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An Electronic Demagnetization Stage for the 0.5K to 1.8K Temperature Range

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We have designed and built a demagnetization stage to operate between 1.8K and 0.5K, a somewhat unusual temperature range for adiabatic demagnetization refrigeration. The lower bound in temperature is dictated by the thermal performance of our ^{3}He gas heat switch, the upper by NASA specifications. We intend this demagnetization stage to be the first stage of a proposed two-stage continuously operating adiabatic demagnetization refrigerator (ADR) which will operate between 1.8K and 50 mK. Here we discuss thermal, mechanical, magnetic and chemical considerations which led to novel features in our salt pill design.

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1. INTRODUCTION

We are engaged in experiments probing the phase diagram of ^{3}He-^{4}He mixtures in aerogel. Our work has demonstrated that the morphology of the interface between ^{3}He and ^{4}He rich mixtures depends on deposition conditions. In particular, a mixture deposited in aerogel below the phase separation temperature creates a metastable "film" state in which the ^{4}He rich phase coats the aerogel strands with a thick film. Further we have observed that this film state collapses at a critical ^{4}He concentration. We interpret this collapse as the onset of capillary condensation at a critical film thickness. Gravitational effects on the film thickness precipitate this collapse as the film is thicker at the bottom of the aerogel than on average. Performing our experiments in the absence of gravity will allow us to probe a wider range of film thicknesses. The temperature range of interest is below

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100mK.

We have need, then, of a means of cooling to 50mK in a microgravity environment. Standard versions of the ^{3}He-^{4}He dilution refrigerator need gravity to operate. A refrigerator developed for space use[1] has a disappointing efficiency, cooling power and minimum temperature compared to earth-based systems. In recent years several adiabatic demagnetization refrigerators (ADR) have been developed for space applications, particularly for use in cooling astronomical detectors[2]. These refrigerators are single-shot devices with a typical cooling power of 1μW at 100mK. We are developing a cyclic ADR that will have a performance competitive with conventional 100μmole/sec dilution refrigerators which typically have cooling powers of $\approx$100μW at 100mK.

A schematic view of the proposed refrigerator is shown in Figure 1. The cooling source for the refrigerator is a pumped liquid ^{4}He heat-sink at 1.8K. A thermal switch connects a paramagnetic salt "pill" to the 1.8K thermal reservoir, and a second identical heat switch connects it to a 0.5K intermediate temperature platform. Two parallel refrigerators operate out of phase to provide a stable temperature. While demagnetizing one salt pill to extract heat, the other is regenerated at 1.8K, then demagnetized to 0.5K to extract heat while the first system is regenerated. Starting from this 0.5K platform, a similar paired system would achieve temperatures $\leq$50 mK. The overall cycle time is one hour, so the demagnetization and regeneration should take 1800 seconds each.

2. THEORETICAL THERMAL PERFORMACE OF FIRST STAGE

A 100μmole/sec dilution refrigerator at 50mK, extracts 15μW and in 1800 sec, 0.54J/K of entropy is removed[3]. Sinking this entropy to 0.5K over a 1800 sec regeneration time (allowing for non-ideal effects) produces a 0.3mW heat load. Similarly, the heat load to 1.8K is 2mW. The thermal performance criteria for the first stage described in this paper are that it should maintain 0.5K for 1800 seconds with a heat load of 0.3mW and then regenerate in 1800 seconds.

Gadolinium sulfate is attractive in this temperature range because of its low gram-ionic weight and large J value (7/2). It has an ordering temperature of 0.182K. Brillouin function calculations of entropy indicate that if we demagnetize 0.1 moles of the salt (the amount in our salt pill) to maintain a constant temperature of 0.5K with a field between 1.2T and 0.2T we will be able to absorb heat in the amount $\Delta Q = \Delta S \times T = 1.4J/K \times 0.5K = 0.7J$.

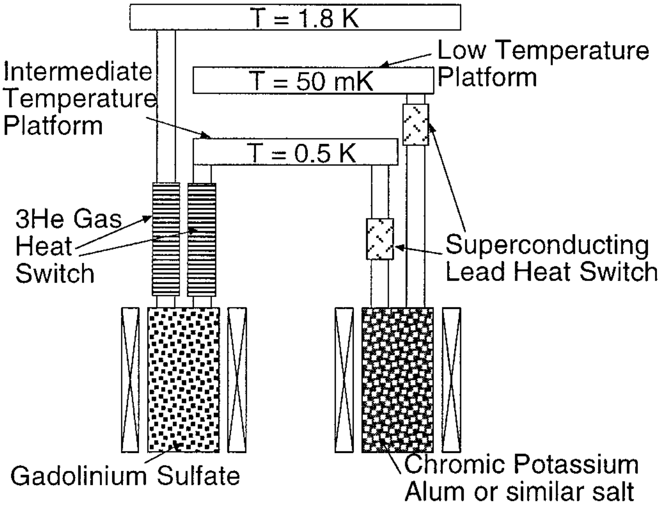

Fig. 1. Schematic two-stage refrigerator with a single salt pill for each of the two stages. Our proposed ADR will have two salt pills in parallel for each stage

Thus, at a heat input of 0.3mW, the holding time is on the order of 1/2 hour.

3. DESIGN AND CONSTRUCTION

3.1. ^{3}He Gas Heat Switch

We have previously reported[4] on the design, construction and performance of a ^{3}He gas heat switch to operate between 1.8K and 0.5K. Briefly, the switch consists of two concentric copper cylinders with a small gap between them which may be filled with ^{3}He gas ("on" state) or pumped out with a miniature charcoal adsorption pump ("off" state). The measured conductance of this switch is 50μW/K at 1.5K when "off" and 8mW/K at 0.5K when "on".

3.2. Construction of salt pill

The salt is contained in a cylinder (8cm long and 2cm diameter) constructed of a 'fibreglass' of paper soaked in 1266 Stycast epoxy (see Figure 2). This was relatively simple to construct and produced a strong, flexible,

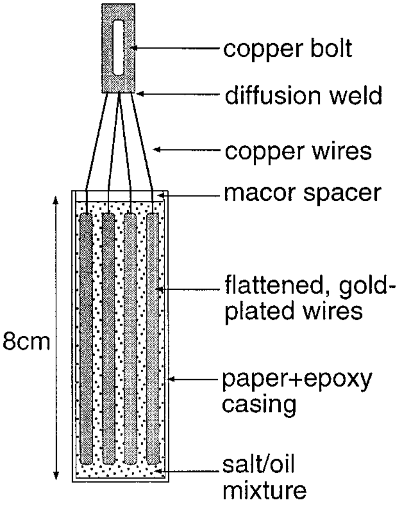

Fig. 2. The salt pill

leaktight casing. 20 copper wires (1mm diameter) are embedded in the pill to make thermal contact with the salt. These pass through a sealed macor spacer at the top of the cell and are anchored into a copper bolt by a diffusion weld. The section of the wire inside the pill has been pressed flat to increase surface area, and also goldplated to safeguard against corrosion (although the salt does not seem to be particularly corrosive).

Gadolinium sulfate has a very low solubility in water[5] (approximately 4g/100g water at T = 0°C) so we were unable to make a supersaturated aqueous solution. Instead we made a slurry of the powdered salt and Dow Corning 704 diffusion pump oil (oil is preferable to water because of water's expansion on freezing). To pack it in to the cell (around the embedded wires) we made a dilute salt/oil slurry which could be poured into the pill casing. The pill was then put in an ultrasonic bath for ten minutes. This made the salt settle to the bottom, leaving a layer of clean oil which could be siphoned off the top. We repeated this procedure several times to fill the cell casing with 0.1 moles of the powdered salt, achieving a packing fraction of over 50%.

3.3. Thermal Resistance Between Salt Pill and Copper Bolt

The two main contributions to the thermal resistance between the salt and the anchoring copper bolt are the resistance between the copper and the salt/oil slurry, and the thermal resistance of the wires themselves.

The thermal boundary resistance R_B between copper coil foil and a chromic potassium alum/oil slurry (a reasonable approximation for our system) is given by[3] $R_B = \frac{0.008}{AT^3}\frac{m^2K^4}{W}$ where A is the area of the interface and T the temperature. By pressing the wires flat we increased the surface area of the interface and decreased the boundary resistance by a factor of 2. For one flattened wire, the calculated boundary contribution to thermal resistance is 130K/W.

To calculate the thermal resistance, R_W, of the wires themselves we use the well-known Wiedemann-Franz law relating thermal conductivity κ and electrical conductivity σ: $\frac{\kappa}{\sigma} = L_0T$, where $L_0 = 2.44 \times 10^{-8} W\Omega/K^2$. To increase the conductivity, the assembled copper wires, macor spacer and copper bolt were heat-treated at 1200K for 16 hours in a low partial pressure of oxygen. (This heat treatment also served to make the diffusion weld between the wires and copper bolt). Electrical measurements showed an improvement of the resistance ratio to over 3000. The calculated thermal resistance of one wire is 40K/W.

The total calculated thermal resistance for one wire is then 170K/W, and for 20 wires there is a total thermal resistance of approximately 10K/W. (The thermal resistance of the copper bolt is less than 5% of this and can be neglected).

3.4. Other Considerations

To keep our cycle time short, fast ramping of the magnetic field is required, so eddy current heating is a concern. To minimise such heating in the anchoring copper bolt, slits were cut through the bolt to restrict the size of eddy current loops. The heat leak due to thermal radiation from 4K to 0.5K is extremely small and can be neglected.

4. PERFORMANCE

Our thermal cycle consists of magnetizing the salt pill at 0.005T/s to 3.2T, precooling to 1.8K, then demagnetizing at 0.01T/s to a final field of 0.5T. The final temperature obtained was 0.5K. Doubling or halving the rate of demagnetization had no noticeable effect on the final temperature. The temperature was measured by means of a ruthenium film thermometer mounted on the copper stage between the anchoring copper bolt and the heat switch. This thermometer read 1.8K after approximately half an hour of precooling. Precooling for an hour or more did not change the temperature reading from the thermometer but produced a slight improvement in the final temperature (less than 0.05K), suggesting that the salt pill is not in complete thermal equilibrium after half an hour of precooling.

By applying a heat load with a heater mounted on the copper stage above the salt pill we were able to measure the heat capacity of our salt pill at 0.5K in a 0.5T field. Applying 0.11J of heat over 3 minutes raised the temperature from 0.48 to 0.6K, giving a heat capacity of 0.8J/K. This is consistent with the theoretical prediction for 0.1 moles of the salt. With no applied heat load, the heat leak through the heat switch at 0.5K is 10μW, which is consistent with the previously measured heat leak through the ^{3}He gas heat switch in its off state.

5. CONCLUSION AND FUTURE WORK

Based on these preliminary results the performance of our salt pill looks very promising. The next step in testing is to maintain a stable temperature in the presence of an applied heat load while slowly demagnetizing the salt pill. Our design could possibly be optimized further by increasing the packing fraction of salt in our pill (perhaps by pressing) and decreasing the precooling times.

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