PAUL SCHERRER INSTITUT

Memorandum

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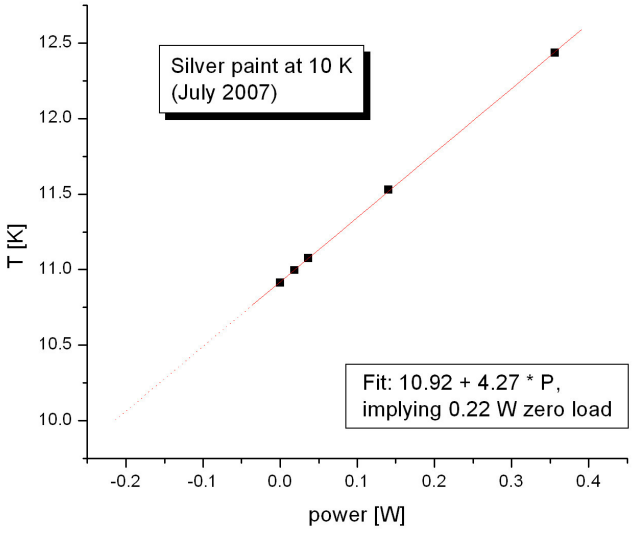
Cooling LEM samples below 2.7 K

There is now one cryostat with two possible inserts (MANGO and LEMON) that can in principle cool down to 1.4 K (see LEM documentation, sample environment and/or appendix 3). However, in all cases the lowest temperature on the sample is 2.34 K according to Morenzoni et al. (PSI Scientific Reports) and in practice about 2.7 K. Reason is the heat load of about 0.2 W due to radiation (light) on the sample.

Lower temperatures on the sample can therefore only be achieved by

- decrease of the heat load
- increase the coupling between the sample and the cold He.



The first is kind of hard, so let's focus on the second option.

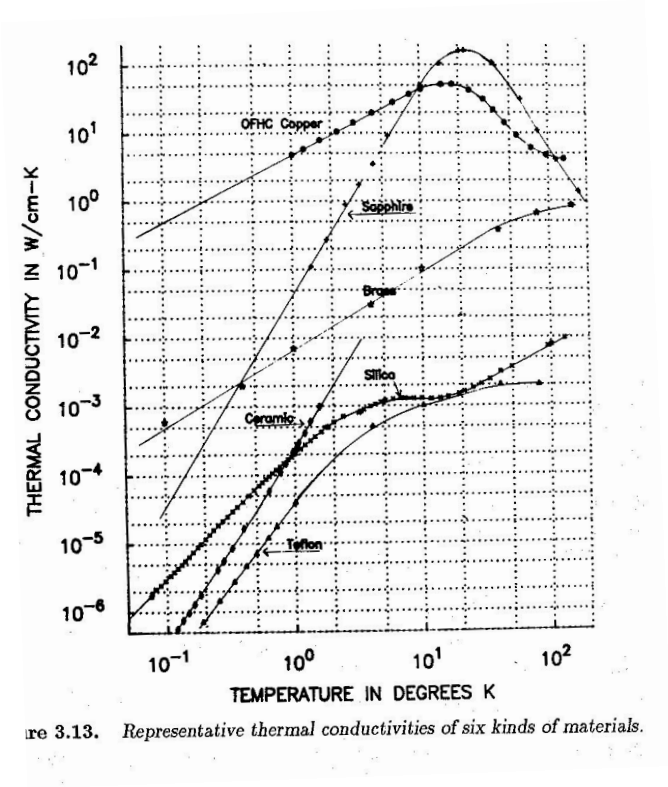
There are a number of obstacles that the heat flow from the sample to the He has to overcome:

1. interface between sample and substrate (in most cases)
2. interface between substrate and sample plate (Ag-coated Al plate)
3. interface between Al sample plate and In foil
4. interface between In foil and sapphire
5. interface between sapphire and In foil
6. interface between In foil and Al base plate.
7. interface between Al base plate and Cu cold finger
8. interface between Cu cold finger and cold He (liquid or gas)

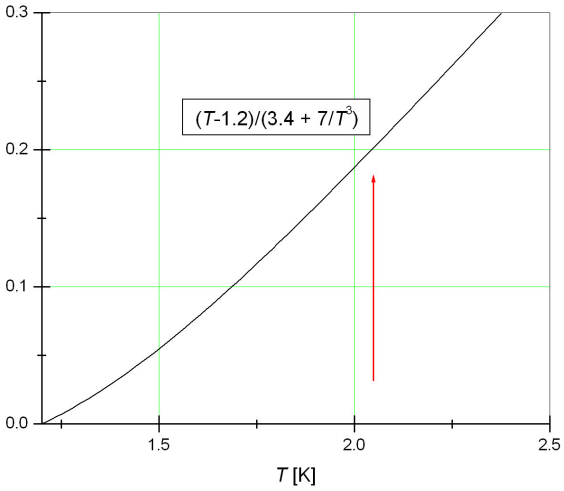
For interfaces 1 through 7 an –optimistic- engineering value for the heat resistance is 30 T^{-3}[K^4 cm^2 W^{-1}] (1). Assuming a diameter of 60 mm for all interfaces we reach at 1 T^{-3}[K^4 W^{-1}], or in total 7 T^{-3}[K^4 W^{-1}]

The 8-th interface suffers from the so-called Kapitza resistance (2), in this case (Cu-He) 45 T^{-2}[K^3 cm^2 W^{-1}], or (the diameter is much smaller) 3.5 T^{-2}[K^3 W^{-1}], or 2.5 K/W.at 1.2 K

Another problem that may exist with the heat conductance inside the materials could be the sapphire. The heat conductance at low temperatures is 0.014 T^3 [W K^{-4} cm^{-1}]. For the LEM case (6 cm diameter, 0.6 cm thick, 1.2 K) 0.9 K/W.



re 3.13. *Representative thermal conductivities of six kinds of materials.*



In other words, an estimate for the lowest temperature at the sample is given by

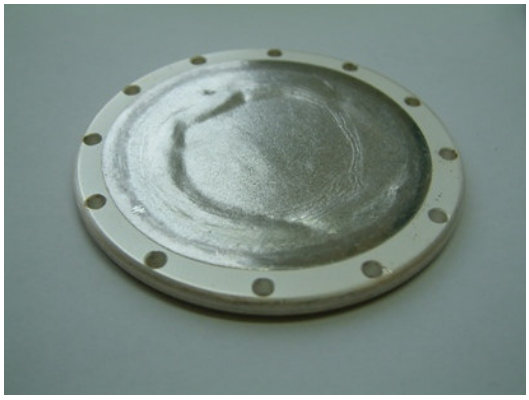
$$\frac{T-1.2}{3.4+\frac{7}{T^3}} = 0.2$$ which leads to 2.1 K

Clearly, the interfaces are the bottleneck. The sapphire contribute only 0.9 K/W in the worst case (at 1.2 K), causing a temperature difference of 0.9 * 0.2 = 0.18 K.

The He-copper interface inside the UHV insert is worst culprit. Therefore, even decreasing the temperature of the He would **increase** the temperature on the sample.

Improvements:

The best would be to have the sample on a silver plate, and provide that plate with a silver sinter on the backside Cooling can then be done by directly spraying He on the silver sinter. The plate should of course be electrically isolated, e.g. by means of a ceramic insulator device. Unfortunately, helium gas at a pressure around 1 torr has a very low breakdown voltage, order 200 V/cm, so this option can only be used when no or almost no HV has to be applied. Still OK e.g. for the meander experiment of Amit Keren.



Consequently, in general we do need the sapphire plate as an insulator. If we then go for the minimum number of interfaces, that sapphire plate should be covered (sputtered) on both sides with silver or sample with silver and cryo-side with copper. The sapphire plate should be part of the sealing between the flow cryostat and the high vacuum. On one side (the cryostat side) a sinter should be added, on the other side the sample should be directly mounted (glued) on the silvered sapphire. Thermometer and heater are on the cryo-side.

Minor improvements can be easily achieved by evaporating Indium on both sides of the sapphire to improve the contact with the In foil, or even replace the In foil. On the right hand side a sample-plate with In foil used once to cool the sample.

Major inprovements:

- Lowering the Kapitza boundary by building a sinter into the cryostat.
- Build a heat exchanger in the return gas line to better cool the thermal screen

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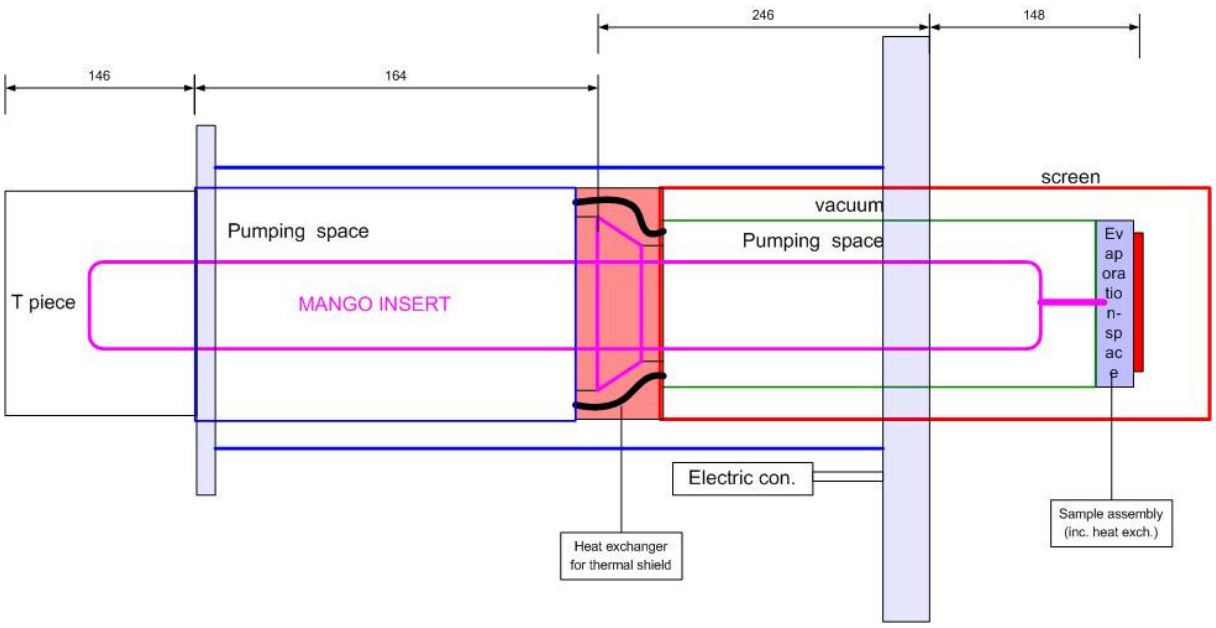
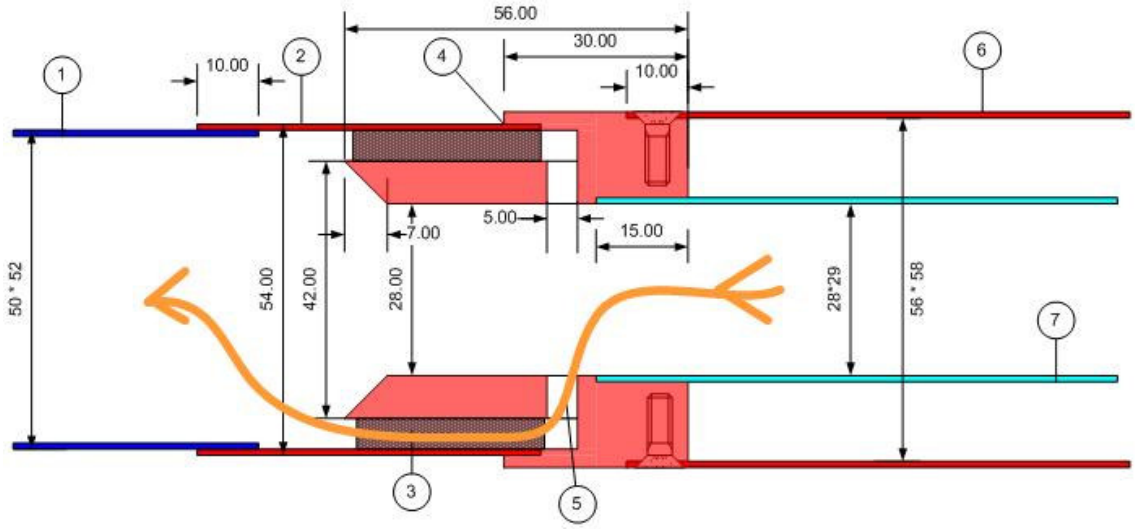


A sketch of the insert is shown in the figure above. Here the MANGO may be replaced at will by the LEMON.

The critical parts are the heat exchanger for the thermal shield and the sample assembly (3). In the next figure a suggestion for the first item is shown. The major part is made from copper to guaranty the heat conduction. In order to extract as much 'cold' from the He gas it should flow over as large a surface as possible, however without restricting the pumping speed too much. The latter is not important for the Konti

cryo's since the pressure of the gas is much higher at temperatures above 3 K. Cryovac uses a long tube in the form of a helix. For the MANGO a short path has been chosen, while filling that path with coarse grain sintered copper (4,5), see orange flow line in the figure below.

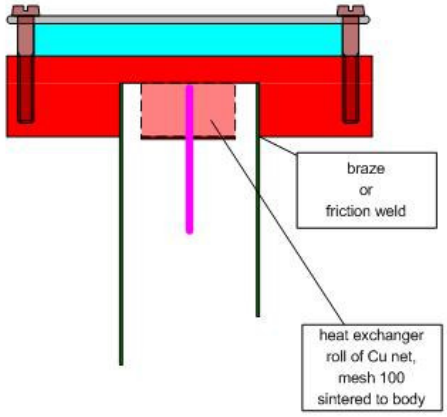
The tube running to the room temperature part is stainless steel, while the one going to the sample assembly should made out of Ti-6Al-4V, a light and very strong titanium alloy. This choice was inspired by the low heat conductance, high strength and small thermal expansion. See appendix 1.

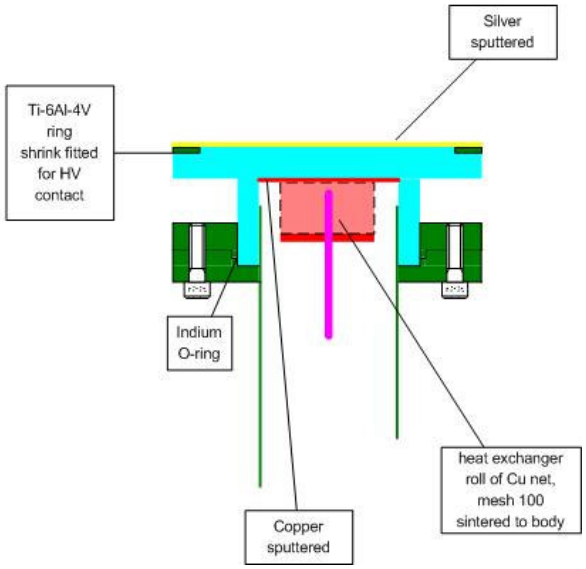


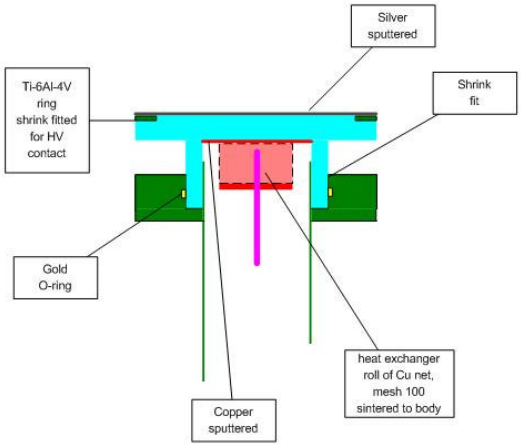
Heat exchanger for cooling heat shield.

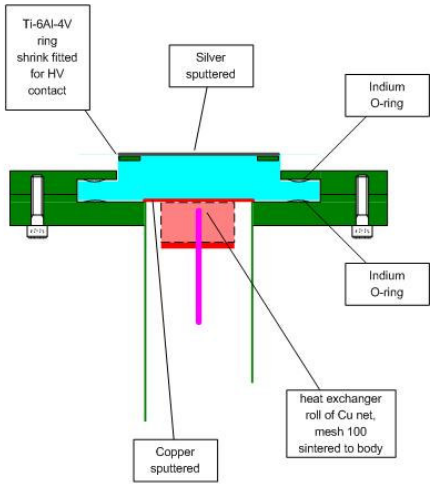
Legende:
1. RVS tube
2. Cu tube
3. Cu 'sinter', pieces of Cu-wire, 0.5 dia, 2 mm long, heated in 2 10^-4 air to 1000 degree, 3 hours
4. Cu tube should be attached by snug fitting + (afterwards) silver-solder
5. Large number of holes around circumvention for pumping He gas
6. Cu tube as heat screen (Au plated)
7. T-6Al-4V tube

Several sample assemblies:









copper | aluminium | sapphire | Ti-6Al-4V | vespel | cold He from MANGO | silver | gold

Above a number of suggestions to reduce the heat resistance between liquid/gas He and the sample.

Top Left:

This solution is not much different from the present situation. However, there is now a heat exchanger between the He and the copper, and the lower -so-called- base plate is absent. The heat exchanger is a loose roll of woven Cu net, mesh 100 (wires of 0.11 diameter, 0.14 mm aperture, open area 30 %). This net is sintered between the copper block and a copper plate by heating the whole thing to 1000 ^{0}C in 2x10^{-4} torr air. The small amount of air oxidizes eventual magnetic impurities in the copper, thereby improving its heat conductance considerably. The liquid/gaseous He coming from the MANGO is led to the bottom of this roll and the gas is pumped along the perimeter. As usual, In-foils should be used at both sides of the sapphire or the sapphire plate should be coated with Indium on both sides. Thermometer and heater can be mounted under the copper block. It would be an advantage to silver plate the top of the copper-block so that in special cases the sample can be glued directly onto the block. In principle this design has no “unknown” properties and could be built right away.

Top Right:

In all other three solutions the sapphire plate is cooled more directly by the He *and* the sapphire plate acts as part of the vacuum seal.

In this first example, the sapphire plate has a special tube-shaped extension which is fitted to the titanium body by an indium O-ring. See ref. 6, figure 5. Here the strength and the low thermal expansion of the Ti-alloy come into the footlight. At the top of the sapphire device a flat titanium ring is shrink fitted to the sapphire. Subsequently, the top surface (sapphire plus ring) is sputtered with a silver coating. The sample should be glued onto this silver coating and the high voltage can be applied via the Ti-ring. The inside of the tube should be sputter coated with Cu to enable sintering the heat-exchanger. Care has to be taken not to “blow out” the sapphire device. In other words, the cryostat should be evacuated *before* the sample chamber is evacuated.

Bottom Left

This is a variant of the design described above. The indium O-ring sealing is replaced by a Au ring. Tightening of the seal is to be achieved by using the (small) difference in thermal expansion between sapphire and the Ti-alloy. Of course the titanium holder should only deform elastically. In view of its tensile strength and elastic modulus (see app. 1) the holder can be made 0.8 % smaller then its final size at room temperature. Even if we take some safety, 0.5 % is still OK. At the same time, the Ti alloy can be heated to 880 ^{0}C without loosing its properties, so that a thermal expansion of (880 – 30) * 9 x 10^{-6} = 0.75 % can be achieved. That is of the right order of magnitude in view of the strength and leaves sufficient space for a Au-seal.

If the special shape of the sapphire needed in both designs above can not be obtained, then we can simply use a thicker piece of sapphire, even 12 mm in stead of 6 m will lead to an increase of the final temperature on the sample of only 0.2 K.

Bottom Right

If the Au-shrink fitting appears to be impossible, but the ‘direct’ contact with the sapphire coated on the sample site with silver and on the He side with copper is promising, then the sapphire could be pressed between to In O-rings.

Realization of tests

In order to test the possibilities described above, I suggest to built the vacuum insert with an extra In-sealed pair of flanges in the T-tube so that several sample-assembles can be mounted. To measure the temperature of the “sample” a dummy thin film sample of $Ge_{0.82}Au_{0.18}$ of which the electrical resistance varies as $T^{0.84}$ from 1 to 300 K. (7) and appendix 2.

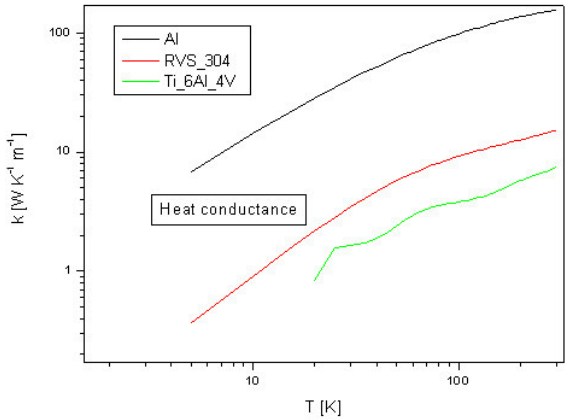
Appendix 1, properties of T-6Al-4V

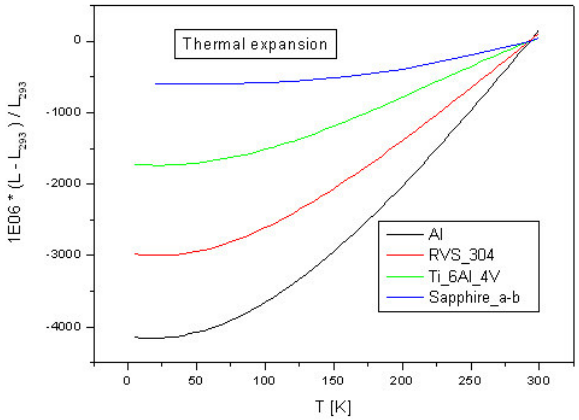
Physical Properties

Density	4.42 g/cm^3
Melting range	1649 ^{0}C
Electrical resistivity	170 Ωcm
Coefficient of Thermal Expansion (0-300 ^{0}C)	9.2 10^{-6}
Tensile Strength	1000 MPa
Elastic Modulus	114 GPa
Hardness	36 Rockwell C

Composition

C	< 0.08 %
Fe	<0.25 %
N_2	<0.05 %
O_2	<0.2 %
Al	5.5 – 6.7 %
V	3.5 – 4.5 %
Ti	Rest





Appendix 2, resistance of $Ge_{0.82}Au_{0.18}$ from ref. 7

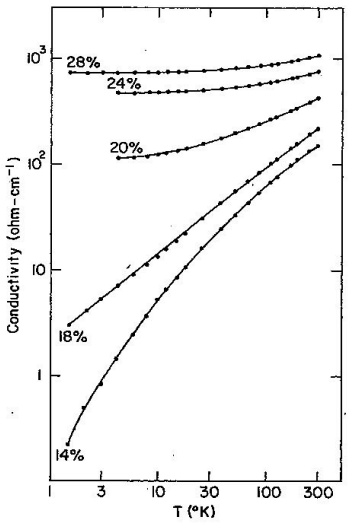
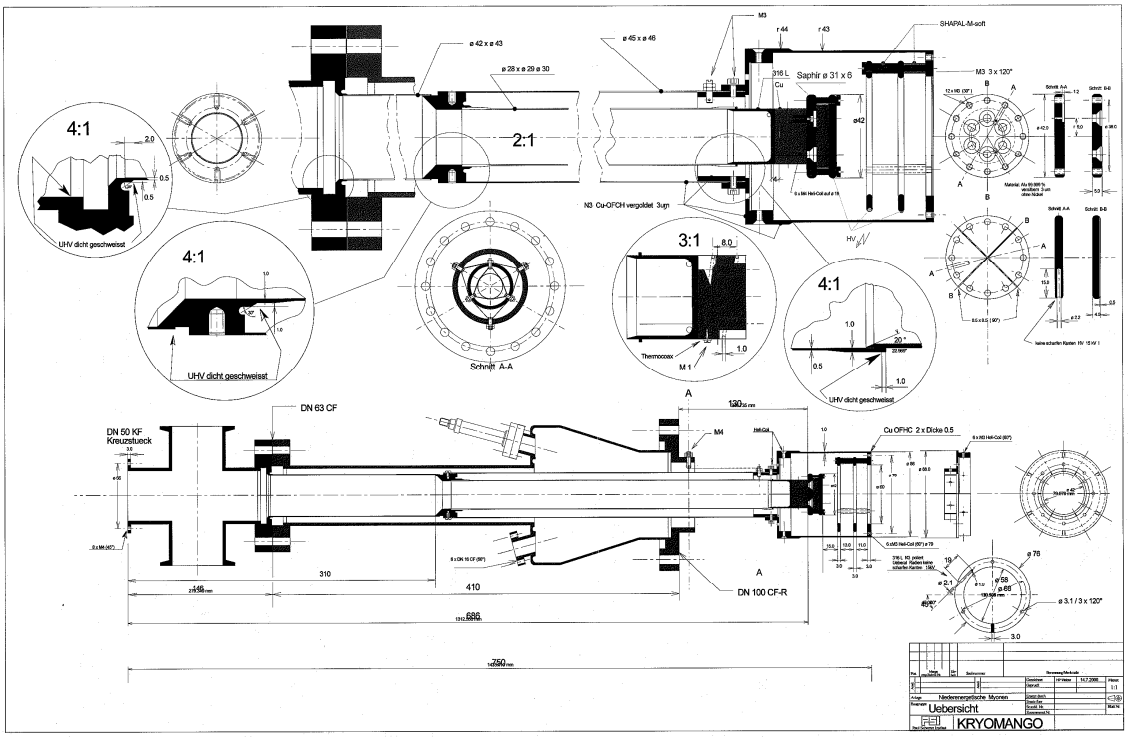


FIG. 1. Conductivity vs temperature for polycrystalline $Ge_{1-x}Au_x$ samples. With proper heat treatment at approximately 130 C the conductivity can be made to follow a $T^{+0.84}$ dependence over three decades in temperature.

Appendix 3: Original MANG0 insert



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