Characterisation of the weak thermal link on the Kelvinox dilution refrigerator

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I. INTRODUCTION

In the field of quantum magnetism, temperatures in the miliKelvin range are often required. Although there are many machines capable of this, dilution refrigerators are currently the only device capable of achieving and maintaining temperatures below 300mK. A dilution fridge uses the fact that a phase separation presents in a mixture of liquid ^{3}He and ^{4}He. At this phase separation, it is possible to evaporate the ^{3}He from a concentrated to a dilute phase, leading to a cooling power. This evaporation allows for minimum temperatures of $\sim 1\,K$ and record cooling powers of $\sim 25\,\mu W$ at $10\,mK$ [1].

The downside of dilution refrigerators is that temperature control above 1K temperatures is generally tricky due to the destruction of the two phases in the liquid helium. This paper presents the system designed to overcome these instabilities for the Kelvinox dilution refrigerator at LQM used mainly for AC susceptibility measurements. The solution proposed in this paper is a so called *weak link*, A connector rod which has a poor thermal coupling to the mixing chamber. The desired parameters for the weak-link were a base temperature of $< 100\,mK$ and the ability to heat the weak-link (and thus sample) up to $6\,K$ while keeping the mixing chamber below 1K.

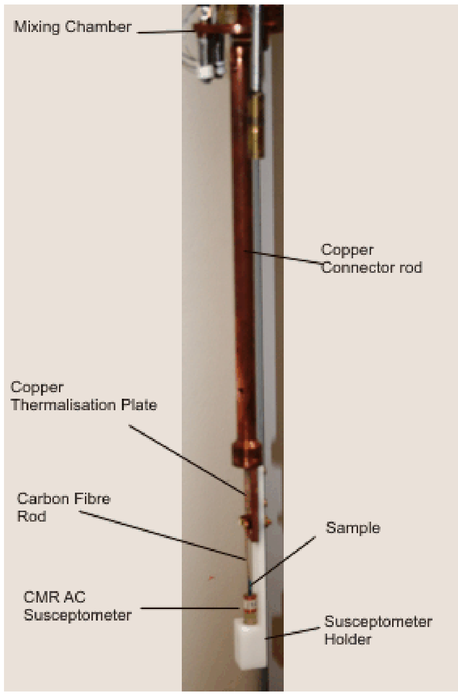

Figure 1: Figure showing the conventional mounting of the sample and A/C susceptometer to the mixing chamber of the dilution fridge. Primarily copper pieces ensure a good thermal contact between the sample and the mixing chamber. Near the susceptometer, delrin plastic is used to reduce noise in the signal.

II. CONVENTIONAL OPERATION OF THE DILUTION FRIDGE

To further illustrate the problem with high temperature control, the current sample holder mechanism as shown in Figure 1 will be briefly described. The operation of a dilution fridge will not be considered as this is already well documented and understood (e.g. [2, p149-188]). As can be seen in Figure 1, the connection between the mixing chamber and the sample consists mainly of a long copper rod known as the *cold finger*. Very close to the susceptometer, plastics are used as, unlike copper, they do not present magnetic moments. The thermalisation between the sample and the cold finger is done with several copper wires, typically $4 \times 100\,\mu m$ wires are used.

Using this kind of thermal connection, the base temperatures achieved near the bottom of the cold finger are the same as those on the mixing chamber ($\sim 60\,mK$). The thermalisation between the two is instantaneous compared to the sample rate of the thermometers ($0.5\,Hz$). These two parameters indicate that the thermal connection is very good and may be considered to be a perfect thermal connection. AC susceptibility measurements have shown that with the exception of several samples, the thermal connection to the sample is also perfect (compared to the measuring rate of the susceptometer).

As mentioned, the problem with this setup is that it is difficult to control the temperature accurately. In fact this is true for all temperature ranges as the cooling power of the fridge is non-linear with the temperature. One problem which seems to be a design flaw of the temperature control system is that the available heating power is separated into separate heating ranges, different by an order of magnitude. The PID controls appear to change the temperature by using a percentage of the current range. This causes an instability at around

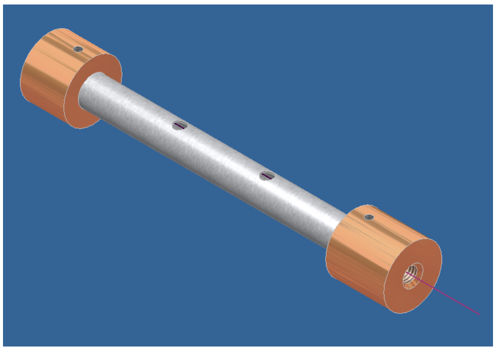

Figure 2: 3D model of the weak link piece. The top copper block (left) is screwed into the mixing chamber and is connected using a stainless steel tube to the bottom copper block.

$200\,mK$, where it is necessary to change the heater range from $200\,\mu W$ to $2\,mW$. The effect is that once the range switches to the higher of the two, the temperature essentially oscillates around the desired temperature by up to $100\,mK$ and takes $\sim$ 10 minutes to stabilise. Another instability of this nature occurs at a temperature of $\sim 1.2\,K$, once again due to the change in heater ranges. The final problem is achieving temperatures of greater than $\sim 1.5K$. At this temperature, the heating power delivered by the system is insufficient to change the temperature. The system will sit at $1.5\,K$ for up to 1 hour until effectively all of the helium in the mixing chamber has been evaporated. At this point in time, the temperature will suddenly jump, often to temperatures greater than $4\,K$ in several seconds.

III. WEAK LINK DESIGN

To eliminate these three instabilities and to generally improve temperature control, the weak link was devised. The basic concept for the weak link is to have two reasonably large thermal masses attached by a medium which conducts heat poorly. Thus the design was to connect two copper blocks with a thin walled stainless steel tube. The initial design is shown in Figure 2. In addition to the steel tube, several copper wires are used to attach the two copper blocks, increasing the thermal link to the desired range. The weak link must be able to on the one hand keep the sample at 6 K while the mixing chamber stays at ~1 K. On the other hand, the thermal coupling must be good enough so that the sample can thermalise at low temperatures to achieve a base temperature of less than 100 mK. The required number of wires for the desired thermal link is studied in detail in later sections.

One unforeseen problem with using a weak thermal link is the heating caused by eddy currents while changing the DC magnetic field. It was found that while testing the weak link that this heating effect meant that instead of ramping rates typically in the range

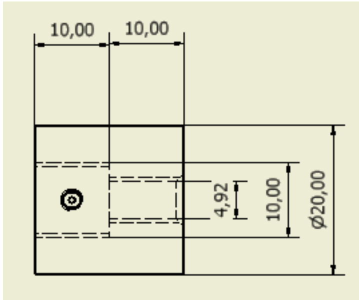

Figure 3: Schematic of the lower copper block of the weak link (all distances shown in mm)

of $0.01 - 0.1\,T/min$, only ramping rates of less than $0.003\,T/min$ could be used while at base temperature. This very slow ramping rate would mean that every single field scan measurement would take ten times longer, thus the weak link would need to be modified.

To modify the weak link, it is necessary to understand the best way of minimising the heat generated while keeping a relatively large thermal mass at the same time. The heat generated by eddy currents in a solid material is given by [2, p234]

$$\dot{Q}_e = \frac{GV\dot{B}^2}{\rho}, \tag{1}$$

where ρ is the electrical resistance, V is the volume of the material, $\dot{B}$ is the field change rate and the geometry factor G is

$$G = \begin{cases} r^2/8 & \text{for a cylinder with radius r,} \\ (d^2/16)[k^2/(1+k^2)] & \text{for a rectangle of width } w \text{ and} \\ & \text{thickness } d \text{ where } k = w/d. \end{cases}$$

Using this equation, it is possible to calculate the heat generated by the lower copper block in the weak link. The dimensions used for the copper block are shown in Figure 3 and give a volume of $5.3 \times 10^{-6}\,m^3$. Taking the electrical resistivity of copper to be $8 \times 10^{-12}\,\Omega m$ (20 K value [3, p90]) and using a field ramp rate of $0.1\,T/min$, the eddy current heating is calculated to be $23\,\mu W$. This number is comparable to the cooling power of the fridge at 100mK, indicating that it would not be possible to do field sweeps below this temperature at such a rate. An additional problem is that the heat flow between this copper block and the weak link is relatively small. This implies that the temperature of the sample will be much higher than the value calculated. The effect of this problem along with a possible solution will be explored in detail in Section IV D.

IV. CHARACTERISATION OF THE WEAK LINK

A. Weak Link with no wires

To determine the correct number of wires needed to connect the two ends of the weak link, it is first necessary to determine the thermal connection of the stainless steel connection itself. The easiest way to do this is to mount a thermometer and a heater to the lower copper block and to heat it while monitoring the mixing chamber temperature. The desired temperatures are $\sim 6\,K$ on the weak link thermometer while keeping the mixing chamber around 1K. At the same time, the thermal connection must be good enough so as to have a reasonable base temperature. For a reasonable base temperature, a temperature of $< 100\,mK$ is desired.

When cooling down the weak link, it was found that with a mixing chamber base temperature of 55 mK, the weak link temperature was 170 mK, indicating the necessity for copper wires. The weak link was then heated to a temperature of 5-6 K (the exact figure is uncertain as the thermometer is only calibrated up to 4K). This resulted in the mixing chamber stabilising at a temperature of 450 mK. As the required mixing chamber heating powers to maintain the mixing chamber at a certain temperature can be easily read off from logs, it was possible to determine the maximum heat load. Calculations showed that a further 1.5 mW of heat could be supplied to the mixing chamber through the weak link while maintaining a mixing chamber temperature of 1 K.

B. Calculating the required number of wires

As the weak link on its own doesn't allow a large enough heat flow, copper wires must be used to connect both ends. The heat flow across a temperature difference per unit length $\frac{dT}{dx}$ along a material with temperature dependent thermal conductivity $\lambda(T)$ and cross section A is given by [4]:

$$\dot{q}_{cond} = \lambda(T) A \frac{dT}{dx}$$

If the cross section A is uniform, then this equation can be intergrated to give:

$$\dot{q}_{cond} = \frac{A}{L} \int_{T1}^{T2} \lambda(T) dT$$

Using this equation it becomes possible to determine the heat flow through a copper wire of a given diameter. To make the process of attaching the wires more simple, it was decided that 100 μm copper wires would be used to make the thermal connection across the weak link. As has been mentioned, it is desirable to have the largest number of wires while still allowing for the mixing chamber to be in the region of 1K while heating the sample up to 6K. Using a linear form for the thermal conductivity, $\lambda(T) = x \times T$, where the limits of x were found by fitting experimental data [5] to be $350 < x < 1800$ depending on the purity of the copper. With these two values of thermal conductivity, it is found that a single copper wire with one end at 1 K and the other at 6 K will have a heat flow of $500\,\mu W - 2.5\,mW$.

The other desired factor in the performance of the weak link is the ability to cool the sample down below 100 mK. Assuming the mixing chamber to be at 60 mK and the sample at 100mK, the cooling power flowing through the wires is calculated to be $450 - 880\,nW$ per copper wire. As a cooling power in the range of a few microwatts is required to effectively get below 100 mK, in this case, it was decided that the weak link should be tested while using $4 \times 100\,\mu m$ wires.

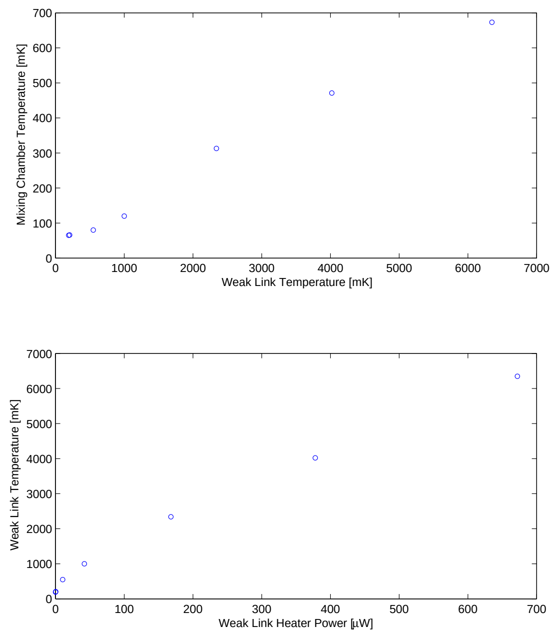

Figure 4: Graphs showing the mixing chamber temperature as a function of weak link temperature (top) and the weak link temperature as a function of heating power applied (bottom)

C. Weak Link with $4{\times}100\,\mu$m wires

With the four wires screwed onto the copper blocks of the weak link and a thermometer and heater (42 Ω wire) attached to the lower block, the following results were obtained. First, the best base temperature acheived on the mixing chamber was $54.5\,mK$, which corresponded to a temperature on the weak link thermometer of $75\,mK$.

Figure 4 shows the heating power required for different weak link temperatures along with the resulting mixing chamber temperature. From these graphs, it is clear that the thermal conduction from the wires is worse than was initially expected. This is most likely due to the thermal contact between the wires and the copper blocks of the weak link. These results also demonstrate that a larger number of wires could be used to connect the two ends of the weak link while maintaining the desired parameters. It should be possible to use four 200 μm wires instead of 100 μm ones while still keeping the mixing chamber in the region of 100 mK. This would be beneficial as the 200 μm wires are much easier to work with and less susceptible to damage. The second important result to take from Figure 4 is that the cooling power across the copper wires with the mixing chamber at base temperature is very low. This also suggests that larger (or more) wires should be used to increase the possible cooling power.

Figure 5: Photo of the modified lower copper block of the weak link. The solid block has been cut into strips along most of the length to reduce eddy current heating.

D. Eddy Current Heating Effect

Using the same experimental configuration as above, with mixing chamber and weak link temperatures of 100 mK and 115 mK respectively, the field was ramped at rates from 0.001 - 0.01 T/min to see if the eddy current heating was a problem. With the slowest scans (0.001 - 0.002 T/min) the heating was negligable. On the other hand, as soon as the field ramp rate was higher than 0.002 T/min, the heating became noticable and the weak link temperature started to rise. With a ramp rate of 0.005 T/min the heating was so large that the temperature of the weak link rose above 500 mK. This clearly shows that even at very slow ramping rates, the heating produced by eddy currents in the copper is far too large.

Eqn.1 indicates that there are only three parameters which can effectively be changed: the volume, the geometry or the material (electrical resistivity). As we require a good thermal connection between the sample and the lower part of the weak link, the material must be a good thermal conductor (which at low temperatures makes copper the best choice). As a large mass is required the volume cannot be considerably changed. Thus the only parameter which can be changed is the geometry. This is done by cutting the copper block effectively into strips, thus reducing G dramatically. This piece was then reinforced using Stycast 1266 and additionally the cylinders were cut to give flat faces. The modified weak link is shown in Figure 5.

Once again the eddy current heating was calculated for this new form of copper. It is somewhat interesting to note that in this configuration, 99% of the heating produced is due to the solid copper piece (at the top of the block) which is roughly half the volume of the total piece. For this calculation, the transversal holes in the solid part of the copper were ignored, implying the heating is slightly over estimated. The heating was found to be 2.86 μW for the same field ramping rate of 0.1 T/min.

This factor of 10 in decreased cooling power should threoretically allow for the sample to be kept at 100 mK while ramping the magnetic field at a rate of 0.1 T/min. As has already been shown, using four 100 μm copper wires to bridge the weak link would give a cooling power of $1.8 - 3.5\,\mu W$. This should therefore be sufficient for field scans to be carried out at the desired rate of $0.01 - 0.1\,T/min$ at any temperature between 100 mK and 6 K. As was seen earlier however, this is most likely not the case in reality due to the heat flow across the wires being much less than the theory would suggest.

V. CONCLUSIONS

In this paper, we set out to create a simple weak link to allow samples to be heated up to 6 K while keeping the dilution fridge running noramlly. The initial design of the weak link was found to give an insufficient thermal conductivity and was thus increased using four 100 μm copper wires. This was found to improve the performance of the weak link - allowing for a base temperature of $\sim 75\,mK$ compared to $\sim 175\,mK$ without the copper wires. In addition to this if the sample side of the weak link was heated up to 6 K, the mixing chamber remained at a temperature $< 700\,mK$. This implies that the number of copper wires could be further increased, possibly even to $4 \times 200\,\mu m$ wires.

An unforseen problem with the weak link design was that ramping the field caused considerable eddy current heating in the copper. This heating was calculated to be 23 μW when ramping the field at a rate of 0.1 T/min and resulted in making field scans near impossible on an acceptable time scale. To solve this, the weak link was modified by effectively cutting the copper block into strips of copper. The effect of this is that the eddy current heating should be reduced to $< 3\,\mu W$ for the same ramping rate.

Although the initial results of the weak link are promising, further experimentation needs to be carried out. First, the modified weak link must be tested to check whether the eddy current heating has been removed to a large enough extent. Secondly the weak link should be bridged with more wires as the base temperature heat flow is still extermely low. Ideally the weak link should have enough wires so that the effect of heating the weak link to 6 K heats the mixing chamber up to 1 K.

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