Development of a Miniature Adiabatic Demagnetization Refrigerator: An Instrument to Cool Detectors to 100 mK

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ABSTRACT

ADRs (adiabatic demagnetization refrigerators) have been used for many years to cool various types of detectors to temperatures below 1 Kelvin. They have several advantages over other types of low temperature refrigerators and have become the cooling instrument of choice for many astrophysics experiments. Unfortunately, their size, complexity and moving parts have limited the number of applications for which they are useful. We present the design and development of a Miniature ADR (MADR) that eliminates many of the drawbacks of current ADRs, and will allow for their use in a variety of applications.

1. Introduction

The detection of small signals from sources of interest has been an aim of science for many generations. In the past fifty years there has been a revolution in techniques and instruments used to achieve these goals. More and more of the pioneering experimental work in physics, astrophysics, and even molecular biology require that detections be made at levels that even ten years ago would have been considered lost in the noise of the instruments. Much of the progress can be attributed to the development of state of the art detectors such as bolometers, microcalorimeters, transition edge superconducting devices, and others. All of these detectors require continued cooling to temperatures around 100 mK. While detector development has experienced great growth, the development of new refrigeration technology lags behind. It was our goal to design, develop, and test a miniature adiabatic demagnetization refrigerator (MADR). We envision this refrigerator being available for use in both small and large scale experiments, a reliable, simple, and miniature cooling solution for many different applications.

2. Refrigerator Background

There are three types of refrigerators that can be used to cool detectors to below 1 Kelvin. ^{3}He refrigerators, dilution refrigerators, and adiabatic demagnetization refrigerators (ADRs) have all been used in various experiments. In this section, we describe each of these refrigerators including their operation, performance and drawbacks. It should be noted that all of these refrigerators must be installed in a cryostat to operate. A cryostat is an instrument that is used in almost all low temperature experiments. You begin with a sealed container called a dewar and use a vacuum pump to remove the air. This is important because as you cool the dewar, you don't want the air to exchange heat between the inside of the cryostat and the outside, and the gasses in the air would liquify or freeze at the low temperatures we deal with. After the dewar is under a vacuum, we precool the dewar by transfering liquid nitrogen into tanks inside the dewar, which are accesible through fill lines. After allowing the system to come to thermal equilibrium (typically overnight), we empty the liquid nitrogen from the internal tank and fills it with liquid helium. This way, the liquid helium at 4.2 K is surrounded by a liquid nitrogen shield at 77 K. This greatly reduces the boil off that would occur if the liquid helium stage saw the 300 K shell of the dewar. The liquid helium tank is thermally connected to a cold plate. This is the staging area for the refrigerators and all the experimental apparatus. Bath temperatures lower than 4.2 K can be achieved by pumping on the liquid helium bath. This is a result of the fundamental thermodynamics equation $PV = RT$, where R is the molar gas constant. With a constant volume, when the pressure is reduced, the temperature must also go down. Typically, one can reach temperatures of 2 K by this method.

2.1. ^{3}He Refrigerators

^{3}He refrigerators work by the simple process of evaporation cooling. Figure 1 from Betts shows a representative schematic of a ^{3}He refrigeration system. The main components of the system are housed in a pumped liquid helium cryogenic dewar with a liquid nitrogen shield. As shown in Figure 1, the ^{3}He refrigeration system is thermally connected to the pumped liquid helium bath. The ^{3}He is then pumped on by an external pump to reduce its vapor pressure. As is the case with the ^{4}He, as the pressure of the system is brought down, its temperature decreases. ^{3}He has the highest vapor pressure of any known gas which corresponds to it being able to reach the lowest temperatures when pumped on, typically about 300 mK. Because of the high price of ^{3}He it is efficient to incorporate a return tube for the pumped ^{3}He gas as shown in the figure. A modification of the refrigerator that does not require a second pump for the ^{3}He makes use of a charcoal sorption pump. In this case, the ^{3}He is completely contained in an apparatus that replaces the upper levels of the ^{3}He refrigeration system in the picture with a copper casing containing charcoal. By heating and cooling the charcoal, it is possible to raise and reduce the pressure of the ^{3}He vapor and reach 300 mK without the use of an extra pump.

^{3}He refrigerators have been in use since the mid 1960's with excellent results, they are fairly simple and relatively easy to use. However, they do require the use of expensive ^{3}He, they require either two pumping lines or the heating of parts of the system to significant temperatures, and they can only reach 300 mK. While 300 mK is very cold, many experiments use detectors whose noise can be significantly lower at temperatures not much lower. Their main drawback, however, is that the charcoal pump requires heating to about 20 K, which creates a significant heat load on the cryostat, and makes ^{3}He refrigerators fairly ineffeicient.

2.2. Dilution Refrigerators

The dilution refrigerator as pictured in Figure 2 operates on principles very similar to that of the ^{3}He refrigerator. One begins with a mixture of liquid ^{3}He and liquied ^4He in the mixing chamber. The liquid ^{3}He phase is lighter than the mixture of ^{3}He and ^{4}He and floats on top. Of course, to optimize the cooling process, one begins at a temperature of 300 mK, which is achieved by pumping on the liquid mixture as with the simple ^{3}He refrigerator. To reach temperatures below this, the ^{3}He-^{4}He mixture acts as a pump on the liquid ^{3}He phase. The mixture is an effective pump because the dilute ^{4}He acts like a vacuum due to its almost zero heat capacity, entropy, and viscosity [Betts]. So, by removing ^{3}He atoms from the dilute ^{3}He and ^{4}He mixture, it is possible to induce ^{3}He atoms from the ^{3}He phase to move down into the mixture; as they would if they were being pumped out of the ^{3}He phase and into the mixture. One can remove ^{3}He atoms from the mixture through the use of a still, as indicated in the figure. While the ^{3}He refrigerator loses cooling power at 300 mK because of the reduction of the vapor pressure of ^{3}He with temperature, the "mixture pump's" osmotic pressure approaches a constant. Dilution refrigerators are therefore able to reach temperatures of 5 mK.

The preceeding discussion, and the associated figure are extremely simplified. Dilution refrigerators and their operation are very complex, and typically require a large setup outside fo the apparatus. It is mainly the complexity that prevents the wide-spread use of dilution refrigerators. For many experiments, it is not worth the trouble of dilution refrigerators to reach temperatures below 50 mK. I mention 50 mK because that is the approximate lowest temperature that adiabatic demagnetization refrigerators can reach.

2.3. Adiabatic Demagnetization Refrigerators

Unlike ^{3}He and dilution refrigerators, adiabatic demagnetization refrigerators (ADRs) do not use ^{3}He to reach low temperatures. Instead, they use a paramagnetic salt to reach temperatures as low as 50 mK. Because they do not need extra external pumps, ADRs are somewhat simplier to use and setup than ^{3}He and dilution refrigerators; they do however, have their own drawbacks which will be addressed later in this section. Now, I will examine the three main components of an ADR, a heat switch, a salt pill, and a superconduting magnet. I will then describe the operation and performance of a conventional ADR, followed by a look at their drawbacks. See Figure 3 for a schematic of an ADR from Wilson.

3. ADR Components and Operation

3.1. Heat Switches

There are many situations in low temperature experimental physics that require some method to make and break thermal contact between a thermal bath and another area of the experiment. There are three standard ways to achieve a change of several orders of magnitude in thermal conductivity. Mechanical, gas-gap, and material based heat switches have all been used effectively for this purpose.

Mechanical heat switches with drives inside of a cryostat typically use a motor, spring, or solenoid to make and break contact. Professor Peter Timbie's[1] lab has succesfully used a solenoid driven mechanical heat switch in several experiments (see Figure 3 for a cross-section of the heat switch used). Through the use of a cam and lever arms, when the solenoid is pulsed with a current, the lever arms either open or close, thereby breaking or making contact with an extension of the salt pill. This is, of course, a simplified overview, but we are not concerned so much here with the operation, but rather the advantages and disadvantages. Mechanical heat switches do have an advantage over gas-gap and material based heat switches in that when they are in their "off" state, they are truly off, there is no thermal contact between the bath and the experimental space (ignoring small heat leaks through wires, suspension, etc.). Both gas-gap and material based heat switches do have some residual conductivity, but mechanical switches have a much lower conducivity in their on state. Mechanical heat switches also have the disadvantage of using moving parts at cryogenic temperatures. Oils and lubricants that would normally be used would typically be frozen at the temperatures we are dealing with, so there is always a concern about the reliability of the switches. In my experience with a mechanical heat switch, I found that the cam would occasionaly get stuck in position between on and off, and once there, the solenoid was unable to provoid enough torque to open or close the switch. So while mechanical heat switches do offer the convenience of truly being off, they can be plagued by reliablity problems as well as low thermal conductivity (I refer the reader to Wilson, where much of this paragraph was adapted from, for a more detailed discussion).

Gas-gap heat switches use ^{4}He gas to provide thermal contact between the bath and the refrigerant. Typically, a stainless steel

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[1]Professor Timbie is the author's thesis advisor in the University of Wisconsin-Madison Department of Physics. He can be contacted via e-mail at timbie@wisp5.physics.wisc.edu

tube is used (due to its low thermal conductivity) to contain the gas between the bath and refrigerant. Usually connected to the tube is a charcoal sorption pump similar to the one described for ^{3}He refrigerators. When good thermal contact is desired, the charcoal pump is heated to aproxiametly 20 K, which expells most of the ^{4}He gas that the charcoal abosrbs at low temperatures. The ^{4}He gas in the tube is then typically at atmospheric pressure and is a very good thermal conductor. When the switch is to be turned off, one allows the charcoal pump to cool to a temperature below 10 K. Once around 10 K, the charcoal begins to absorb almost all of the ^{4}He gas, leaving a vacuum in the tube. In this gas, the residual conductivity is due to the stainless steel tube, which can be made relatively thin walled to achieve a on/off conductivty ration of about 3 orders of magnitude (See Dubond et.al. for a more detailed discussion). Gas-gap heat switches can be very effective for some uses, but they do have several significant drawbacks. The entire heat switch apparatus tends to be large, which does not bode well for a miniature refrigerator. Also, the cycling of the heat switch is slow due to the time it takes to absorb and expel the ^{4}He gas. And, as with the ^{3}He refrigerator, the heating of the charcoal pump adds a significant heat load and reduces the efficiency of the refrigerator.

Material based heat switches make use of the properties of superconductors or magnetoresistors in magnetic fields. For these heat switches, the material is typically solderd between a connection to the bath and a connection to the refrigerant. The only other component to the switch is a magnet that can be turned on and off, typically a superconducting solenoid. Superconductors can achieve switching ratios of about 10^3 which is about equivalent to that of a gas-gap heat switch. Magnetoresistive heat switches (or thermal magnetoconductors) can achieve switching ratios of 10^4, which is surpassed only by the infinite ratio of mechanical heat switches.

At bath temperatures around 4 K, there are only a few superconductors that are below their critical temperatures and prove to be effective heat switches. These are Niobium, Lead, and Tin. Below their critical temperatures, these metals are superconductors and their thermal conductivity begins to decrease rapidly with temperature. So, in this state they prove to be effective insulators. To turn the switches "on", one need only apply a magnet field from about 500 to 5000 gauss depending on the material and the temperature. The superconductivity of the metal is then broken, and it becomes a good thermal conductor. It turns out that superconducting heat switches reach their best switching ratios at reservoir temperatures below 1 K, and as a result are not suitable for use in simple ADR design.

Magnetoresistive materials behave in the opposite fashion from superconductors. At low temperatures, they prove to be exceptional thermal conductors, surpassing even copper in thermal conductivity. When a magnetic field of 6000 gauss or greater is applied in a certain direction to the crystal structure of the magnetoresistors, the thermal conductivity drops by several orders of magnitude. The conductivity can be destroyed further with stronger magnetic fields until the thermal conductivity reaches the level of the crystal lattice conductivity. Applying the magnetic field decreases the electronic contribution to the thermal conductivity. Some examples of magnetoresistors are beryllium, gallium, cadmium, and tungsten. More on magnetoresistors will be discussed in section 5.

3.2. Salt Pill

The salt pill provides the cooling power for the ADR. The name comes from the fact that a paramagnetic salt is cased in some sort of container, hence the name salt pill. The salt pill consists of the salt, its housing or container, wires running through the pill, and two thermal contacts connected to the wires inside. One can see a cross-section of a typical salt pill in Figure 3. There are several compounds that can be used for the salt, the most common ones are feric ammonium alum (FAA) and gadalinium gallium garnet (GGG); my work has only dealt with FAA slat pills so I will restrict my discussion to such pills. The housing is typically made of stainless steel; while the wires running through the salt are gold, and the thermal contacts are typically copper, sometimes with a gold coating. I will first describe the growth and construction of a typical salt pill and then discuss its properties and operation (for a more complete discussion of salt pills and their growth, I refer the reader to Wilson).

Professor Timbie's group has a good history of constructing salt pills for various experiments. The technique employed is bery similar to that of other groups, so a review of this groups methods should provide an adequate introduction. One typically begins with a thin walled stainless steel tube. Stainless is used to reduce thermal conductance and eddy current heating. A cap with a gold coated copper extension is welded to one end of the tube, the extension protrudes slightly into the volume of the tube. Many gold wires are soldered to the intruding gold coated extension. These wires are spread out to evenly fill the volume of the tube. Gold wires must be used because FAA corrodes most metals very quickly, but does not corrode gold, so it is very important that no copper be in contact with the FAA. Once the housing is prepared, we fill the volume of the tube with a saturated solution of FAA. The gold coated extension is then put in a bath of ice water. A heating coil is wrapped around the housing to keep the solution from crystalising too quickly. With the copper extension in the ice water, FAA crystals begin to form on the gold wires in the volume of the tube. This is desirable because

FAA is a poor thermal conductor, and the wires are in place to act as the heat transferers. Over the course of several days, the salt pill housing is removed from the heating coil, allowing the FAA to crystalize throughout the volume of the tube. After the tube is filled with crystalized FAA, another cap with a gold coated copper extension is welded to the open end of the tube. Care must be taken in this stage because the desireable properties of FAA are destroyed if it is heated to temperatures above 40 degrees Celsius. After the second cap is welded on, the salt pill is ready for use. Aside from taking care not to heat the salt pill above 40 degrees, one must also be sure that the salt pill housing is vacuum tight, because the salt will dehydrate if exposed to a vacuum, causing it to lose its desirable properties.

The process by which the salt pill cools the experimental space is a simple thermodynamic process. Figure 4 provides a convenient reference for the following discussion. The electrons in the salt begin with their spins randomly orientated, i.e. the system's entropy is high. At this point, the salt pill is thermally connected to the bath. A magnetic field of typically 3 to 4 Tesla and aligned with the long axis of the salt pill is then created around the salt through the use of a superconducting magnet. The application of this field causes the spins of the electrons to align along the direction of the field; this is an isothermal process because it takes place at the constant bath temperature. With the magnetic field at full strength, contact between the salt pill and the bath is broken by the heat switch. The magnetic field is then slowly lowered; as this occurs the electron spins begin to become disordered again. However, the cooling process is adiabatic, i.e. the entropy remains constant, so for the salt's electrons to gain entropy, entropy must be transfered from another part of the system. The only thermal connection the salt pill has is to the experimental space, one's detectors or sample. So, experimet stage loses entropy and as a result it cools to temperatures as low as 50 mK. For a full thermodynamic treatment of adiabatic demagnetization refrigeration, I refer the reader to Reif. With a 100 cc salt pill, one can typically keep experimental stages at a temperature of 100 mK for 24 to 36 hours, depending on heat leaks into the system. The cycling of the refrigerator typically takes 30 minutes.

3.3. Superconducting Magnets

As described above, a magnetic field of 3 to 4 Tesla (note that the earths magnetic field is about 10^{-5} Tesla) is required to align the electron spins of the salt. The only way to achieve this, and the subsequent demagnetization is with a superconducting magnet. Typically, a solenoid is used with the salt pill resting in the bore of the magnet (see Figure 3). Solenoids that can achieve 3 to 4 Tesla are available from a variety of commercial vendors such as American Magnetics, Inc.[2] and Cryomagnetics, Inc.[3]. These solenoids typically require currents of 10 to 20 Amperes. An important part of the magnet system is shielding. The magnet is typically in close proximity to the experimental stage, and the detectors that one may have at the stage are often adversly affected by strong magnetic fields. Hence, it is vital that a shield of some sort be place around the magnet. Professor Timbie's group has had success with shields made of Vanadium Permendur (again see Wilson for a more detailed examination of typical ADR manget systems).

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[2]World Wide Web address http://www.americanmagnetics.com

[3]World Wide Web address http://www.cryomagnetics.com

3.4. ADR Drawbacks

While ADRs may be the most desirable refrigerators to use for many applications, they are not without their drawbacks. As mentioned above, many ADRs make use of mechanical heat switches, which can be unreliable and have poor thermal conductivity in their on state. The reliability factor is especially improtant when one considers the use of ADRs on space based or remote location experiments; if a problem were to develop with the heat switch, the possibility of fixing it is small. The salt pill of a standard ADR also presents several problems. The typical stainless steel housing of the pill has a large heat capacity and adds a significant thermal load to the system, cutting down performance. Also, the large size of the pill requires a long (30 minute) cycling time. Due to the large size of the pill, the superconducting magnet and shield must also be large, which as a result usually dominate the mass of the system. Finally, the thermal isolation of the ADR requires complex suspension by Kevlar©strings as shown in Figure 3. These drawbacks have driven the design and development of a miniature adiabatic demagnetization refrigerator (MADR).

4. Miniature Adiabatic Demagnetization Refrigerator

My work on the MADR began in the summer of 1999 with a University of Wisconsin-Madison Letters and Science Honors Program grant. Professor Timbie suggested that I work on developing a miniature ADR. At that time, we knew only that we wanted to decrease the size of the salt pill and to incorporate everything into a planar geometry. So, I began to work on the components of the MADR with the drawbacks of typical ADRs in mind. In this section, I will describe the novel components and design of the MADR along with theoretical background where needed. Also included in this section will be simulation and experimental results.

4.1. Mechanical Design

The design for the MADR simplifies construction by incorporating all the components onto a planar layer of Pyralux, which is a layer of thin Kapton[4] film with a layer of copper bonded onto it. The Pyralux is stretched accros an aluminum frame which is connected to the thermal bath. A thermal circuit such as the one pictured in Figure 5 is etched from the Pyralux. While the Kapton does link the cold stage to the bath, it is a very poor thermal conductor (conductivities of about 1 μW are expected). The Kapton must be tensioned so that any mechanical oscillations occur at frequencies higher than any detector sampling frequency, which usually do not exceed 50 Hz for applications we are concerned with. We intend to tension the system in a manner similar to that of a snare drum. We will begin with a circle of Pyralux with a diameter of 15cm (corresponding to the length of the MADR). We will then tension it as one tensions a snare drum; once tensioned, we will attach the Pyralux to the aluminum frame and then cut away the excess material. In this way, the Pyralux will be tensioned across the aluminum frame with the same tension achieved by the snare drum method. On can calculate the lowest vibration frequency by this method with the equation

$$f_1 = \frac{0.766}{D}\sqrt{\frac{T}{\sigma}} \tag{1}$$

where D is the diameter of the membrane, T is the tension, and σ is the density. The maximum tension that the Kapton can withstand

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[4] Registered Trademark of DuPont

is the product of the tensile strength and the thickness. The tensile strength of Kapton at cryogenic temperatures is 2.28X10^8 $\frac{N}{m^2}$ and the thickness of the Kapton is 1.27X10^{-4}m. It is reasonable to assume that we can stretch the Kapton to one-half of its tensile strength without fear of it breaking. Using these numbers, we get about 15000 $\frac{N}{m}$. The density of Kapton is 1475 $\frac{kg}{m^3}$. With a thickness of 5mil, we get a lowest mode of vibration at 2200 Hz; which means we can tension the Kapton to much less than its tensile strength. If we want vibrations to be above 100 Hz, we need only tension the Kapton to 71.7 $\frac{N}{m}$.

The simple one salt pill and one heat switch design will be built in the summer of 2001 to test the components and the operation. This prototype will not be an efficient refrigerator because the hold time will only be approxiametly three times the cycling time. Therfore, after the components have all been tested, the continous cooling MADR pictured in figure 6 will be built. This design features two salt pills so that while one is keeping the experimental stage cold, the other is being cycled in preparation. This design allows for the experimental stage to be kept cold as long as the thermal reservoir is available. The operation will be describe in section 4.5.

4.2. Salt Pill

In conventional ADRs, the salt pill and its housing dominate the heat capacity of the refrigerator. As a result, reducing the size of the salt pill was a driving factor in the design of the MADR. After some simulations and design considerations, it was determined that a salt pill with 1 CC of FAA would be sufficient for our purposes. The smaller salt pill allows for faster cycling, smaller housing, and a smaller magnet. The amount of heat that can be extracted from the experimental stage by a certain volume of salt is a function of the specific heat of the salt at different fields and temperatures.

$$Q = MT(S_{tot}(B_{residual}, T_{low}) - S_{tot}(B_{max}, T_{bath})) \tag{2}$$

$$S_{tot} = S(B,T) + S_{lat} \tag{3}$$

$$S(B,T) = R\ln\left[\frac{\sinh\left[(2J+1)\frac{x(B,T)}{2}\right]}{\sinh\left(\frac{x(B,T)}{2}\right)}\right] + R\frac{x(B,T)\coth\left(\frac{x(B,T)}{2}\right)}{2} - R(2J+1)\frac{x(B,T)\coth\left[(2J+1)\frac{x(B,T)}{2}\right]}{2} \tag{4}$$

$$S_{lat} = 3.52\frac{10^{-3}T^3}{3} \tag{5}$$

$$x(B,T) = g * \beta\frac{B}{kT} \tag{6}$$

In the above equations, B is the magnetic field, T is the temperature, M is the number of Moles per CC, T is the temperature in Kelvin, S is the specific heat as a function of magnetic field and temperature, S_tot is the sum of the electronic and lattice specific heats. R is the molar gas constant, J is the spin and angular momentum of a FAA electron, g is the Landau g-factor which is equal to 2, β is a constant equal to $9.27 * 10^{-24}$, and k is the Boltzman factor. Using these equations, we calculate the amount of heat that a 1 CC salt pill can remove from the experimental stage, given a B_{max} of 3 Tesla for bath temperatures from 5.7 to 1.5 K. The results are given in Joules and are pictured in Figure 7. One can see that the refrigerator becomes more efficient at lower temperatures, so it is desirable to pump on the liquid Helium bath to reach temperatures of about 2 K, where the salt pill can remove about 2 mJ.

Because of the small heat removal figure for a 1 CC salt pill, the MADR will not be able to cool the experimental stage to 100 mK in one shot. However, with cycling time of 60 seconds, a single pill would be able to cool the experimental stage in steps. Figure 8 plots the number of cycles required to cool a 100 g copper experimental stage to 100 mK as a function of bath temperature. So, even with a single 1 CC salt pill, it is possible to reach 100 mK. The advanced MADR design featuring two parallel salt pills will be discussed later.

As shown in the figure 5, the salt pill is integrated onto the planar layer of Pyralux. This can be achieved by growing the salt directly onto the Pyralux. One must first etch small traces of copper in the area that the salt pill will be grown; this is important to reduce eddy current heating that occurs in metals due to changing magnetic fields. The copper traces will then be coated with a gold solution to prevent corrosion. The gold coated traces serve the same purpose as the gold wires in a conventional salt pill, the transfer the heat between the salt pill and the cold and hot stages. Once the Pyralux is prepared, the salt can be grown in a small cylinder temporarily attached to the Pyralux as described above. We wish to avoid using a housing with a heat capacity greater than that of the salt pill. Therefore, instead of using a stainless steel housing, we intend to coat the surface of the pill with a layer of GE Varnish (obtainable from Lakeshore Cryotronics, Inc.[5]). The GE Varnish has proven in tests to protect the salt from dehydration in a vacuum, but to be ensure long salt lifetime, we intend to coat the varnish with a thin layer of gold.

4.3. Superconducting Magnets

Conventional ADRs need only use a single solenoid to cycle the salt pill, but due to the MADR's planar geometry, two solenoids will need to be used, one above the Pyralux, and one below with a gap between them for the salt pill. Miniaturizing the magnet system and the associated shielding is one of the main goals of the MADR. Therefore, materials that would be able to increase the field were considered. The first material to consider was obviously iron; however, its ability to boost the field saturates well below 3 Tesla, so other materials were considered. Following Hoard et.al. and

[5]World Wide Web address http://www.lakeshore.com

Schauer, the use of holmium as a flux concentrator was examined. Holmium is a rare earth metal that has ferromagnetic properties and saturates at 3.9 Tesla, ideal for our application. Both Hoard and Schauer show significant in the cental magnetic field of geometries similar to the MADR magnet system. The magnetic field in a gap between two solenoids with holmium poles in their bores is given in Hoard to be

$$H_c = M_s[\cos(\arctan\left(\frac{2R}{g+2L}\right)) - \cos(\arctan\left(\frac{2R}{g}\right))] + H_b \qquad (7)$$

Where H_c is the resultant central field, M_s is the materials constant magnetization value, R and L are the radius and length of the holmium poles, g is the gap between the two poles, and H_b is the applied field. Using 3 Tesla, 1.2 cm, 2.8 cm, and 1.7 Tesla for M_s, R, L, and H_b respectively, the dependence of the central field on the gap size shown in figure 10 is obtained. One sees that the maximum gap size to obtain a 3 Tesla field is aproxiametly 1 cm, which is more than compatible with a 1 CC salt pill in the gap.

After some prelimanary simulations of different solenoids with the set of programs POISSON[6], American Magnetics, Inc. was contracted to build two conduction cooled superconducting magnets. The magnets were wound with superconducting wire on an aluminum bobbin. The magnets have a bore diameter of 2 cm and a winding length of 2.54 cm and a maximum current of 22 Amperes. Given these characteristics, along with holmium magnetization and permiability data from Schauer used for holmium pole pieces 2 cm in diameter and 2.8 cm in length, and a gap size of 1.2 cm, POISSON was used to calculate the magnetic field in the gap. A central field of 3 Tesla was obtained in the simulation with a fall off of less than 10

[6]available as freeware from the Los Alamos Accelerator Code Group, e-mail laacg@lanl.gov

As with the magnet system in a standard ADR, the shielding of the MADR magnets is a very important consideration. The magnets will be less than 10 cm away from the detectors or samples at the experimental stage and as a result must be well shielded. Many of the detectors that the MADR would be used to cool are adversely affected by magnetic fields. The shielding in cunjuction with the holmium pole pieces also provides a return path for the magnetic flux as indicated in the arrows of figure 9. This return path improves the maximum magnetic field attainable by the system. I have completed some simulations of different shielding geometries using POISSON, but have yet to find one that gives the desired 1 Gauss (1 Gauss equals 10^{-4} Tesla) stray field at a distant of 10 cm from the magnet bores. The simulations run have used vanadium permendur and Cryoperm 10[7]. Vanadium permendur has a low permiability and a high saturation field; as a result it is an excellent shield for most of the magnetic field. In the simulations, the vanadium permendur contains all the magnetic field except for several tens of Gauss. This small stray field is then captured in the Cryoperm 10, which has a very high susceptibility and low saturation field, making it ideal for containing small fields. Stray fields of 5 Gauss at 10 cm from the magnet bore have been obtained in simulation, but more design changes are required to reach the desired 1 Gauss.

For the simple one salt pill MADR, two magnet systems will be needed. One for the salt pill and one for the material based heat switch discussed below. The design for the continous cooling MADR calls for 6 magnet systems, which will be quite pricey. Further work must be done to determine if a reduction in the number of magnet systems required is possible.

[7]Cryoperm 10 is a registered trademark of Vacuumschmelze, World Wide Web address http://www.vacuum.de/

4.4. Heat Switch

A great deal of the work done on the MADR has involved the research and development of a material based heat switch. Both mechanical and gas-gap heat switches were immediately ruled out as alternatives due to their large size and the difficulty of incorporating them into a planar geometry. This section will give a brief review of the search for a material to use as a heat switch and the criteria used to evaluate the different options. A theoretical treatment of both superconductivity and magnetoresistivity is included. Finally, I present the results of the search and the accompanying experimental data.

4.4.1. Superconductors

The obvious materials to first consider for a magnetically operated heat switch are superconductors. At cryogenic temperatures, the application of small magnet field can change the thermal conductivity of the material by orders of magnitude. A literature search presented Peshkov and Parshin's excelent review of experimental data for several superconductors at low temperatures. As mentioned in the heat switch section above, most superconducors present excellent on-off conductivity ratios at temperatures below 1 K. For bath temperatures from 2 to 4.2 K, only niobium, lead, and tin present any chance of providing a switiching ratioof at least 100. To determine the power conducted by a heat switch, the equation

$$P = \frac{A}{L} \int_{T_l}^{T_h} \kappa(T) dt \qquad (8)$$

is used. Here, P is the power conducted, $\frac{A}{L}$ is the area-length ratio of the switch, $\kappa(T)$ is the thermal conductivity of the switch as a function of temperature in $\frac{W}{cm*K}$, and the limits of integration are the high and low temperatures that the heat switch connects. The conductivity of most materials at low temperatures follows a power law formula of form

$$\kappa(T) = a * T^b \tag{9}$$

where a and b are material dependent constants. From the conductivity data in Peshkov and Parshin, the constants for Nb, Pb, and Sn in both the on and off states were determined.

For niobium, a is 0.05 and b is 2.4 for the off state below 2 K. Using 1 μW as the maximum power that the switch can allow to leak to the cold stage in the off state[8], and integrating from 0.1 K to 2 K, we see that the $\frac{A}{L}$ ratio must be $3X10^{-6}$ cm. Not only is this area-length ratio physically unfeasible, but such a small and long switch would not be able to conduct much power in the on state. For lead, with a equal to 0.3 and b equal to 2.17, we find that the area-length ratio must be $5.9X10^{-7}$ cm for the same conditions as described for niobium. Obviously then, if we reject niobium we must also reject lead. Tin's constants are a equal to 0.5 and b equal to 3.4. With conditions as above, we find that the area-length ratio must be $1.6X10^{-7}$ cm. While superconductors are feasible for use as heat switches at temperatures below 1 K, they do not constitute an option for the MADR.

4.4.2. Magnetoresistors

Following Dubond, we next considered the use of materials that exhibit magnetoresistive behavior. As mentioned above, at low temperatures magnetoresistors have very large thermal conductivities; the application of a magnet field of order 1 Tesla along a certain direction destroys the electron contribution to the conductivity and results in a drop of several orders of magnitude.

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[8]the heat leak into the system through the Kapton into the system is expected to be 1 μW, this lead us to choose the heat switch leak to be 1 μW so that the Kapton leak does not dominate the system

We examined the possibility of using a beryllium single crystal as the heat switch. Beryllium is a magnetoresistor, and Radebaugh's work indicates that switching ratios of 10^3 can be achieved with an area to length ratio of order 10^{-3}. We were excited about the possibility of using beryllium, but inquires as to the availability of single crystal beryllium with a cross section of about .0025 cm^2 revealed that such a sample is not readily available. Such a crystal would need to be machined from a larger crystal, and we were unable to find any companies that would be able to machine the beryllium due to its dust's carcinogenic nature and the fact that it is not high on the Moh's hardness scale. We next examined the possibility of using single crystal gallium as the heat switch. Engel's et.al. present data that indicates that gallium would be an adequate heat switch with a length of 2 cm and a crossection of 0.0025 cm^2. Unfortunately, gallium melts at 303 K, only a few degrees above room temperature; the fear of the heat switch melting when not cool forced us to abandon the possibility of using gallium. The final magnetoresistor that we considered was tungsten; it should be noted that tungsten does exhibit superconductivity but does not transition until 15 mK, and the superconductivity can be destroyed with a magnetic field of only 10 times that of the Earth's. The results presented in both papers by Batdalov were very encouraging. They showed that switching ratios of 10^4 and greater are possible with tungsten. Inquires into the availability of single crystal tungsten with a square cross-section of .5mm X .5mm and a length of 2 cmwere incouraging. We recieved price quotes for a (100) orientated crystal from Princeton Scientific[9] and from Mr Semicon, Inc.[10] We purchased the single crystal from MR Semicon, Inc. due to its

[9]World Wide Web address http://www.princetonscientific.com

[10]World Wide Web address http://www.mrsemicon.com

slightly lower qoute (850$asoppossedto$995 from Princeton Scientific). The dimensions of the crystal were determined from the data presented in Batdalov and Red'Ko (1980) and shown in figure 11.

As with the superconductors, the desired area to length ratio of the crystal can be determined from the desired heat leak and the thermal conductivity's temperature dependence. For an applied field of 2.2 Tesla, the power law constants for the tungsten single crystal are a equal to 0.0018 and b equal to 1.39. For the .05 X .05 X 2 cm crystal, the heat leak from 2.5 K to 0.1 K is 8.4 μW, which while greater than the 1 μW desired is still an acceptable figure. Given the salt pill's 1.6 mJ capacity at 2.5 K, the hold time of the single pill will be 190 S. With the two salt pill design of the MADR in figure 6 and a salt pill cycling time of 60 S, continous cooling will be achievable. It should be noted that in order for the salt pill to be cycled in 60 seconds, the heat switch in the on state must be able to remove the necessary heat from the salt pill to the bath. So it is necessary to make sure that the area to length ratio of the heat switch is not so small that it inhibits the necessary flow of heat. From equations 2 through 6 above, we can determine the heat that must be remove from the salt pill in a cycle cycling from a starting temperature of 2.5 K and a small field to 3 K and a 3 Tesla field. We use 3 K as the high temperature because the application of the field to the salt typically induces a raise in temperature of 3 K due to eddy current heating. The heat that must be removed is calculated to be 59.2 mJ. This means that in a 60 S cycle, the heat switch must be able to conduct 1 mW of power. From figure 11, we see that the on conductivity of tungsten is nearly a constant 100 $\frac{W}{cm*K}$ at temperatures below 4 K. From equation 8, we find that the .05 X .05 X 2 cm single crystal is able to conduct 62.5 mW, which is more than enough power to cycle the salt pill in 60 S. We must also consider the conductivity of the copper thermal busses between the salt pill and the thermal reservoir. The purity of a material can be expressed in terms of the ratio of the resistance at room temperature and at liquid helium temperature and is known as the residual resistivity ratio (RRR). We determined the RRR of the copper used in Pyralux to be approxiametly 50. Pobell gives data on the thermal conductivity of copper with a RRR of 40 in his figure 3.16. Using this data, we find that the copper obeys a power law with coefficients a equal to 0.53 and b equal to 1.12 below 20 K. Using equation 8 again with P equal to 1 mW, we find that the area to length ratio must equal .0012 cm. With a thermal bus length of 2 cm and the copper thickness of .0127 cm, we find that the width of the thermal bus must be at least 0.2 cm. It should be noted that in the case of the "on" conductivity, the copper does limit the total conductivity of the connection. In summary, the data presented in Batdalov and Red'ko indicates that a tungsten single crystal along with a magnet system that can produce a 2 Tesla field results in an effective heat switch below 4 K.

4.4.3. Magnetoresistance Theory

The magnetoresistance phenomenon was first observed in the early 1900's. Experimenters noticed that the application of a magnetic field perpendicular to the direction of current flow in some metals resulted in an increase in the resistance of the metal. With tungsten, we are concerned with transverse magnetoresistance, which occurs when the current flows in the x-direction and the field is applied in the z-direction. The increased resistance translates to a decrease in the thermal conductivity through the Wiedemann-Franz law which states

$$\kappa\rho = L_0 T \tag{10}$$

Where κ is the thermal conductivity, ρ is the resistance, and L_0 is the Lorenz number which is equal to $2.44 * 10^{-8} \frac{W\omega}{K^2}$. The increase in resistance occurs because the magnetic field causes the moving electrons to spiral around the field lines, which leads to a loss of the electrons momentum in the direction of the current flow and the increase in resistance. For more detailed explanations of the theory see both Wagner and *The Encyclopedia of Physics*.

4.4.4. Experimental Results

While the data presented in Batdalov and Red'ko indicates that a tungsten single crystal with dimensions of 0.05 X 0.05 X 2 cm will be an effective heat switch, we desired to test the thermal conductivity in the off state of the single crystal obtained from MR Semicon, Inc. The testbed was a liquid helium dewar with a liquid nitrogrn shield obtained from Precision Cryogenics, Inc.[11] The two solenoid magnet system from American Magnetics, Inc. was used to produce magnetic field of up to .82 Tesla at the crystal site. The single crystal was soldered between two copper busses and across the Kapton gap of a small piece of Pyralux. Each copper bus was bolted to a copper plate, with a thin layer of vacuum grease applied to increase the area of contact. One copper plate was connected thermally to the reservoir via a copper stand. The other plate was thermally insulated from the reservoir by a G-10 tube. The power conducted by the G-10 tube, approxiametly 10 μW was subtracted from the tungsten power conduction. The Pyralux was stretched between the two copper plates in the gap between the two magnets so that the tungsten single crystal was centered in between the bores of the magnets. For a picture of the setup, see figure 12. A ruthenium oxide thermistor obtained from Lakeshore Cryotrincs,

[11] World Wide Web address http://www.precisioncryo.com/

Inc. was attached to each of the copper plates. Also, a 1050 metal film resistor was attached to the thermally insulated plate. Magnetic field measurements were made using a hall probe placed in the center of the gap between the two magnets and a digital readout box from Cryomagnetics, Inc.

Data was taking by applying voltages of less than 1 V across the resistor, which disipated power into the thermally insulated plate. The steady state temperature reached by the plate at different magnetic fields leads directly to the thermal conductivity of the single crystal at that field. The power dumped into the plate is equal to the square of the voltage applied divided by the resistance. The power conducted by the single crystal is then obtained by subtracting the G-10 and Kapton contributions to the conductance. The thermal conductivity is then

$$\kappa = (\frac{V^2}{R} - P_I)(\frac{L}{\delta TA}) \tag{11}$$

where V is the applied voltage, R is the resistance P_I is the power conducted by the Kapton and G-10, L is the length of the single crystal, δT is the difference in temperature between the insulated and the thermally connected copper plates, and A is the cross-section of the single crystal. Data was taken from approxiametly 2.5 K to 5.5 K at fields of .57, .68, and .82 Tesla. The temperatures below 4.2 K were obtained by pumping on the liquid helium bath. The calculated thermal conductivities at the different fields and temperatures can be seen in figure 13. Comparing the data in figure 13 with the data in figure 11 from Batdalov and Red'ko, we see that the our results compare favorably with Batdalov and Red'ko's. We conclude that the tungsten single crystal obtained from MR Semicon, Inc. will be an effective heat switch at temperatures below 4 K.

4.5. Operation

The simple MADR in figure 5 will be constructed in the summer of 2001 to test all of the components together. It will not be able to achieve any significant refrigeration because of the small size of the salt pill. The continous cooling MADR of figure 6 will be a fully operational refrigeration system that could be used in many applications. The operation and cycling will be controlled by and electronic circuit. Refering to the figure, we note that the 4 different heat switches are numbered; in the following discussion I will be refering to these numbers as well as the upper salt pill as ”salt pill 1” and the lower salt pill as ”salt pill 2.” We begin with all the components in thermal equilibrium with the bath at 2.5 K. The cycling can start with either salt pill 1 or 2, so here lets start with salt pill 1. As we start, heat switches 1 and 2 are open and heat switches 3 and 4 are closed. A magnetic field is applied to salt pill 1, and after it thermalizes with the reservoir, we close heat switch 1 and then slowly ramp down the salt's magnetic field to cool the experimental stage by some small amount. As the field is being ramped down, we begin the cycle for salt pill 2, applying a magnetic field. Once the field of salt pill 1 is zero, all the heat switches are operated, 1 and 4 open and 2 and 3 close. Then, the field of salt pill 2 is slowly ramped down, during which the field around salt pill 1 is brought up. After salt pill 2's magnetic field is zero, all the switches are operated, and we begin the entire cycle again. This is repeated as necessary until the experimental stage reaches the desired temperature. To reach 100 mK from 2.5 K, figure 8 shows that the cycle would only have to occur two times. Once the experimental stage has been cooled to the desired temperature, the salt pills can then be used with small magnetic fields to remove as necessary the energy associated with heat leaks into the system. In this way, the experimental stage can be kept cold as long as there is a thermal reservoir to operate from.

5. Possible Applications

The MADR is being developed in Professor Timbie's lab for use in the detection of small temperature anisotropies in the Cosmic Microwave Background Radiation (CMBR). To detect these small signals, instruments known as bolometers are used. Bolomoters absorb incoming radiation, and the corresponding temperature increase leads to a change in resistance which can be measured with either and applied voltage or current. Many detectors work on similar principles to bolomoters. Bolomoters and other detectors must be cooled to temperatures below 1 K because it is in this region that their resistance most strongly varies with temperature, and very often, the signal trying to be detected is very small. At high temperatures, the atoms in a detector have more energy and therefore move around more and introduce thermal noise. By cooling detectors to low temperatures, the thermal noise can be reduced.

The MADR will be able to be used in applications other than astrophysics. Molecular biology uses cryogenic detectors to study proteins and other molecules via mass spectrometry. Hilton et.al. (1998) indicates that the MADR would be a valuable addition to many labs involved in mass spectrometry. The analysis of semiconductor defects requires low temperature detectors for X-ray microanalysis and could possibly benefit from the use of the MADR, see Hilton et.al. (1999). Finally, thermal neutron detection also requires the use of low temperature detectors, another possible application for the MADR. Thermal neutron detection is important for nuclear waste clean-up, oil recovery, and analytical chemistry, see Richardson et.al. There are many other low temperature detector applications, and the MADR could be of benefit to many of them.

6. Future Work

As mentioned above, there is still work to be done on both the design and construction of the MADR. Following, is a laundry list of tasks and goals.

1. Grow salt pill and construct housing
2. Complete magnetic field simulations to determine shielding geometry and construct shield and holmium pole pieces
3. Obtain magnet system and shielding for magnetoresistive heat switch
4. Integrate components onto tensioned Pyralux for the prototype MADR test
5. Determine if less than six magnet systems are needed for the continous cooling MADR
6. Obtain necessary components for, build, and test the continous cooling MADR

7. Conclusion

The design and data presented above for the miniature adiabatic demagnetization refrigerator shows the feasibility of building a refrigerator that addresses the drawbacks of the systems available to reach temperatures of 50 mK or greater. The simple geometry and setup, indefinite hold time, and lack of moving parts makes the MADR an attractive option for many different applications. The work remaining to be done on the project presents no forseeable hurdles, and a prototype should be completed by August 2001, with the continous cooling MADR not far behind.

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Fig. 1.— A schematic of a recirculating ^{3}He Refrigerator from Betts

Fig. 2.— A simple diagram of a dilution refrigerator from Betts

Fig. 3.— Conventional ADR schematic from Wilson

Fig. 4.— Representation of the alignment of electron spin during different stages of the cycling of an ADR. In the first box, the spins are random and the salt is at the bath temperature. In the second box, one sees the electron spins are ordered, due to a strong magnetic field that aligns the spin in its direction. In the third box, one can see the spins of the electrons after the magnetic field has been reduced, the temperature of the salt is now the "refrigeration" temperature

Fig. 5.— Design for prototype MADR to test components

Fig. 6.— Envisioned final design of MADR with two salt pills for continous cooling.

Fig. 7.— Cooling power of salt pill versus temperature.

Fig. 8.— Number of cycles to reach 100 mK versus starting bath temperature.

Fig. 9.— Schematic of magnet system to be used for salt pills.

Fig. 10.— Plot of magnetic field versus gap size with holmium pole inserts from Hoard et.al.

Fig. 11.— Thermal conductivity of a tungsten single crystal versus temperature and at different magnetic fields from Batdalov and Red'ko. Curve's 1, 2, 3, and 4 correspond to no magnetic field, .595 Tesla, .78 Tesla, and .96 Tesla.

Fig. 12.— Picture of apparatus used to test thermal conductivity of tungsten single crystal. The two superconducting magnets sit above and below a small piece of Pyralux with the single crystal spanning an etched gap. The Pyralux is fastened to two copper plates. One of the plates is thermally connected to the thermal reservoir via a copper stand, while the other is thermally isolated bt a G-10 tube. Ruthenium Oxide thermistors are attached to each copper plate, and a resistor is mounted on the thermally isolated plate to heat it up.

Fig. 13.— Thermal conductivity of single crystal tungsten versus temperature at different magnetic fields with vertical error bars. The crosses, triangles, and squares correspond to measurements at fields of 0.57 Tesla, 0.67 Tesla, and 0.82 Tesla respectively.