cryostat components.

Component	T (K)
Lower plate	2.7
Upper plate	44
Outer radiation shield	44.5
Inner shield	4.0
^{3}He fridge bottom temp.	0.36
^{4}He fridge bottom temp.	1.53

The temperatures of the various components of the system during operation are shown in Table 1. In order to assess the optical power that may leak through the windows, we tested the performance of the cryostat with the using both KG-1 windows and copper blanks in the window flanges on shields; the base temperature of the cell varied by less than 10 mK. The temperature of the cell could be regulated precisely even when the ^{3}He refrigerator was in the dry regime, which extended the range of operation to above 2 K. We also explored another mode of operation of the cryostat in which ^{4}He is used in both cryogen circuits, i.e. ^{4}He replaces the ^{3}He and is pumped on with the turbo pump. In this mode, the base temperature was 0.8 K, but the cooling power was significantly larger using ^{3}He. A cooling curve for the ^{3}He refrigerator using ^{4}He is presented in Fig. 5.

An important constraint on the performance of our cryostat is the base temperature of the ^{4}He refrigerator, which in turn limits the base temperature of the ^{3}He refrigerator. If the temperature of the ^{4}He refrigerator is 2 K or above, the temperature of the

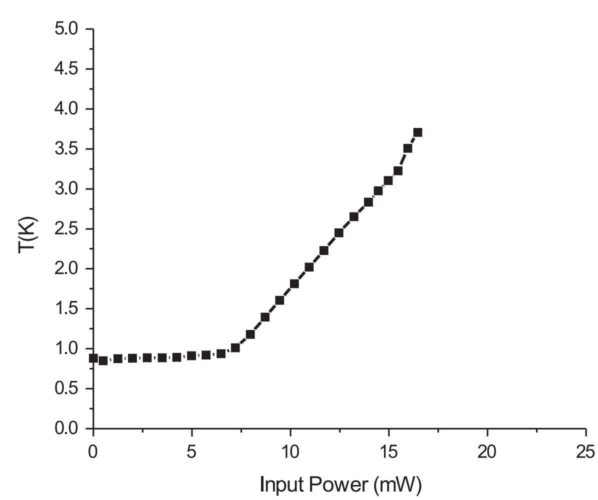

Fig. 5. The cryostat is designed to allow the user to place ^{4}He inside the ^{3}He refrigerator. This mode is useful if more cooling power is needed for temperatures greater than 0.8 K. The machine runs in dry mode above 6.5 mW. This cooling curve was done with an impedance of 5.5×10^{18}m^{-3}.

^3He refrigerator will not go below 1 K. The ^4He refrigerator temperature is currently limited to about 1.5 K due to the small pumping line diameter (6.25 mm) on the room-temperature side of the ^4He gas handling system. In a future upgrade of our system, we plan to increase the pumping tube size which should lower the base temperature to ≈1 K and thereby improve both the cooling power and the base temperature of the ^3He refrigerator.

To warm up the cryostat, the PTR is turned off and ^4He gas is added to the inner and outer vacuum cans. It is important to keep the pressure of the ^3He in the ^3He refrigerator below 66 kPa during warm up to prevent any over pressurization of the system due to the sudden evaporation of the ^3He. No special precautions need to be taken for the ^4He. Warming the cryostat from 4 K to 300 K using conduction from the environment takes approximately 18 h.

4. Summary

The construction and operational details of a ^3He refrigerator with pulse-tube pre-cooling has been described. The cryostat requires only electrical power, liquid nitrogen, and a source of room temperature ^4He gas, and the latter could easily be replaced with a recirculating gas handling system. The rate of use of ^4He gas is 172.5 moles per week. Assuming helium costs $0.5 per mole, the resulting cost is approximately $80 per week. The PTR uses 10 kW, so assuming a cost of $0.15 per kW h, the power cost for operation is $252 per week. Because liquid cryogens do not need to be transferred, experiments can be run uninterrupted for months, allowing for more complex and time consuming measurements. In comparison with pulse-tube based dilution refrigerators [9], our cryostat has a higher base temperature, but a much larger cooling power and a wider temperature range of operation. It can also tolerate brief periods of high power input without suffering thermal runaway. Similarly, our cryostat has a slightly higher base temperature than a sorption-pumped ^3He system [6], but has the advantage of continuous operation.

Acknowledgments

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