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Measurements of Copper Heat Straps Near 4 K With and Without Apiezon-N Grease

A. R. Kerr and R. Groves

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Abstract: This note describes measurements of the thermal resistance of heat straps used in the ALMA Band 6 cartridges. The results suggest that using Apiezon-N grease in bolted joints between gold plated copper parts is advantageous when the contact area is large ($\sim 10 \text{ cm}^2$) but that grease actually increases the thermal resistance when the contact area is small ($\sim 0.5 \text{ cm}^2$). An explanation is suggested.

1. Background

The thermal resistance of a heat strap depends on its material and geometry, and also on the connections at its ends. While the designer of a heat strap can take account of the material and geometry quite well, the contribution of the end connections is not easy to predict, and it is therefore necessary to measure the thermal performance of a heat strap of a given design before it enters production.

The material of choice for cryogenic heat straps is copper, either OFE (C10100) or OF (C10200), both of which were formerly known as OFHC. When annealed, these materials have very high thermal conductivity, with a maximum in the temperature range 8-30 K, depending on the residual resistivity ratio (RRR) [1]. The RRR depends on physical defects caused by work hardening and on the chemical purity. Below ~ 10 K, annealing can increase the thermal conductivity by more than an order of magnitude, depending on the purity and original degree of work hardening, but above 60 K there is little to be gained by annealing as the thermal resistance is dominated by electron-phonon interactions rather than electron interactions with physical defects or impurities.

The ends of a heat strap are usually either bolted or soldered to the cold plate and to the object being cooled. Several types of bolted joints were evaluated in [2] where it was shown that gold plated copper surfaces were superior to bare copper or bare copper with indium, and that a layer of indium in gold plated joints was of no benefit. More recently, Salerno *et al.* [3] have advocated using Apiezon-N grease [4] use in thermal joints to reduce thermal resistance. The benefits of using Apiezon-N in bolted joints at the ends of gold plated copper heat straps are explored here.

It is sometimes convenient to solder a heat strap to a component, or to use solder to assemble multi-piece heat straps. Sn/Pb solders are desirable for their physical properties and their compatibility with copper and brass. However, a concern is that Sn/Pb alloys become superconducting below $\sim 8 \text{ K}$ [5]. As a superconductor is cooled below its critical temperature, the density of free electrons decreases with a corresponding decrease in thermal conductivity, and opinion is divided as to whether Sn/Pb solders are appropriate for heat straps operating $\sim 4 \text{ K}$. Fig. 15 of Radebaugh *et al.* [6] shows the thermal conductivity of "Pb-Sn solder" at 4 K to be about 5% of its room temperature value.

In the present work, the thermal resistance of two heat straps designed for the ALMA Band 6 cartridge was measured. The heat straps include Sn/Pb/Ag 62/36/2 solder joints and bolted connections, and they are measured with and without Apiezon-N grease in the joints.



Fig. 1. The arrangement for measuring heat strap #1. The left hand end of the heat strap is bolted to the copper block bearing temperature sensor B, which is bolted to the cold plate next to temperature sensor C. The right hand end of the heat strap is bolted to the upper copper block bearing temperature sensor A and the heater.



Fig. 2. The arrangement for measuring heat strap #2. The left hand end of the heat strap is bolted to the copper block bearing temperature sensor B, which is bolted to the cold plate next to temperature sensor C. The right hand end of the heat strap is soldered to the finger projecting from the flag-shaped copper piece bearing temperature sensor A.

2. Experiment

The heat straps and the arrangement for measuring their thermal resistance is shown in Figs. 1 and 2. In each case, the right hand end of the heat strap is attached to a heated copper block. The left end is attached to another copper block which is bolted to the cold stage of the refrigerator. Temperature sensors A, B, and C measure the temperatures of the heated block, the cold block, and the cold plate of the refrigerator, respectively. With the refrigerator cold, temperature readings were taken at a number of heater power levels. After each power adjustment, the system was allowed to stabilize for 15 minutes. Initial measurements were made without using grease, and subsequently the measurements were repeated with Apiezon-N grease applied between the refrigerator cold plate and the cold block, between the heat strap and the cold block, and, in the case of heat strap #1, between the heat strap and the heated block.

3. Results

The main quantity of interest is the incremental (differential) thermal resistance of the heat strap including its end connections, which is a function of temperature. This is calculated as

$$R_{P_N} = \frac{\left(T_A - T_B\right)_{P_N} - \left(T_A - T_B\right)_{P_{(N-1)}}}{P_N - P_{(N-1)}} ,$$

where T_A and T_B are the readings of temperature sensors A and B, and P_N and P_{N-1} are consecutive heater power settings. Taking the difference of the temperature readings at each sensor removes the uncertainty due to the offset errors of the temperature sensors (±0.25 K for Lakeshore DT-670A Si diode sensors at ~4 K). All measurements were made with a refrigerator base temperature of 3.4 ± 0.25 K with the heater off. With the 0.5 W maximum heater load, the refrigerator base temperature increased by 0.5 K.

The measured results are shown in Figs. 3-6 for the two heat straps with and without Apiezon-N grease. The left hand graphs 3(a)-6(a) show the total temperature change produced by the heater between the different pairs of temperature sensors:

for the curves labeled Heat strap,

$$\Delta T|_{P} = (T_{A} - T_{B})|_{P} - (T_{A} - T_{B})|_{P=0},$$

for the curves labeled Cold end to refrig., $\Delta T|_P = (T_B - T_C)|_P - (T_B - T_C)|_{P=0}$,

for the curves labeled Hot end to refrig., $\Delta T|_P = (T_A - T_C)|_P - (T_A - T_C)|_{P=0}$,

and for the curves labeled Refrigerator,

$$\Delta T|_P = T_A|_P - T_A|_{P=0}.$$

The right hand graphs 3(b)-6(b) show the incremental thermal resistance R, as defined above, between the different pairs of temperature sensors.



Fig. 3. Heat strap #1, three copper strips soldered together at one end and bolted to the hot and cold blocks, as shown in Fig. 1. No grease. (a) Temperature change vs heater power. (b) Incremental thermal resistance.



Fig. 4. Same as Fig. 3, but Apiezon-N grease used in the bolted joints. (a) Temperature change vs heater power. (b) Incremental thermal resistance.



Fig. 5. Heat strap #2, a single copper strip attached to the hot plate with 60/40 solder and bolted to the cold plate, as shown in Fig. 1. No grease. (a) Temperature change vs heater power. (b) Incremental thermal resistance.



Fig. 6. Same as Fig. 5, but Apiezon-N grease used in the bolted joints. (a) Temperature change vs heater power. (b) Incremental thermal resistance.

4. Discussion

The thermal resistance results of Figs. 3-5 are summarized in Fig. 7(a) and (b). It is clear that the incremental thermal resistance of the heat straps varies substantially with heater power and the temperature T_{hot} of the hot end of the heat strap. This is consistent with the temperature dependence of the thermal conductivity of copper and Sn/Pb solder, both of which increase with increasing temperature in this temperature range.



Fig. 7. Summary of the incremental thermal resistance measurements on the heat straps with (pink curves) and without (brown curves) Apiezon-N grease: (a) the multi-layer heat strap, and (b) the single-layer heat strap. The contact areas of the bolted joints are between 0.04 and 0.16 in² (0.25 and 1 cm²) – see Figs. 1 and 2.

A surprising result is that the use of Apiezon-N grease in the bolted joints of the heat straps actually *increases* their thermal resistance. For the single-layer heat strap, the increase in thermal resistance caused by the grease is about 1 K/W, and for the multi-layer heat strap the increase is about 3 K/W. This difference is consistent with the different number of bolted joints on the two heat straps: both have two parallel screws at the cold end, while the multi-layer strap has a single screw at the warm end and the single-layer strap has none (it is soldered).

It is also of interest to look at the thermal resistance of the cold block to refrigerator interface. This has four bolts and was measured with and without Apiezon-N grease. The results for two cold blocks are shown in Fig. 8. It is clear that the grease has reduced the thermal resistance of these joints. This apparently contradicts the results in Fig. 7 but is explained below.

While further measurements are needed to obtain a quantitative understanding of when Apiezon-N grease is advantageous in a bolted thermal joint, a simple explanation exists to explain the above apparently contradictory results. Consider first a clean metal to metal joint. According to Rose-Innes [7] "the thermal resistance between two bodies [in a vacuum] is independent of their area of contact but is inversely proportional to the force pressing them together." This is a result of the actual contact being made at just a few points with a contact area much smaller than the apparent contact area. As the force is increased, the material around the contact points deforms to support it with a contact area proportional to the force, to first order. The rest of the apparent contact area is a vacuum gap which provides no thermal conduction. It is well known that even a small amount of surface oxide or contamination can greatly increase the thermal resistance of a joint, and in the present case one effect of the Apiezon-N grease is to degrade the thermal contact at the points of actual contact. At the same time, the grease fills the voids and provides some conduction where there was none without it. For relatively small contact areas, such as those at the ends of the two heat straps, the reduction in thermal resistance due to grease filling the voids is less than the increase caused by grease at the actual contact points, and the refrigerator in these measurements, the reduction of thermal resistance due to grease filling the voids is destined areas, such as that between the cold block and the refrigerator in these measurements, the reduction of thermal resistance due to grease filling the voids is grease to points and the

grease reduces the overall resistance. Fig. 9 shows a thermal equivalent circuit of a bolted joint with grease.

These results appear contrary to those of Salerno *et al.* [8] who find that Apiezon-N grease reduces the thermal resistance of joints between gold plated copper components at 4 K by almost an order of magnitude. He also finds that the thermal resistance of a single contact between gold plated copper surfaces is larger than that of our complete three layer heat strap including the end connections. However, the conditions of measurement were very different. Salerno's measurements were made on thick cylindrical samples with a contact area of 82 mm². These were pressed together axially with a maximum force of 670 N – an average pressure of 8.2 N/mm². In comparison, our heat straps were of thin copper (see Figs. 1 and 2), bolted at their ends with M2 screws and washers of 4 mm diameter. The force applied by the screw was therefore distributed over the 12.6 mm² area of the washer less the 4.5 mm² screw hole, a total area of 8.1 mm². The tensile strength of the M2 screws is 2700 N. Assuming the screw is tightened to one third of its tensile strength, the average pressure would be 111 N/mm², which is 13 times greater than the maximum used in Salerno's measurements. The rule that the thermal resistance of a joint (in a vacuum) is proportional to the total force applied and independent of the area will not apply to a joint containing grease, in which case the area would be expected to be a factor. We conclude that Salerno's results are not in conflict with ours.



Fig. 8. Thermal resistance of the cold block to refrigerator connection with and without Apiezon-N grease: (a) the multi-layer heat strap, and (b) the single-layer heat strap. The area of the joint was 1.7 in² (1.1 cm²) in each case.



Fig. 9. Thermal equivalent circuit of a gold-to-gold contact with grease. Without grease, in a vacuum, $R_{GrAu} = 0$ and R_{GrGep} is infinite.

Figs. 3-6 also give the thermal resistance of the refrigerator itself. It is close to 1 K/W for heater powers up to 0.5 W, the highest power used in these experiments.

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6. References

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[7] See [5], p. 91.

[8] See [3], Fig. 12.