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Title: THE LOW TEMPERATURE THERMAL RESISTANCE OF HIGH PURITY COPPER AND BOLTED COPPER JOINTS

Author(s): A. R. Kerr and N. Horner

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THE LOW TEMPERATURE THERMAL RESISTANCE OF HIGH PURITY COPPER AND BOLTED COPPER JOINTS

A. R. Kerr and N. Horner
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SUMMARY

This report describes measurements in the range 3-6 K of the thermal resistance of high purity (99.999%) copper strips and bolted copper joints. The effects of annealing and work-hardening the copper are examined. Gold- plated copper joints and unplated copper joints were tested, and also joints with indium gaskets and silver epoxy adhesive.

METHOD OF MEASUREMENT

Measurements were made in a vacuum cryostat cooled by a closed-cycle Joule-Thomson refrigerator. With no heat load the cold plate reached about 3.3 K, and with a 1 watt load its temperature rose to ~ 6 K.

Temperatures were measured using two Lakeshore DT-470 silicon diode sensors calibrated at 4.2 K in liquid helium. Throughout this report the readings T1 and T2 will be taken to include the 4.2 K offset corrections.

The tracking of the sensors was measured by attaching them at symmetrical locations on a heatable cold plate. Figure 1 shows the reading T2 of sensor #2L vs. the reading T1 of sensor #1L; the difference (T2-T1) is shown on an expanded scale. Also shown is the expression TOS = $0.03 + 0.009x + 0.004x^3$, where x = T1 - 6. This expression is a good fit to the difference (T2-T1) over the temperature range considered.

In all measurements, the indicated temperature differences (T2-T1), after correction for the substantial liquid helium offsets (~ 0.5 K in each sensor), were corrected by the smaller amount TOS (typically ± 0.02 K).

The term *thermal resistance* as used in this report refers to the incremental thermal resistance d(T2-T1)/dW -- *i.e.*, the tangent to the temperature difference vs. power curve -- as opposed to the slope (T2-T1)/W of the chord on the temperature difference vs. power curve.

To measure the thermal resistance of a specimen, one of its ends was attached to the cold plate of the refrigerator, and the other end to a heater resistor. Temperature sensors were attached either side of the specimen. Figure 2 shows the arrangement used in measuring the thermal resistance of a copper strip. Note that the temperature at the colder sensor can rise as high as ~ 6 K, depending on the heat load and the thermal resistance between the sensor and the cold plate.

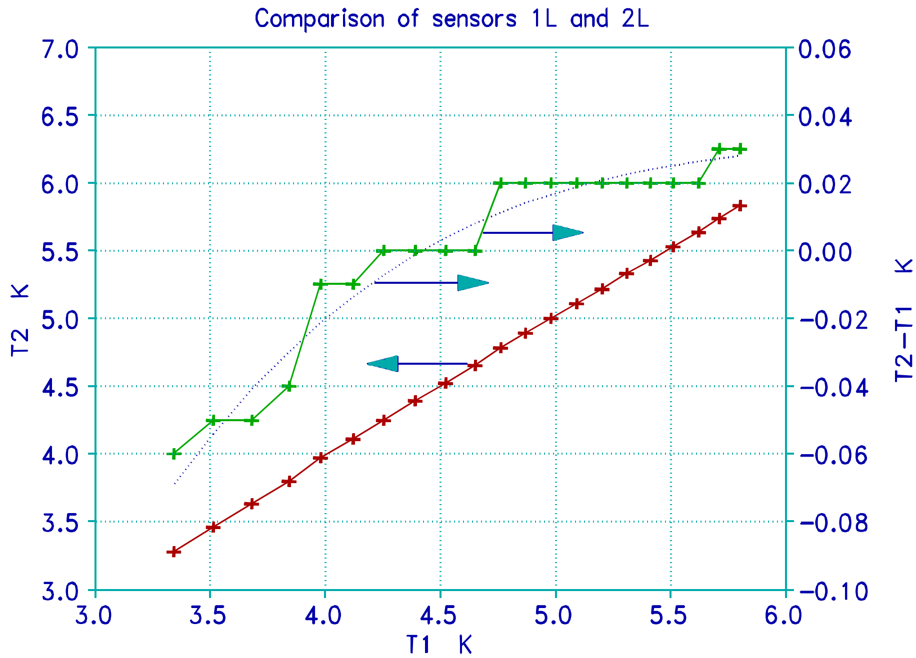

Fig. 1. The reading T2 of sensor #2L vs. the reading T1 of sensor #1L. The difference (T2-T1) is shown on the right scale. Also shown is the expression TOS = 0.03 + $0.009x + 0.004x^3$, where x = T1 - 6 (right scale).

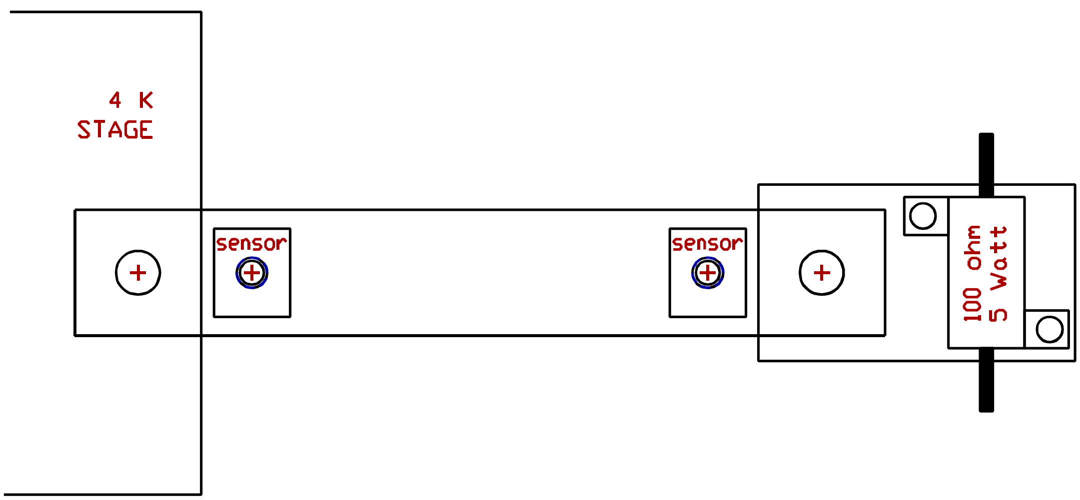

Fig. 2. Location of heater resistor and temperature sensors when measuring the thermal resistance of a copper strip.

HIGH PURITY COPPER

The thermal conductivity of 99.999% pure copper in the 4-20 K range is roughly an order of magnitude higher than that of either certified OFHC Cu (99.99% pure) or ETP Cu (99.90% pure) [1].

The measurements described here were made on 99.999% pure copper strips 0.50" wide x 0.094" thick cut from sheet stock and connected as shown in Figure 2. The distance between the temperature sensors was 1.8".

Figure 3 shows the results of measurements on the following samples from 3/32" sheet stock:

Unannealed.

Annealed. This specimen was annealed at 550°C for 3.5 hours in a nitrogen atmosphere, then oven cooled.

Work-hardened. After annealing as above, the specimen was bent sharply at 90° in four places, then bent flat again.

S-bend. After annealing as above, this specimen was bent into an S-shape with two 90° bends of approximately 0.3" radius.

The work-hardened specimen was measured again after re-annealing, and its thermal resistance found to have returned to the fully annealed condition.

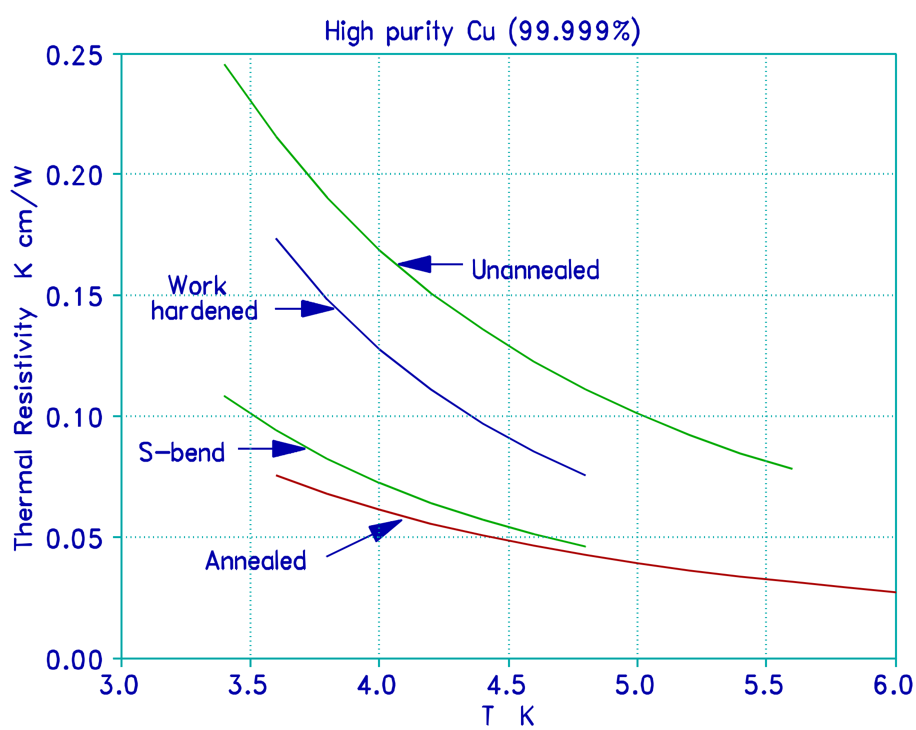

Fig. 3. Measured thermal resistivity of 99.999% pure copper. Preparation of the specimens is described in the text.

The measured thermal resistivity of annealed 99.999% Cu is compared with published values [2] in Figure 4.

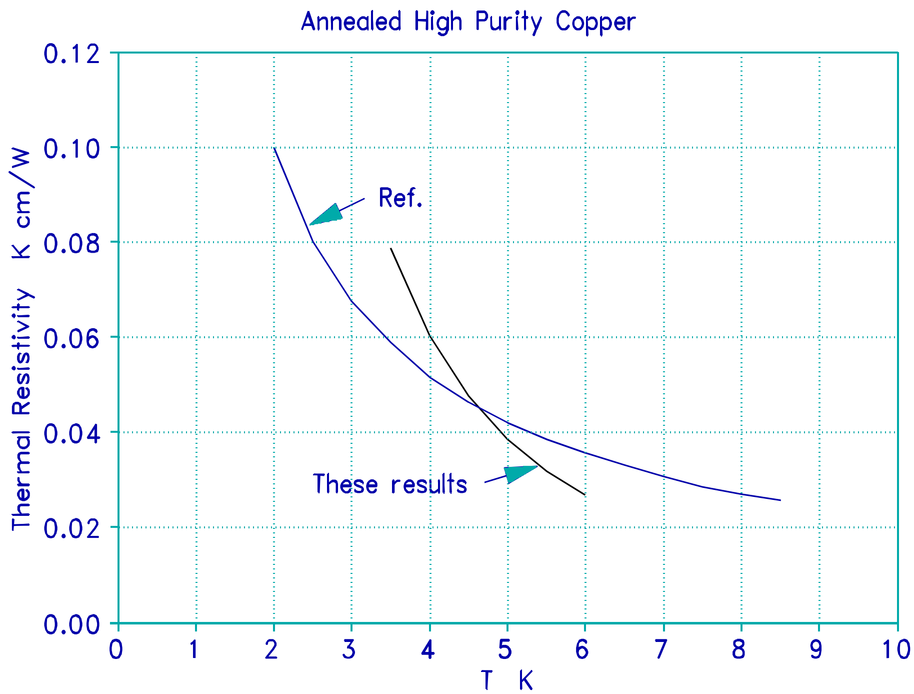

Fig. 4. Comparison of published data for 99.999% Cu [2] with the present measurements.

THERMAL RESISTANCE OF BOLTED JOINTS

To measure the thermal resistance of bolted joints between copper pieces, a stack of ten joints in series was used as shown in Figure 5. The contact area is 0.5" x 0.5". The #8-32 stainless steel bolt through the joint was tightened to the manufacturer's recommended torque of 28 in.-lb.

The thermal resistance of the following 10-joint assemblies of Cu pieces was measured.

Au-plated Cu pieces.

Clean unplated Cu pieces.

Tarnished unplated Cu pieces.

Au-plated Cu pieces with In gaskets.

Clean unplated Cu pieces with In gaskets.

Tarnished unplated Cu pieces with In gaskets.

Au-plated pieces alternating with clean unplated pieces.

Au-plated pieces alternating with clean unplated pieces, with In gaskets.

The results are summarized in Figures 6 and 7. The contribution of the copper to the total measured thermal resistance in Figures 6 and 7 is estimated to be 0.01-0.03 K/W (per joint) at 4.2 K, and arises mostly in the copper strips between the temperature sensors and the ends of the stack.

All copper pieces were cut from 3/32" thick 99.999% purity stock, annealed at 550°C for 3.5 hours in a nitrogen atmosphere, then oven cooled. After annealing, the pieces were cleaned with abrasive cloth and degreased. Plated pieces received 200 µinches of Pur-A-Gold 125 high purity gold. Pieces were tarnished by boiling in water for four hours, then air drying -- this produced an appearance similar to copper stored in air for a few years. Indium gaskets, where used, were 0.006" thick.

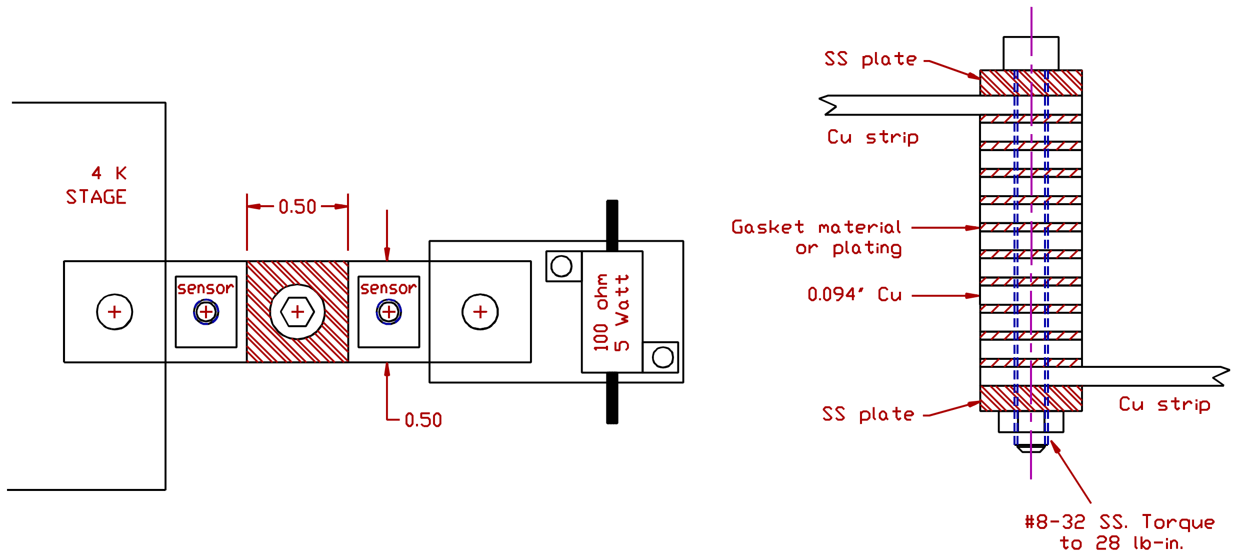

Fig. 5. Measurement of a stack of ten joints in series. The contact area is 0.5" x 0.5".

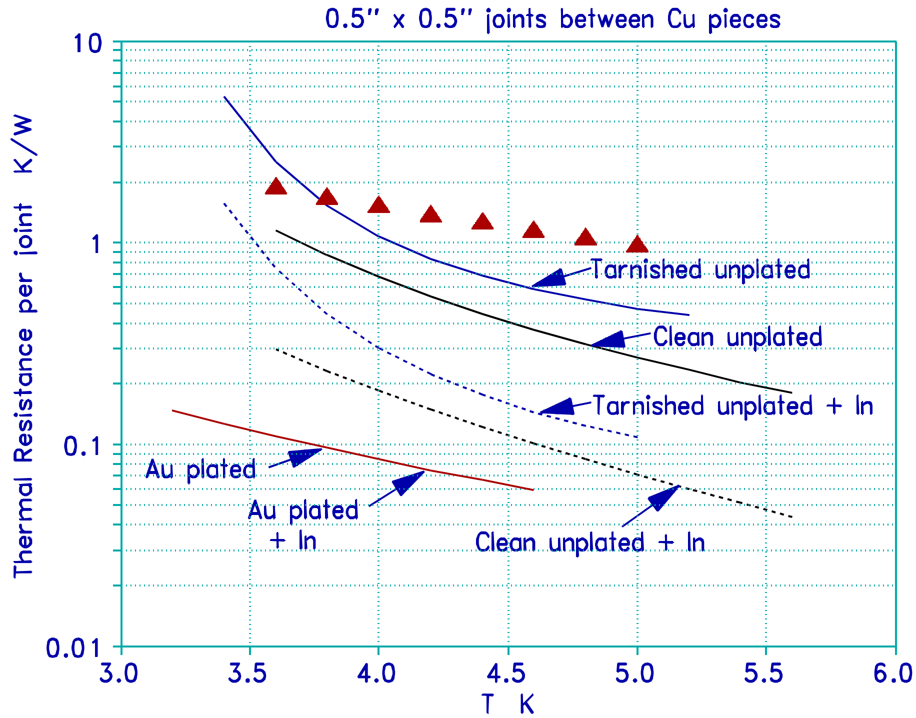

Fig. 6. Thermal resistance (per joint) of various 0.5" x 0.5" joints. Note that no significant difference was observed between gold-plated joints with or without indium. The points marked ▲ indicate the thermal resistance of an annealed high purity copper strip 3" long x 0.5" x 0.094", shown for comparison.

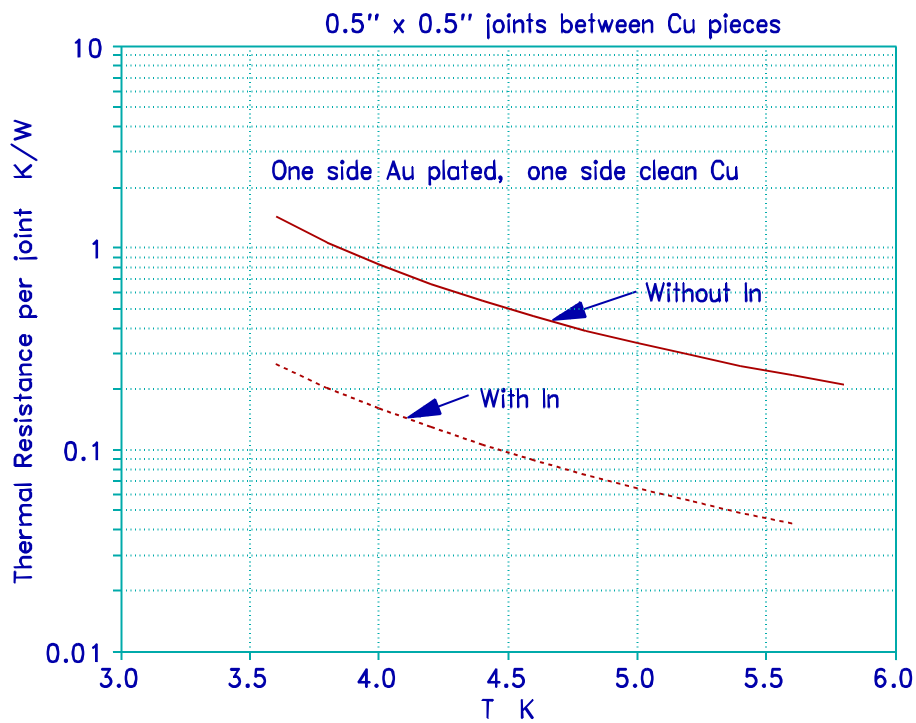

Fig. 7. Thermal resistance (per joint) of 0.5" x 0.5" joints with one side gold-plated copper and the other side clean copper.

OTHER GASKET MATERIALS

It has been suggested by Deutsch [3] that, based on electrical resistance measurements, the thermal resistance of bolted copper to copper joints can be improved by a factor of five using the silver epoxy adhesive Eccoshield VSM [4] in the joints. However, he also reported that copper-indium-copper joints were a further factor of two better than those with VSM. As we have found that gold-plated copper joints are about a factor of two lower in thermal resistance than Cu-In-Cu joints, we deduce that Cu-VSM-Cu joints should be substantially more resistive than gold-plated copper joints.

To determine whether Eccobond VSM might improve gold-plated copper joints, we tested gold-plated joints with and without VSM in the range 3.5-4.5 K, but found no significant difference.

DISCUSSION AND CONCLUSIONS

Thermal Resistivity of 99.999% Pure Copper

It is clear from our measurements that annealing high purity copper can reduce the thermal resistivity of sheet stock or work-hardened material by almost a factor of three.

While multiple sharp bends increase the thermal resistance substantially, 90° bends of 0.3" radius only increased the resistance of an annealed 1.8"-long x 3/32"-thick strip by ~ 10% per bend.

Our measured thermal resistivity of annealed high purity copper strips was within ± 34% of the published values from 3.5-6 K. The reason for the more rapid variation with temperature is not known.

Copper-to-Copper Bolted Joints

From the above results, we conclude that copper plated with pure gold (200 µinches of Pur-A-Gold 125) gave the best thermal contacts. Gold-plated joints were lower in thermal resistance by a factor of ~ 10 than joints between tarnished copper surfaces.

Addition of an indium gasket made no significant difference in the case of the gold-plated specimens.

For unplated joints, clean copper with indium was a factor of ~ 6 better than tarnished copper without indium. For both tarnished and clean copper, the presence of an indium gasket improved the resistance by a factor of ~ 3.6.

These observations are consistent with the theory of thermal conduction in pure metals, according to which the heat current is carried primarily by electrons (as opposed to phonons) [5]. The presence of even a thin oxide layer would then increase the contact resistance substantially. We speculate that the reduction in thermal resistance in a tarnished copper joint with an indium gasket may be a result of the increased effective contact area due to the soft indium conforming microscopically to the oxide covered surfaces.

Prior to these measurements we had thought that the presence of an indium gasket might seriously degrade the thermal contact resistance at temperatures approaching the critical temperature of indium (3.4 K). However, this appears not to be the case; an indium gasket improved the resistance of tarnished or unplated copper joints by a nearly constant factor of 3.6 from 3.4 to 5.6 K.

The use of Eccoshield VSM silver epoxy did not significantly improve the thermal resistance of gold-plated joints.

We have not investigated the affect of different bolt torques -- all our measurements were made using 0.5" x 0.5" contact areas and a #8-32 stainless steel bolt torqued to 28 in.-lb.

Sources of Error

Temperature calibration. The main uncertainty in these measurements arises from the lack of absolute temperature calibration. The only absolute calibration point for the sensors was 4.2 K; at other temperatures, one sensor was taken as reference, and the other sensor's reading corrected by the (measured) tracking error. This is not expected to contribute major errors in the present context.

Power measurement. Power to the heater resistor was determined from the measured voltage at the power supply and the heater resistance (100 Ω) and lead resistance (16 Ω). Because of uncertainties in the resistances when cold, the uncertainty in the heater power is ± 5%.

REFERENCES

[1] R. B. Scott, *Cryogenic Engineering*, van Nostrand, 1959.

[2] R. L. Powell and W. A. Blaniped, "Thermal Conductivity of Metals and Alloys at Low Temperatures," NBS Circular 556, September 1954.

[3] M. Deutsch, "Thermal Conductance in Screw-Fastened Joints at Helium Temperatures," *Cryogenics*, pp. 273-4, May 1979.

[4] Emerson & Cuming, Inc., Canton, MA 02021.

[5] C. Kittel, *Introduction to Solid-State Physics*, John Wiley, 1968.

ADDENDUM to EDTN No. 163:

THE LOW TEMPERATURE THERMAL RESISTANCE OF HIGH PURITY COPPER AND BOLTED COPPER JOINTS

A. R. Kerr and N. Horner
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We have found that the type of copper tested in NRAO Electronics Division Technical Note No. 163, "*The Low Temperature Thermal Resistance of High Purity Copper and Bolted Copper Joints*," was not, in fact, 99.999% copper, as reported, but had a purity of 99.98%, corresponding to oxygen free copper (alloy C10200).

The error was discovered when we tried to buy more of the supposedly 99.999% copper and found that such high purity material was not available. Samples of our stock labelled "99.999%" were then sent to Alpha Analytical Laboratories for analysis. The result, 99.98% Cu with 0.018% sulfur and 0.0006% silver as the only significant impurities, is consistent with alloy C10200 oxygen free copper, as can be seen in the table below.

The table lists the composition [1, 2] of several types of copper in order of decreasing thermal conductivity [3]. The low temperature thermal conductivity of these alloys differs by more than two orders of magnitude as a result of the small percentages of impurities.

As our results given in Figure 4 of EDTN #163 were close to those published by NBS [4] for actual 99.999% high purity copper similarly annealed, we conclude that the relatively high percentage of sulfur in our samples was not detrimental to their thermal conductivity. However, as there is no guarantee that future batches of C10200 copper will have such low levels of impurities which affect thermal conductivity, we recommend that C10100 oxygen free electronic copper be used for critical thermal connections.

Alloy	Name	% Cu min.	Main other elements %	Therm. cond. at low temp.
C10100	Oxygen Free Electronic Cu	99.99	(S ≤ 0.0018%)	Highest
C10200	Oxygen Free Cu	99.95	(S not spec.)	
C11000	ETP Cu	99.90		
C14500	Tellurium Cu	99.90	Te(0.5) P(0.01)	
C12200	Phosphorus deoxidized Cu	99.90	P(0.015-0.04)	
C17200	Beryllium Cu Alloy 25	97.85	Be(1.9) Co(0.25)	Lowest

[1] Data from Copper & Brass Sales, Inc., North Attleboro, MA 02763.

[2] Data from T. E. Conklin, Inc., Brooklyn, NY 11217.

[3] R. B. Scott, *Cryogenic Engineering*, van Nostrand, 1959.

[4] R. L. Powell and W. A. Blaniped, "Thermal Conductivity of Metals and Alloys at Low Temperatures," NBS Circular 556, September 1954.