Thermal conductance measurements of bolted copper to copper joints at sub-Kelvin temperatures

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We have measured the thermal contact conductance of several demountable copper joints below 1 K. Joints were made by bolting together either two flat surfaces or a clamp around a rod. Surfaces were gold plated, and no intermediate materials were used. A linear dependence on temperature was seen. Most of the measured conductance values fell into a narrow range: 0.1 to $0.2~{\rm WK}^{-1}$ at 1 K. Results in the literature for similar joints consist of predictions based on electrical resistance measurements using the Wiedemann-Franz law. There is little evidence of the validity of this law in the case of joints. Nevertheless, our results are in good agreement with the literature predictions, suggesting that such predictions are a reasonable approximation.

Keywords: contact conductance, copper, sub-Kelvin

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1. INTRODUCTION

If two solid surfaces are pressed together, a thermal resistance will exist at the boundary. This is believed to be due to the fact that any surface will be rough on a microscopic scale, causing the true area of contact to be considerably smaller than the nominal surface area. This contact resistance can be a serious problem in cryogenic systems, particularly at ultralow temperatures where thermal conductivities are low and cooling powers often limited.

A further complication is the possibility of contamination between the surfaces, examples of this being grease, oxide layers on metals, and frozen air. At low temperatures, the conductivity of a dielectric is generally much lower than that of a metal. In addition, a thermal resistance occurs even for two surfaces in perfect contact with each other if one or both is a dielectric. This effect, sometimes referred to as Kapitza resistance [1], is due to acoustic mismatch of the phonons in the two materials in contact. Both these effects become very large at temperatures below 1 K. Therefore even a thin layer of a dielectric material sandwiched between metal surfaces can cause a severe reduction in the joint conductance if it is sufficient to prevent direct metal to metal contact.

Predicting the conductance for a given contact is not straightforward. The actual contact area will depend both on the roughness at the microscopic scale and on the overall surface flatness. Moreover, since the contact points will deform under pressure, increasing the contact area, the conductance will also depend on the force used to press the surfaces together [2–4]. This has the consequence that conductance does not scale with the nominal surface area; it is therefore generally not appropriate to quote conductance per unit area.

While models do exist, predicting absolute values for a given joint is generally not possible (see, for example Ref. [5]) and we must turn to experimental data. Unfortunately, the number of measurements reported in the literature is some-

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what limited. The largest quantity of information on low temperature contacts [6] relates to joints between two copper surfaces, which are usually gold plated. Gold plating prevents the formation of the oxide layer which forms on bare copper, and may play a role in improving the actual contact area since it deforms more easily than copper.

Several papers describe measurements on joints designed for systems to be operated at temperatures below 1 K. These joints consist of direct metal to metal contacts, with pressure between the surfaces being obtained either by screws or differential thermal contraction during cooling.

However, the majority of these results were *not* obtained by measuring the thermal conductance of the joints. Instead, the electrical resistance was measured near 4 K, and the thermal conductance estimated using the Wiedemann-Franz law [7]. This law is known to work extremely well in the case of bulk copper, but there is little evidence of this in the case of joints.

The satisfactory operation of cryogenic systems using these joints shows that the predictions must be accurate to some degree. Nevertheless, there is a lack of direct measurements of the thermal conductance. We have also been unable to find any measurements (electrical or thermal) on joints formed by bolting a clamp around a rod, despite this being a convenient configuration for some applications.

In this paper we report direct measurements of the thermal conductance of several different copper to copper joints at temperatures below 400 mK. These measurements were made as part of a development program for the SPIRE instrument [8] on the Herschel space telescope. The joint designs were based on the specific engineering requirements of the instrument.

No materials were deliberately placed between the contacting surfaces. Introducing a metallic substance such as silver loaded grease or indium (above its superconducting transition temperature) has been found in some cases to produce a modest improvement in conductance at low temperatures [6]. However, such a configuration was not used due to concerns about its reliability in a space environment.

2. MEASUREMENTS

The samples were cooled using a ³He sorption fridge [24]. Under the heat loads required to create a measurable temperature difference across the joints, the stable operating temperature of the fridge was limited to approximately 335 to 450 mK. This was acceptable since the joints were designed to be ultimately used near these temperatures. However, the temperature variation of the conductance cannot be determined from such a limited temperature range. For this reason, one sample was also measured in a paramagnetic salt adiabatic demagnetisation refrigerator (ADR); this enabled measurements to be made down to temperatures below 100 mK. Exchange gas was not used during the cool-down for either refrigerator. Samples were surrounded by a radiation shield thermally anchored at 1.6 K in the case of the ³He fridge, and at the sample temperature for measurements in the ADR.

The samples were connected to the fridge cold plate via one or more bolted joints in series. Measurements were made using the "two heater" technique. With this method, a heater is connected to the "cold end" of the joint – the side which is heat sunk to the fridge. A further heater and a thermometer are mounted on the the other side – the "hot end".

To take data, the thermometer temperature is measured as the power to the hot end heater is varied. To ensure that the cold end temperature remains constant, the power to the cold end heater is adjusted to ensure that the total power dissipated in the sample is constant. The power across the joints between the cold end and the fridge cold plate then remains constant, as does the temperature gradient across these joints. Power levels to the hot end heater were varied in at least four steps between 0% and 100% of the total power level. During measurements, the fridge cold plate temperature was monitored using a separate thermometer to check that it remained at a constant temperature.

This approach has several advantages over the more conventional approach of using a thermometer on each side of the sample. Firstly, by measuring a temperature difference using a single thermometer, calibration errors will to some extent cancel out, as will any excess constant heating of the sample such as that due to radiation. Secondly, keeping the cold end temperature constant simplifies the analysis. Without this heater, the temperature of the cold end would rise as the power was increased due to the thermal resistance between the sample cold end and the fridge cold plate. Thirdly, it reduces the number of thermometers required; heaters are cheaper than calibrated thermometers, and it is easier to make accurate measurements of the dissipated power than it is to measure temperature. (It is true that a further thermometer was used to monitor the fridge cold plate temperature; however, this was not removable from the cold plate, and served more than one sample since several samples were measured during a single cool-down).

Electrical connections to the heaters and thermometers were made using $100~\mu m$ diameter manganin wire, thermally anchored to the body of each heater or thermometer. The only unwanted thermal path to the hot end of the samples was via these wires, radiative power from the radiation shields being

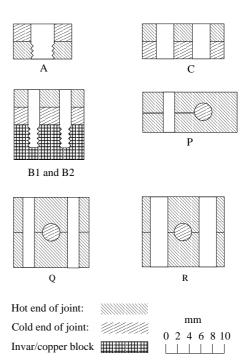


FIG. 1: Cross-sections of the different samples.

Sample	Type	Plating	Area	Screws
A	Flat	Au	87 mm^2	M 4 ×1
B1	Flat	Au/Ag	$68~\mathrm{mm}^2$	$M2 \times 2$
B2	Flat	Au	$68~\mathrm{mm}^2$	$M2 \times 2$
C	Flat	Au/Ag	$159\;\mathrm{mm}^2$	$M3 \times 2$
P	Clamp	Au	94 mm^2	$M2 \times 1$
Q	Clamp	Au	94 mm^2	$M2 \times 2$
R	Clamp	Au	$132\;\mathrm{mm}^2$	$4\text{-}40 \times 4$

TABLE I: Sample details. For the flat samples, the area given excludes the area occupied by the screw holes. For the rod and clamp samples, the area is the surface area of the rod over the length within the clamp.

negligible. The maximum heat leak down these wires is estimated as 0.18 μ W; this is negligible compared with the minimum heater power to the sample hot end of 30 μ W. Calibrated commercial germanium semiconductor thermometers [25] were used for the majority of measurements, with some measurements being carried out using neutron transmutation doped germanium thermometers [9, 10] calibrated against the commercial thermometers.

3. SAMPLES

Details of the different joints measured are given in Table I and Fig. 1. All samples were manufactured from commercial (electrolytic tough pitch) copper; the surfaces were as machined. All samples were plated with gold to a thickness of approximately 2 μ m; in some cases (noted in Table I) the gold was applied over an approximately 5 μ m thick layer

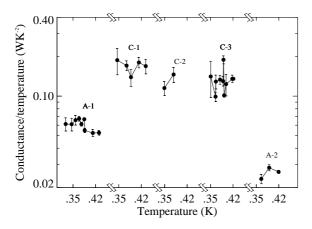


FIG. 2: Results from joints A and C, labelled by run.

of silver. Stainless steel screws were used for all the joints. Stainless steel contracts less than copper as the temperature is reduced, possibly leading to a reduction in contact pressure for the joints. To mitigate this effect, Belleville washers were used in most cases as shown in Table II.

Two types of joint were tested. Samples A to C were formed from two flat surfaces bolted together, while joints P to R each consisted of a clamp which was tightened onto a 3 or 3.2 mm diameter rod. For joint P, the clamp was a single block of copper. A slit of approximately 1 mm thickness allowed the block to be pressed onto the rod. Joints Q and R consisted of two blocks bolted together around the rod.

For sample A, one side of the joint contained a threaded hole into which the screw was tightened. The remaining joints had clearance holes on both sides. For samples B1 and B2 screws were secured by screwing into tapped holes in a copper or invar block (see Fig. 1). Stainless steel nuts were used on the remaining samples.

Joints were cleaned in acetone in an ultrasonic bath before assembly. The screws were tightened using a torque screw-driver. The screwdriver used for some of the measurements was not calibrated below 50 N cm; therefore for these results only an upper limit for torque can be given.

4. RESULTS

The measured conductance at a given cold plate temperature was taken from the relationship between the hot end temperature and the heater power. For each cold plate temperature, a linear dependence was seen; this provides a good consistency check on the results.

Good agreement (i.e. within the error bars) was seen in measurements made on the same sample over several days, during which time the ³He fridge or ADR stage was warmed up to 4 K and back down to the operating temperature. Similar agreement was seen if the cryostat was thermally cycled to room temperature between measurements.

Sample C was measured on two separate occasions (runs

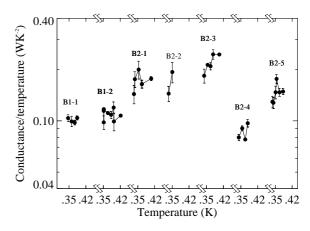


FIG. 3: Results from joints B1 and B2, labelled by run.

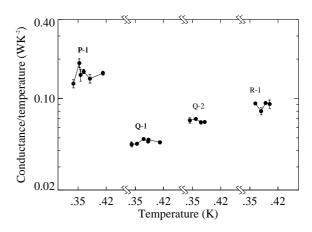


FIG. 4: Results from joints P, Q, and R (³He fridge measurements only), labelled by run.

C-1 and C-2) without the joint having been broken apart; again, good agreement was seen.

A further check was made by measuring a beryllium copper strip with a known thermal conductance. Two measurements were made; the results agreed with each other and with previous measurements [11] to within 10%. The error is most likely to be due to calibration errors in the thermometers; different thermometers were used for the different measurements.

The configuration used for each "run" is given in Table II; a run consists of a series of measurements taken on a given sample without opening the cryostat. The conductance values measured with the ³He fridge are shown in Figures 2 to 4, and with the ADR in Fig. 5; the results are summarised in Table II. Note that ³He fridge results are plotted as reduced conductance (conductance/temperature), assuming a linear dependence of conductance on temperature.

Sample	Run	Torque	Bellevilles? ^a	1 K conductance ^b	Resistance ^c	Notes
A	A1-1	$125~\mathrm{N}~\mathrm{cm}$	y	$0.060 \pm 0.001 \mathrm{W K^{-1}}$	$0.41~\mu\Omega$	
A	A1-2	$<50~\mathrm{N~cm}$	y	$0.026 \pm 0.001~\rm W~K^{-1}$	$0.94~\mu\Omega$	
В1	B1-1	65 N cm	n	$0.101 \pm 0.017 \mathrm{W K^{-1}}$	$0.24~\mu\Omega$	Copper block
B1	B1-2	65 N cm	y	$0.109 \pm 0.002~\rm W~K^{-1}$	$0.22~\mu\Omega$	Invar block
B2	B2-1	< 50 N cm	y	$0.173 \pm 0.006 \mathrm{W K^{-1}}$	$0.14~\mu\Omega$	Invar block
B2	B2-2	$<50~\mathrm{N}~\mathrm{cm}$	y	$0.169 \pm 0.015 \mathrm{W K^{-1}}$	$0.14~\mu\Omega$	Unbroken from run B2-1 (Invar block)
B2	B2-3	$<50~\mathrm{N}~\mathrm{cm}$	y	$0.219 \pm 0.005 \mathrm{W K^{-1}}$	$0.11~\mu\Omega$	Broken and re-made (Invar block)
B2	B2-4	$<50~\mathrm{N}~\mathrm{cm}$	y	$0.086 \pm 0.002 \mathrm{W K^{-1}}$	$0.28~\mu\Omega$	Broken and re-made (Invar block)
B2	B2-5	$20~\mathrm{N}~\mathrm{cm}$	y	$0.146 \pm 0.004 \mathrm{W K^{-1}}$	$0.17~\mu\Omega$	Broken and re-made (Invar block)
С	C-1	100 N cm	n	$0.170 \pm 0.011 \mathrm{W K^{-1}}$	$0.14 \mu\Omega$	
C	C-2	100 N cm	n	$0.131 \pm 0.012 \mathrm{W K^{-1}}$	$0.19 \mu\Omega$	Unbroken from C-1 ^d
C	C-3	100 N cm	n	$0.132 \pm 0.006 \mathrm{W K^{-1}}$	$0.19~\mu\Omega$	Broken and re-made; identical to C-2
P	P-1	150 N cm	у	$0.154 \pm 0.004 \text{W K}^{-1}$	$0.16~\mu\Omega$	
Q	Q-1	65 N cm	y	$0.0467 \pm 0.0004~W~K^{-1}$	$0.52~\mu\Omega$	
Q	Q-2	65 N cm	y	$0.067 \pm 0.001 \mathrm{W K^{-1}}$	$0.36~\mu\Omega$	Broken and remade, identical to Q-1
R	R-1	76 N cm	y	$0.089 \pm 0.002 \mathrm{W K^{-1}}$	$0.28~\mu\Omega$	
R	R-2	76 N cm	y	$0.086 \pm 0.002 \mathrm{W K^{-1}}$	$0.29~\mu\Omega$	Measured in ADR from 100 to
						300 mK. Unbroken from R-1

^aDenotes whether Belleville washers were (y) or were not (n) used with the screws in this measurement

TABLE II: Details of configuration and measured values for each run.

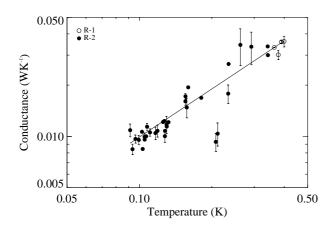


FIG. 5: Results from joint R measured in 3 He fridge (run R-1) and ADR (run R-2).

5. ANALYSIS AND DISCUSSION

The results are shown together in Fig 6. The measured values have not been converted to conductance per unit area; this would be inappropriate since conductance does not in general scale with area (see section 1). Despite the variation in geometry and torques used, our results mostly fall into a surprisingly small range in conductance. A few measurements do however

stand out as having significantly worse conductance than the rest.

The results from sample A were particularly poor. Examination of this joint after measurements showed that the surface of the threaded block had been damaged by tightening the screw, raising a ridge of copper around the hole. This presumably explains the low conductance. For the other measurements, in which clearance holes were used for both sides of the joint, this type of damage was not seen.

The rod-and-clamp joint Q also showed a relatively low conductance. We believe this is due to the fact that – as with all our measurements - the measured value includes the bulk conductance through the blocks making up the joints. For the rod and clamp joints, the bulk thermal resistance should be dominated by the resistance along the rod. The worst case is sample Q, with an approximately 16 mm length of rod between the centre of the sample at one end, and the copper block at the other end upon which the cold end heater was mounted. An examination of the literature shows that the conductivity of commercial copper varies linearly with temperature at low temperatures, with 1 K values generally falling between 130 and 250 Wm⁻¹K⁻¹. Using these values, the 1 K conductance along the rod should be between 0.06 and 0.12 WK^{-1} . The lower limit here is similar to the better of the two measured joint conductances.

For samples P and R, which had a shorter length of rod and higher measured conductances, again the lower limit on bulk conductance in each case is slightly higher than the to-

^bExtrapolated assuming a linear dependence on temperature

^cThe predicted electrical resistance is calculated from the thermal conductance and the Wiedemann-Franz law to aid comparison with results in the literature. No electrical measurements were made.

^dNote that this result is obtained from only two data-points (see Fig. 2)

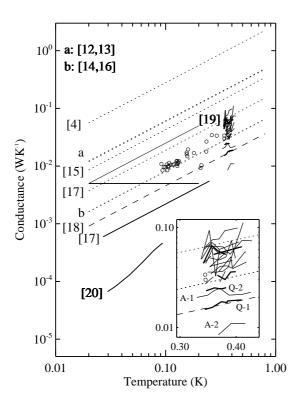


FIG. 6: Comparison of measurements with data from the literature. Results from this work: run R-2 (o); samples A-C (thin solid unlabelled lines); samples P-Q (thick solid unlabelled lines). Data from the literature [4, 12–20] (labelled by reference): direct thermal measurements (solid lines); predictions from the Wiedemann-Franz law (dotted lines); extrapolation from thermal conductance measured above 1 K (dashed line). For clarity, only the best result from each source is plotted (for Ref. [17], the best electrical and the sole thermal result is shown). Note that the measurement from Ref. [19] was a lower limit on conductance; the thinner line shows an extrapolation from this limit assuming the conductance is linear with temperature. Measurements from Refs. [12] and [13] overlay each other, as do the results from Refs. [14] and [16]. The inset shows the area around our ³He fridge measurements, with the addition of labels for some of the runs.

tal measured conductance. We therefore conclude that bulk conductance may be a large contribution to the measured conductance of the rod and clamp joints.

For the flat joints (samples A, B1, B2, and C), the calculated bulk conductance is at least an order of magnitude higher than the measured total conductances, and therefore the effect on the results should be small. This conclusion is supported by results from measuring sample B1 using different heater locations; no difference in measured conductance was seen. In addition, if the bulk conduction dominated, the conductance would not vary when a joint was taken apart and re-assembled.

A much larger set of measurements would be necessary in order to make a proper determination of the effect of variations in the various possible parameters. This is demonstrated by the fact that run B2-5 showed a considerably higher conductance than both the measurements on sample B1, even

though run B2-5 was made with a considerably lower torque, and both samples were nominally identical apart from the details of the gold plating. While it is possible that the difference results from the plating methods used, the effect of other possible variations such as a difference in surface flatness or microscopic roughness cannot be ruled out.

The temperature range for measurements made using the $^3{\rm He}$ fridge is far too small to measure the temperature dependence of the conductance. The results from measuring sample R in an ADR are shown in Fig. 5, along with $^3{\rm He}$ fridge measurements on the same sample. A powerlaw fit to both sets of data is shown in the graph; the resulting fit to conductance $G=aT^b$, where T is temperature, gave parameters $a=0.086\pm0.002\,{\rm WK}^{-(1+b)}$ and $b=0.94\pm0.02$. The temperature dependence is thus approximately linear, as would be expected on theoretical grounds for pure metallic contact. However, as stated above, the measured value may include a significant contribution from the bulk conductance of the rod in the joint.

Our measurements are compared with various results from the literature in Figure 6. Note that the thermal conduction measurements from Ref. [19] were for a somewhat different configuration from ours, being obtained using joints in which one side of the joint was screwed directly into the other. This arrangement appears to allow very good thermal contact, but is impractical for making joints in many circumstances.

The majority of the literature results were obtained by applying the Wiedemann-Franz law to electrical measurements made at around 4 K. While there is considerable evidence for the validity of this law in the case of bulk copper, there are very few results available for joints. Several authors have reported measurements above 1 K in which the thermal conduction was greater than the prediction from the Wiedemann-Franz law [2, 3, 14, 18], sometimes by several orders of magnitude [2]. This can be explained by assuming that the conduction is mostly through a dielectric layer, since a dielectric will allow thermal but not electrical conduction. The observation of a greater than linear temperature dependence of the conductance supports this conclusion. Obvious candidates for the dielectric material are oxide layers and frozen gases.

Rare deviations from the Wiedemann-Franz law in bulk copper appear to be associated with deformation [21–23]. This gives a possible mechanism for failure of the Wiedemann-Franz law in the case of thermal contacts, since the heat flow is likely to be through small raised areas of copper which have been somewhat deformed due to the pressure between the joints. Indeed, failure of the Wiedemann-Franz law was observed for a joint in which the copper was significantly deformed as the joint was made [20]. In this case, the thermal conductance was *less* than the prediction from electrical resistance. (Note that the relatively low value of the thermal conductance was intentional, the aim being to obtain a value which was easily measurable.)

Nevertheless, our results generally fall into the range of the Wiedemann-Franz predictions from the literature. Furthermore, the linear temperature dependence seen for sample R supports the validity of the Wiedemann-Franz law, since violations due to deformation appear to be accompanied by a

deviation from linear temperature dependence.

However, our conductance values are at the lower range of the predictions in the literature. This could be because our joints are genuinely inferior to some of those reported in the literature. This is not unlikely; the engineering requirements of the SPIRE instrument constrained us to use somewhat smaller torques than generally used for such joints. Reduction of the joint pressure due to differential thermal contraction may also have contributed to the low values. However, it also possible that a failure of the Wiedemann-Franz law has caused some of the predicted conductance values in the literature to be higher than the true values.

6. CONCLUSIONS

We have measured the thermal conductance of different configurations of bolted copper joints below 1 K, as part of a development program for the SPIRE [8] instrument on the Herschel space telescope. The joints were made either by bolting two flat surfaces together, or by bolting a clamp around a rod; we are unaware of any previous measurements on the latter configuration.

Our results mostly fell into a relatively narrow range of conductance. The thermal conductance of similar joints has been predicted by other workers using electrical resistance measurements and the Wiedemann-Franz law. Our values fell into the lower end of the range of these results – see Fig. 6.

The temperature dependence of the conductance was measured for one sample and found to be very close to linear. This suggests that, as expected, the thermal contact is primarily metallic, and supports the validity of the Wiedemann-Franz law.

We thus conclude that the predicted thermal conductances in the literature are valid, at least at the lower end of the range.

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