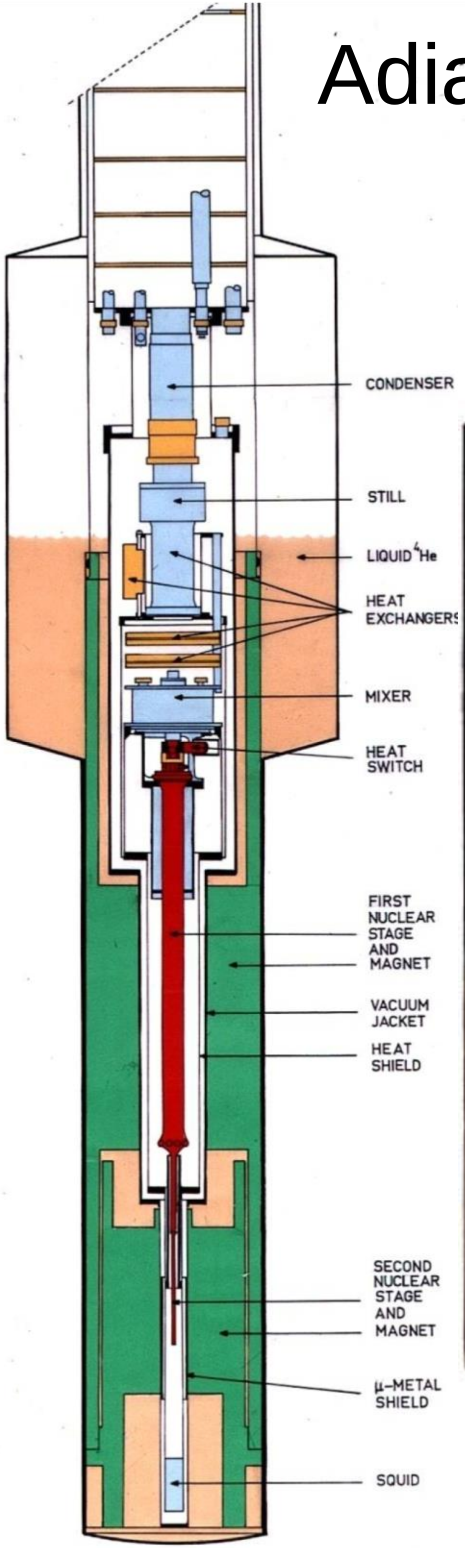
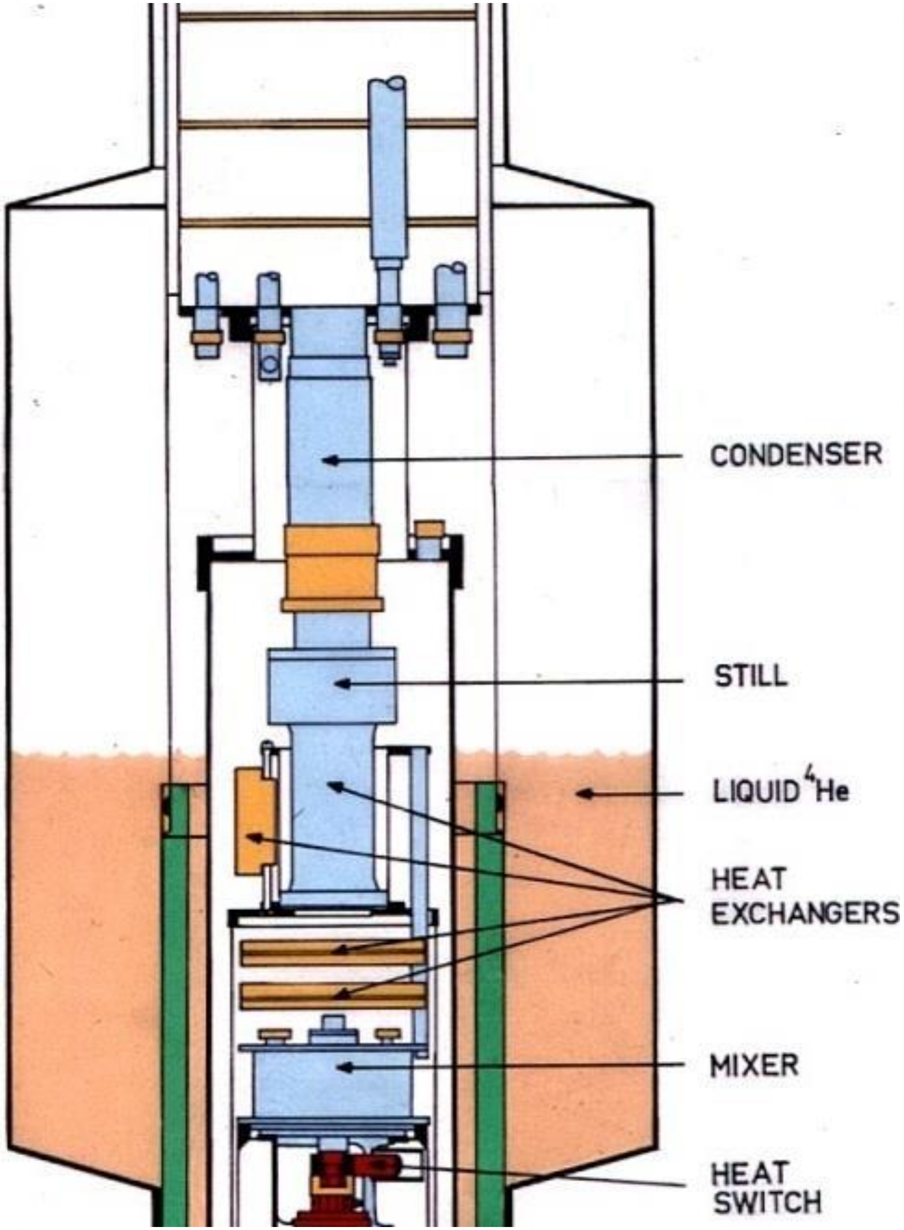
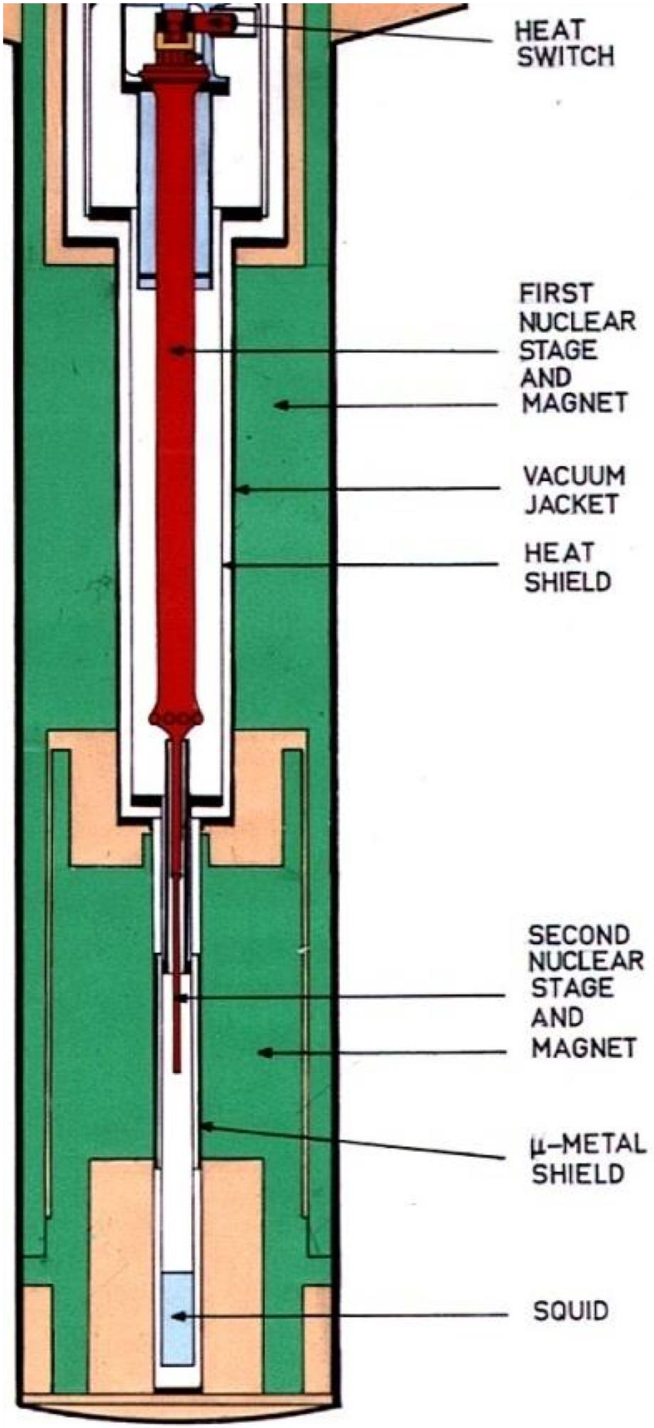
Adiabatic nuclear cooling refrigerator







Thermal contact and isolation

- Best possible **ISOLATION** from the enviroment

Thermal load from the hot surroundings cannot be completely eliminated:

1) thermal conduction
    - support structures
    - wiring for the measurements
    - residual gas in vacuum isolation
2) thermal radiation
3) power produced in the low temperature assembly
    - mechanical vibrations
    - electromagnetic interference
    - signals used for the measurements
    - etc, etc, …

`viscous heating`
`ortho-para conversion`

- Best possible **CONTACT** with the refrigeant

One needs to consider

- proper choice and preparation of the materials
- joints between the parts and contact resistance
- effect of thermal expansion (the structures are put together at room temperature and then cooled down)

Temperature ranges

The problems at high-*T* and low-*T* stages are different

Range:	100 ... 1 K	1 ... 0.01 K	10 ... 0.1 mK
Cooler:	He bath/pulse tube	dilution fridge	nuclear demag
Typical mass:	20 ... 200 kg	5 ... 50 kg	1 ... 5 kg
Typical max *P*:	W ... mW	mW ... µW	µW ... nW ... pW
Main heat Sources:	thermal radiation/ conduction	conduction/ dissipation	dissipated power
CONTACT:	quite easy	some trouble	difficult
ISOLATION:	somewhat difficult	relatively easy	relatively easy

Heat switches

Going from room temperature to mK regime and below, one must switch contact between the cooling stages at certain points.

- initial heat capacities of the warm system are very high
- lower refrigerator stages can be operated only below certain starting temperatures
- single cycle refrigerators require a precooling phase (contact) and the operation phase (isolation)

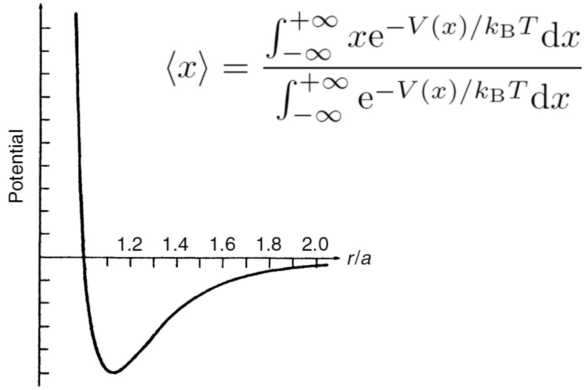
To change between good contact and good isolation, one uses

- heat exchange gas (above 4K)
- superconducting heat switch (below ~ 0.1 K)

Thermal expansion

– results from anharmonicity of
the binding potential

$$\alpha = \frac{1}{l}\left[\frac{\partial l}{\partial T}\right]_p$$



$$\langle x\rangle = \frac{\int_{-\infty}^{+\infty} x\mathrm{e}^{-V(x)/k_\mathrm{B}T}\mathrm{d}x}{\int_{-\infty}^{+\infty} \mathrm{e}^{-V(x)/k_\mathrm{B}T}\mathrm{d}x}$$

– approaches zero at low temperatures
– most of it occurs already above LN2 temperature (~ 77 K)
– stress test can usually be performed at LN2 temperature with high reliability
  * leak testing
  * test of electric wiring

– essential when parts of different materials are connected
– may result in leaks, bad thermal or electric contacts, ruptured parts
– due to change in dimensions parts inside one another may become touching

– metals typically contract 0.2 ... 0.4 % (RT–LT)
– plastics and most dielectrics 1 ... 2 % (RT–LT)
– some special compounds have very low thermal expansion (Pyrex glass, Invar steel)

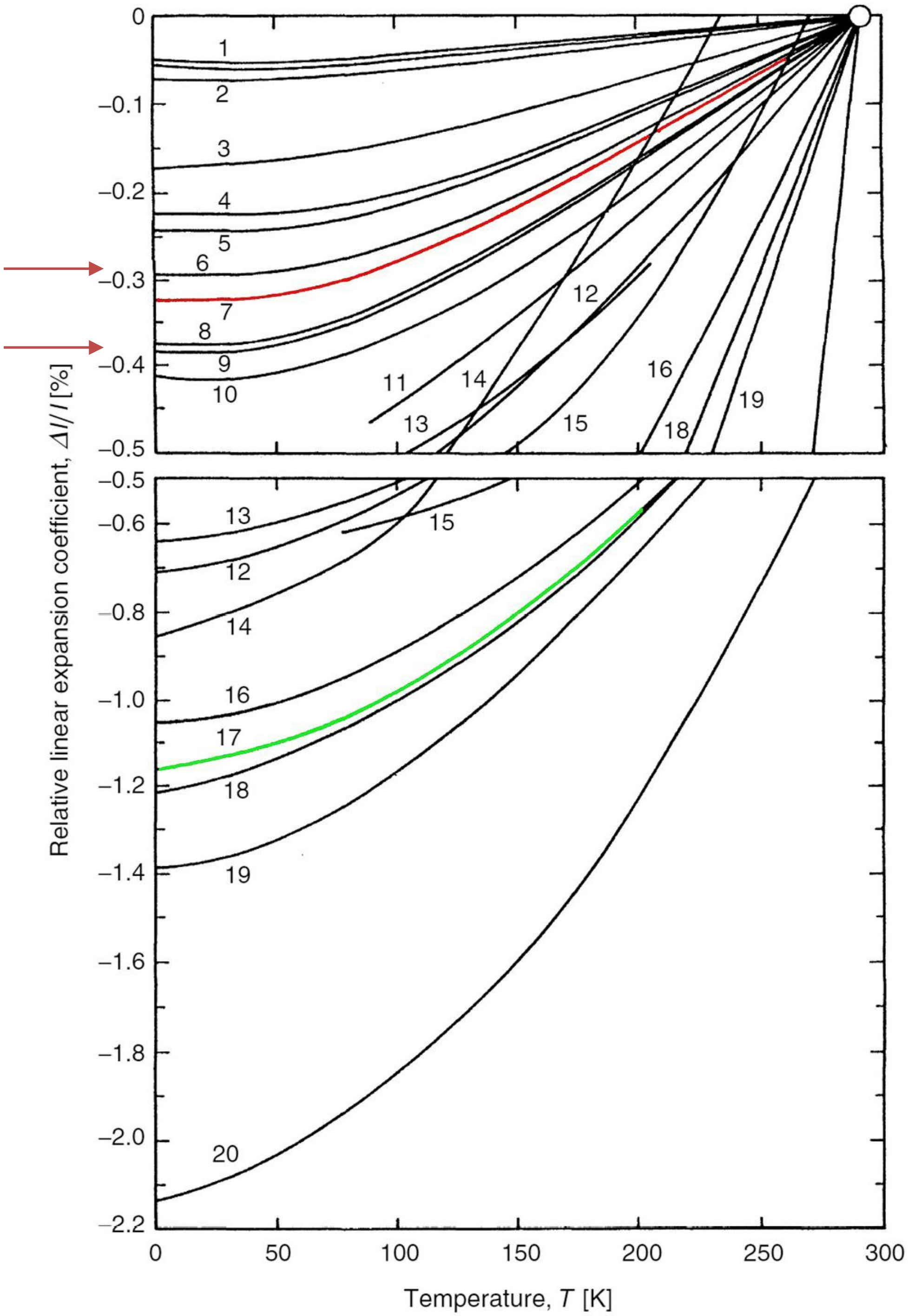
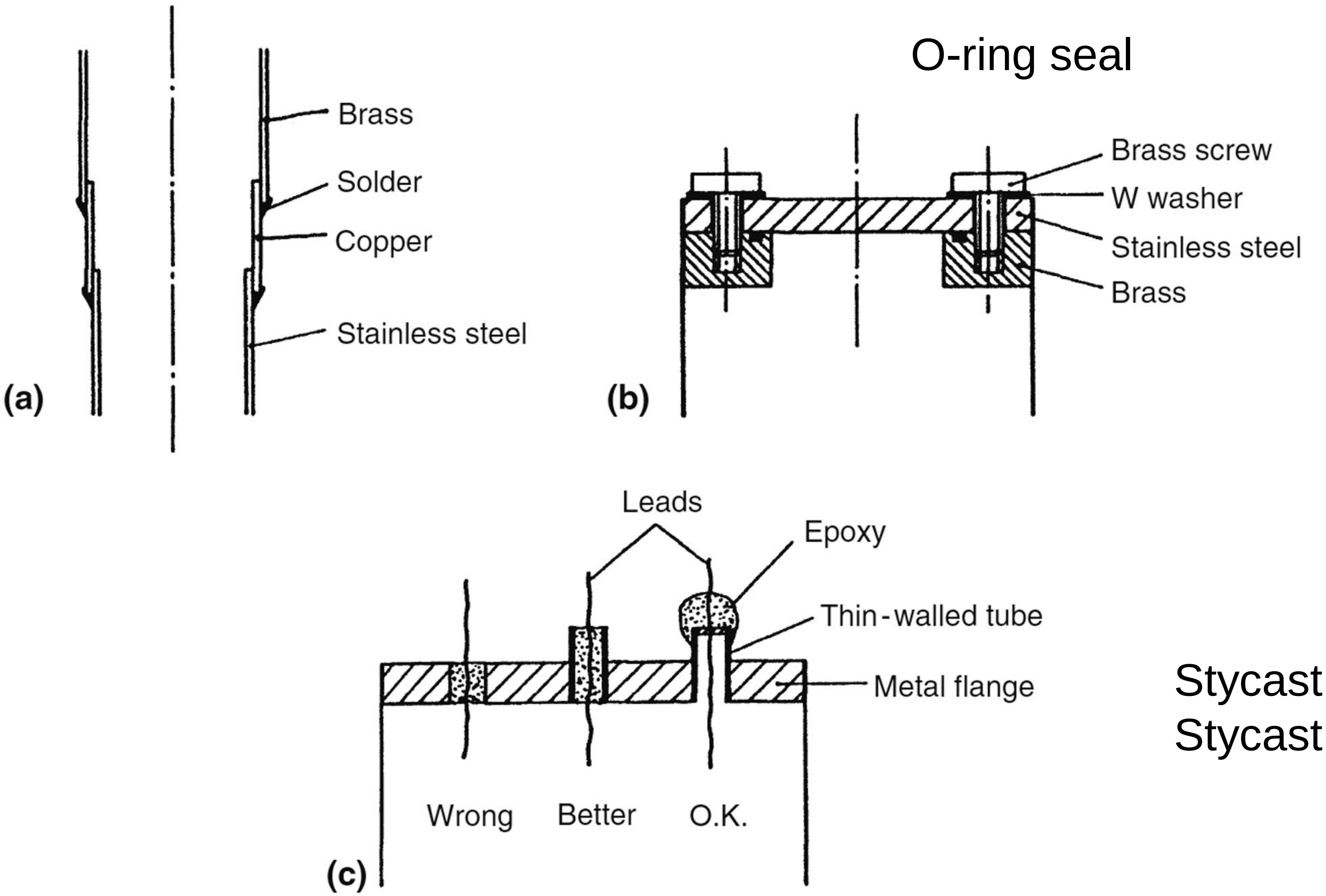


Fig. 3.17. Relative linear thermal expansion coefficient of (1) Invar (upper), Pyrex (lower), (2) W, (3) nonalloyed steel, (4) Ni, (5) $Cu_{0.7}Ni_{0.3}$, (6) stainless steel, (7) Cu, (8) German silver, (9) brass, (10) Al, (11) soft solder, (12) In, (13) Vespel SP22, (14) Hg, (15) ice, (16) Araldite, (17) Stycast 1266, (18) PMMA, (19) Nylon, (20) Teflon [3.76]. Some further data are: Pt similar to (3); Ag between (9) and (10); Stycast 2850 GT slightly larger than (10). The relative change of length between 300 and 4 K is $10^3 \Delta l / l = 12, 11.5, 4.4, 6.3$ and 5.7 for Polypropylene, Stycast 1266, Stycast 2850 GT as well as 2850 FT, Vespel SP-22 and solders, respectively [3.44, 3.55, 3.56, 3.76–3.82, 3.114]. Torlon behaves very similar to Stycast 2850FT [3.114]



O-ring seal

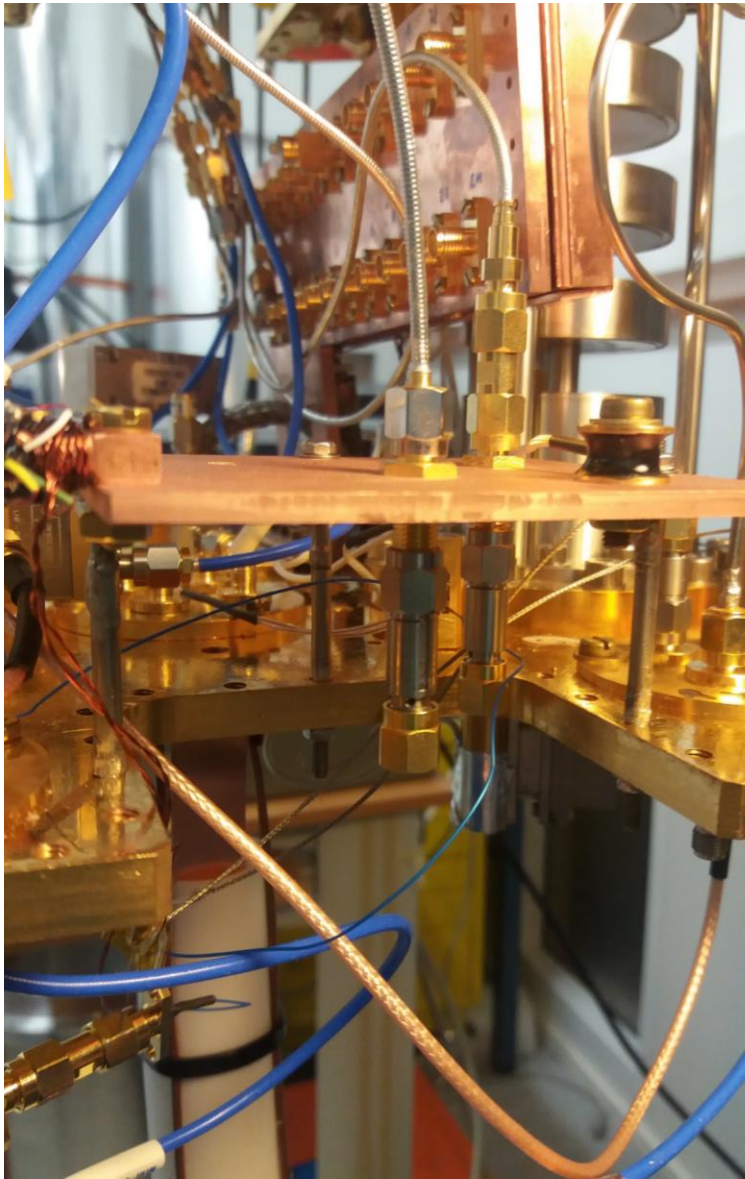
Stycast 1266
Stycast 2850FT

Fig. 3.18. When joining different materials in a cryogenic apparatus one has to take into account the difference in their thermal expansion coefficients. For example: (**a**) The tube with the largest expansion coefficient should be on the outside so that the solder joint is not pulled open during cooldown. (**b**) In an O-ring seal the screw should have a larger expansion coefficient than the flange. The seal will tighten even further during cooldown if a washer with a very small expansion coefficient is used. (**c**) In an epoxy feedthrough for leads the epoxy, with its large expansion coefficient, should contract on a thin-walled metal tube during cooldown rather than pull away. It helps if the tube walls are tapered to a sharp edge. An epoxy with filler should be used to lower its thermal expansion coefficient

Support structures

Thermally isolating support structures, tubing, and wiring can be made of

- teflon, nylon, graphite, alumina (Al_2O_3), glass fiber
- stainless steel, CuNi, manganin, constantan, NbTi



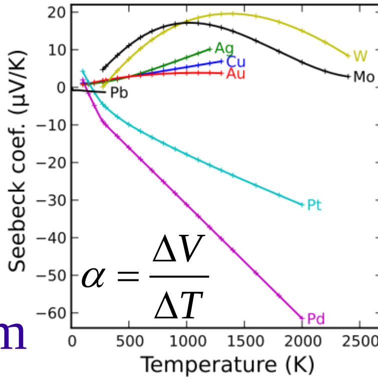
Important material properties:

- workability (can it be processed to the desired shape?)
  good: teflon, nylon, steel bad: alumina
- connectivity (can it be glued, soldered, welded?)
  good: glass fiber, CuNi bad: teflon (alumina)
- strength
  good: steel, glass fiber bad: graphite
- thermal expansion and stresses due to materials
  good: steel, CuNi, glass fiber bad: nylon
- magnetism
  (steel ?)

Note ! Choose right dimensions:

minimize the area/length A/l

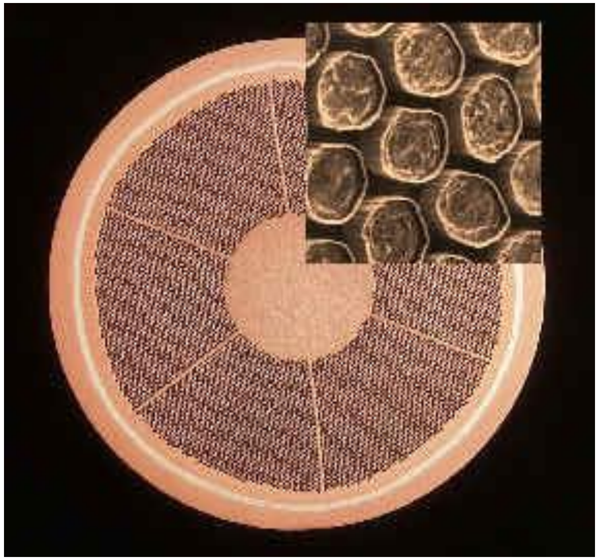
Wiring



$$\alpha = \frac{\Delta V}{\Delta T}$$

Room temperature to 4K bath

* **voltage leads** can be resistive (note thermopower for DC measurements)
  – manganin ($Cu_{0.87}Mn_{0.13}$) or constantan ($Cu_{0.57}Ni_{0.43}$), $\rho \sim 45\ \mu\Omega$cm
  ($\phi = 0.1$ mm , $l = 1$ m ; WF => 40 μW/wire)
* **current leads** ($I \sim 0.01 \ldots 10$ A) must be copper
  – optimize the diameter to balance between thermal conduction (bad for thick wire) and ohmic losses (bad for thin wire)
* **current leads** $\boldsymbol{I > 10}$ **A** copper/brass
  – big SC magnets may be equipped with detachable current leads
  – gas cooling must be provided through inner gas channels or by flat geometry
  – High T_c sections in the leads



4K to 1K inside vacuum jacket

* voltage leads may still be resistive
* current leads should be made out of
  superconducting wire with copper matrix

Inside the vacuum jacket (**below 1 K**)

* all leads (< 10 A) superconducting wires with CuNi matrix
  ($\phi = 0.05 \ldots 0.2$ mm ; WF => 2 ... 30 nW/wire)

+ thermal anchoring at ~ 0.1 K and 10 mK
+ thermal break below 10 mK by using the SC filament only (etch away the matrix)

Attach well and shield (twisted pairs/coax) to avoid inductive disturbance coupling

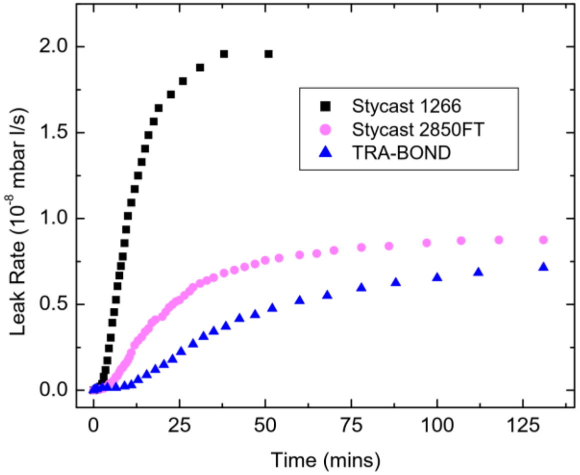
Residual gas

Vacuum space: (only helium has any significant vapor pressure < 4K)

- poorly pumped heat exchange gas
- small leak
- helium diffusion through dielectric materials
  (rubber o-rings, glued feed throughs, glass-fiber walls)

Already pressures as low as $p \sim 10^{-6}$ mbar are significant, as

$$(dQ/dt)/\mathrm{W} \sim a\, A/\mathrm{cm}^2\, p/\mathrm{mbar}\, \Delta T/\mathrm{K}\ ,$$



1.4 mm thickness

$L = \sqrt{Dt}$ $\quad t = L^2/D$

Where $a \sim 0.01$ for helium

For example $\left.\begin{array}{l} A = 600\ \mathrm{cm}^2 \\ T_1 = 4\ \mathrm{K} \\ T_2 \sim 0 \end{array}\right\}$ => $dQ/dt \sim 25\ \mu\mathrm{W}$

Pump carefully! “Cryopumping by fridge” works if no leak

Often a cold charcoal adsorption cartridge is placed inside the vacuum space
(essential for LN2 and LHe dewars)

Thermal radiation

Fixed anchored radiation shields:

- well conducting metal (Cu, Al)
- as shiny as possible (emissivity << 1);
  silver plating improves Cu shields

Superinsulation:

- thin aluminized mylar foil
- used for insulating LHe dewars
- tens or hundreds of layers wrapped in
  between the fixed radiation shields
- helps with residual gas problems also
  (particles have to transport heat between multiple successive layers)

thermal radiation is usually not a serious issue at temperatures below 1 K
(exception: nuclear demagnetization cryostats must have a shield at $T \sim 0.1$ K)

Other heat sources:

Usually not problematic, as these can be suppressed to levels of some nW "easily"
These become an issue for nuclear demag. stages only (nanoscale samples)

Thermal contact

Copper is the standard material for good contacts at low T (nonmagnetic, not supercond.)
– essential to anneal in low oxygen atmosphere to improve conductivity

At very low temperatures (**< 1 mK**) and
in high magnetic fields (**> 1 T**) **silver** is better

- – **nuclear magnetic moment** contributes to heat capacity ($\mu_{\mathrm{Ag}} \sim 1/20\ \mu_{\mathrm{Cu}}$)
- – anneal as Cu, but Ag has somewhat lower melting point (960 vs. 1080 ^{o}C)
- – magnetic impurities not as detrimental as for Cu

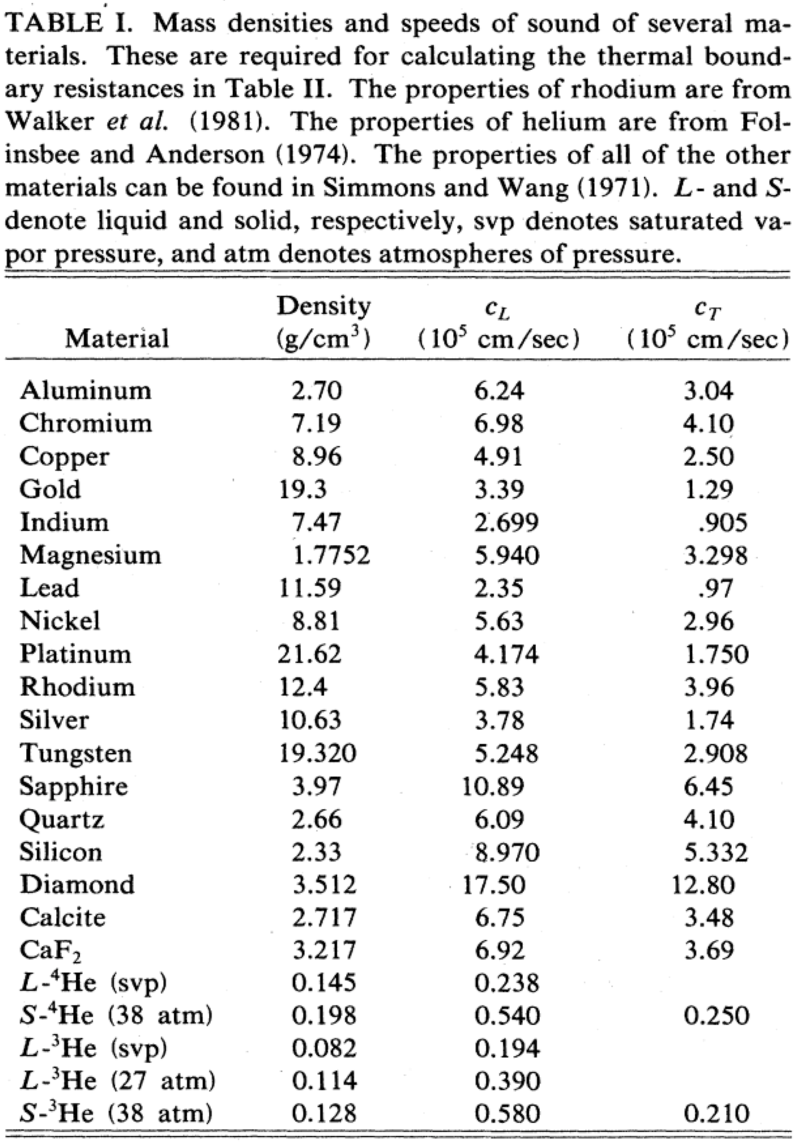


TABLE I. Mass densities and speeds of sound of several materials. These are required for calculating the thermal boundary resistances in Table II. The properties of rhodium are from Walker *et al.* (1981). The properties of helium are from Folinsbee and Anderson (1974). The properties of all of the other materials can be found in Simmons and Wang (1971). L- and S- denote liquid and solid, respectively, svp denotes saturated vapor pressure, and atm denotes atmospheres of pressure.

Material	Density (g/cm^3)	c_L (10^5 cm/sec)	c_T (10^5 cm/sec)
Aluminum	2.70	6.24	3.04
Chromium	7.19	6.98	4.10
Copper	8.96	4.91	2.50
Gold	19.3	3.39	1.29
Indium	7.47	2.699	.905
Magnesium	1.7752	5.940	3.298
Lead	11.59	2.35	.97
Nickel	8.81	5.63	2.96
Platinum	21.62	4.174	1.750
Rhodium	12.4	5.83	3.96
Silver	10.63	3.78	1.74
Tungsten	19.320	5.248	2.908
Sapphire	3.97	10.89	6.45
Quartz	2.66	6.09	4.10
Silicon	2.33	8.970	5.332
Diamond	3.512	17.50	12.80
Calcite	2.717	6.75	3.48
CaF_2	3.217	6.92	3.69
L-^{4}He (svp)	0.145	0.238	
S-^{4}He (38 atm)	0.198	0.540	0.250
L-^{3}He (svp)	0.082	0.194	
L-^{3}He (27 atm)	0.114	0.390	
S-^{3}He (38 atm)	0.128	0.580	0.210

Metal-to-metal joints:

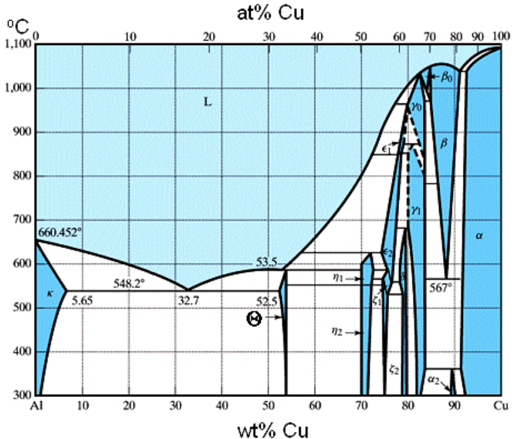
Any discontinuity in the thermal path gives rise
to contact resistance (Kapitza resistance):

$$\Delta T = R_K\, dQ/dt \qquad (R_K \propto A)$$

Surface roughness is important in pressed joints

- – **true contact area**: as small as 10^{-6} the joint area
- – contact area enhanced by **forced deformation**
- – **pressing to yield limit** deforms the structure, but this worsens the bulk conductivity

Thermal contact (cont.)



Welded joint is often the best for pieces of the same material

- TIG welding (surfaces usually oxidize – cleaning necessary)
- diffusion welding (heat up to 0.6–0.8 of melting temperature in vac. under pressing)
- electron beam welding (produces very narrow melted zone)

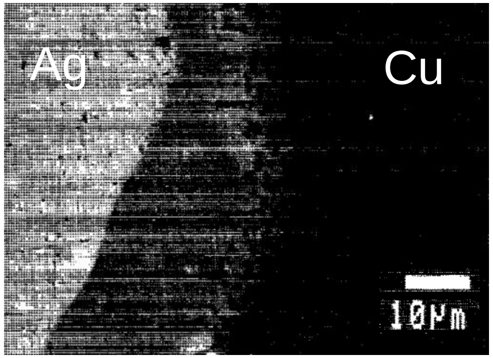
Results improve if annealing is performed after welding

For dissimilar metals some alloying at the joint region inevitably occurs.
The alloyed region is poorly conducting.

- diffusion welding; must compromise between strong joint and reduced alloying, fairly quick operation usually the best (e.g. Al – Cu 15 min at 500 ^{o}C)
- results are usually better if the two metals do NOT easily mix up ("eutectic alloy")
- electron beam welding often works well

NOTE: soft soldering does not produce thermally conducting joint, since tin based soldering agents are superconducting at low temperatures (unless high magnetic field is applied).

In good joint electric contact resistance is of order $10\ n\Omega cm^2$ at 4 K (WF gives an estimate for thermal conductivity).



Yin and Hakonen, RSI 1991

$$R' = 0.55 \pm 0.16\ \mu\Omega\ \mathrm{mm}^2,$$

$=5.5\ n\Omega cm^2$

Kapitza resistance

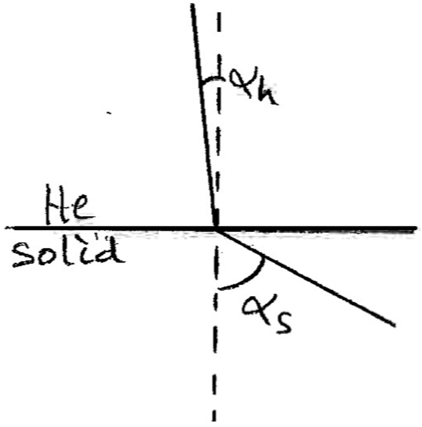
Acoustic mismatch theory

Thermal resistance between helium and solid substances:

* heat is transferred by phonons

* most of the phonons are reflected at the interface

Snell's law: $$\frac{\sin\alpha_h}{\sin\alpha_s} = \frac{v_h}{v_s} \sim \frac{200\ \text{m/s}}{4\ \text{km/s}}$$



=> critical angle $$\alpha_c = \arcsin\left(\frac{v_h}{v_s}\right) \sim 3^o$$

fraction of phonons hitting the critical cone: $$f = \frac{1}{2}\sin^2\alpha_c = \frac{1}{2}\left(\frac{v_h}{v_s}\right)^2 \sim 10^{-3}$$

\+ only part of their energy is transmitted: $$t = \frac{4 z_h z_s}{(z_h + z_s)^2} \approx \frac{4\rho_h v_h}{\rho_s v_s} \sim 2\times10^{-3}$$

(Here, $z_x = \rho_x v_x$ is acoustic impedance)

$$ft = \frac{2\rho_h v_h^3}{\rho_s v_s^3}$$

very small fraction of phonon energy ever crosses the boundary: $f\,t < 10^{-5}$

Kapitza resistance (cont.)

$$\frac{\dot{Q}}{A} \approx ft \frac{U}{V} v_h$$

$$C = \frac{12}{5} \pi^4 N_A k_B \left(\frac{T}{T_D}\right)^3$$

$$ft = \frac{2 \rho_h v_h^3}{\rho_s v_s^3}$$

Bose-Einstein distribution gives the energy flux $\frac{\dot{Q}}{A} = \frac{\pi^2 k_B T^4 \rho_h v_h}{30 \hbar^3 \rho_s v_s^3}$

thus the Kapitza resistance is

$$R_K = \frac{\Delta T}{\dot{Q}} = \frac{15 \hbar^3 \rho_s v^3}{2 \pi^2 k^4 T^3 A \rho_h v_h}$$

when $\Delta T << T$

and $R_K \propto \frac{1}{A T^3}$

This holds well when $T \sim 10 \ldots 200$ mK $\quad A R_K T^3 \sim 10^{-2} \ \frac{\mathrm{m}^2 \mathrm{K}^4}{\mathrm{W}}$

Both at higher and lower temperatures the experimentally determined boundary resistance values are smaller than this!

Some **factors omitted**:

- surface quality
- van der Waals forces (atomically thin layer of solid He on any surface)
- etc.

Kapitza resistance (cont.)

Contact area can be increased by sintering fine metal powder on a metal surface

- silver or platinum powder or mixture of these (particle size typically 40 ... 1000 nm)
- compressed to filling ratio ~ 0.5 ... 0.7 and heated up to ~ 160 ... 200 ^{o}C, so that a porous cake is obtained
- if made on copper, it is necessary to electroplate the surface by thin layer of silver
- effective surface area typically **~ 2 m^2/g**

Phonon spectrum of such sintered powder deviates from that of bulk substance

- longest possible phonon wavelength in the grains is ~ size of the grains
- interconnected grains support "softer" phonons with long wavelengths and slower speed of sound – density of states of these can be very high

$$\frac{dN}{d\omega} = \frac{3V\omega^2}{2\pi^2 v^3}$$

- T -dependence of the Kapitza resistance is lower than T^{-3} (experimental fact)
- thermal coupling between helium and bulk metal may also become limited by the thermal conductivity of He within the tiny pores of the sinter (mean free path cannot be larger than the pore size) or poor conductivity of the sinter material. Therefore, the sinter cake should not be thicker than ~ 1 mm

For ^{3}He there is a magnetic channel for thermal boundary coupling due to **nuclear spin** (I = 1/2). This requires that also the solid contains magnetic moments (such as CMN).

Kapitza resistance between He and plastics is usually less than that to metals (smaller difference in densities and speeds of sound)

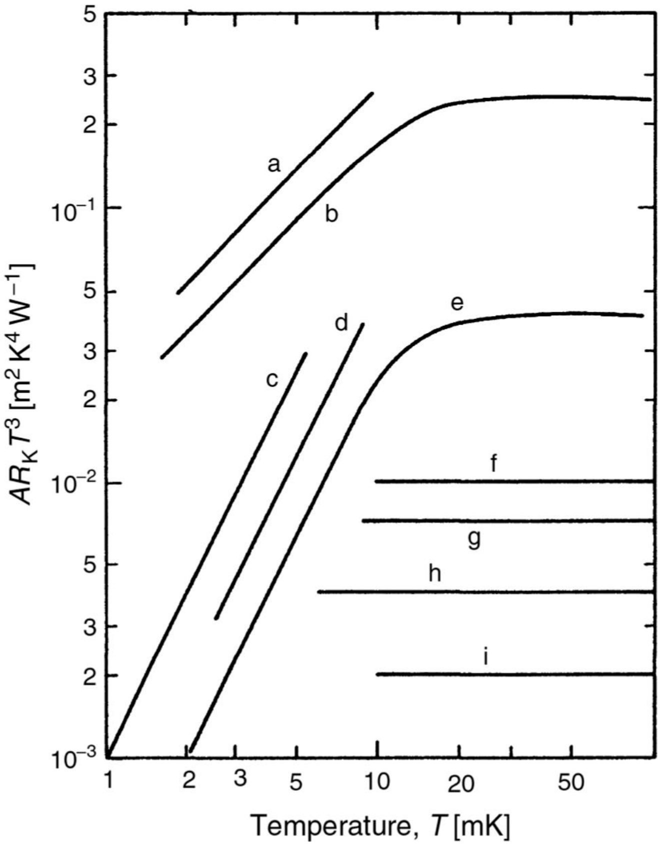
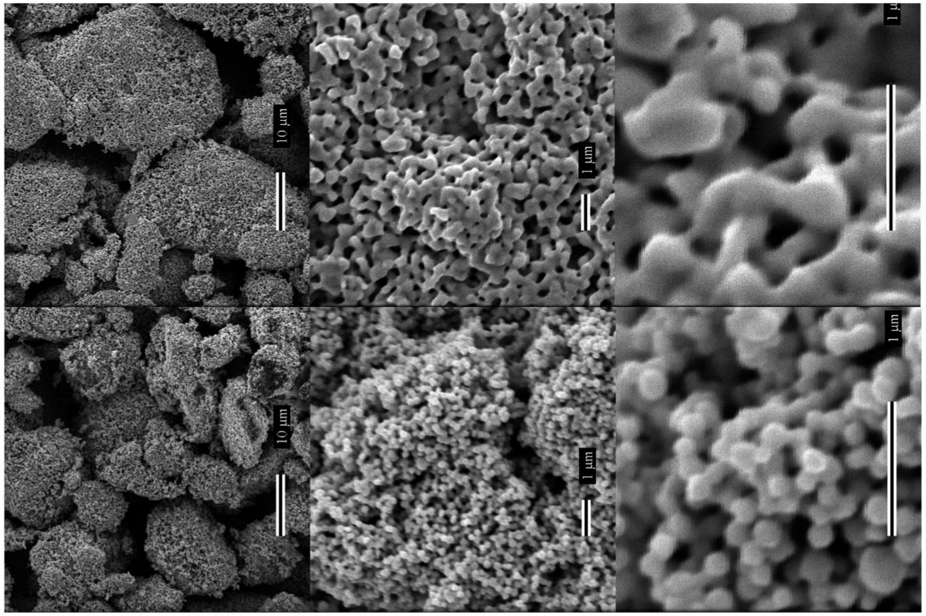


Fig. 4.9. Thermal boundary resistance R_{K} (multiplied by AT^3) between liquid ^{3}He or a liquid $^3\mathrm{He}-^4\mathrm{He}$ mixtures and various solids as a function of temperature. The data are for (**a**) mixture to 400 Å Ag sinter ($25\,T^{-2}$); (**b**) mixture to 700 Å Ag sinter ($16\,T^{-2}$ at <10 mK); (**c**) ^{3}He to 700 Å Ag sinter ($1{,}000\,\mathrm{T}^{-1}$); (**d**) ^{3}He to 400 Å Ag sinter ($470\,\mathrm{T}^{-1}$); (**e**) ^{3}He to 1 µm Ag sinter ($200\,\mathrm{T}^{-1}$ at <10 mK); (**f**) mixture to CuNi ($10^{-2}\,\mathrm{T}^{-3}$); (**g**) mixture to brass ($7 \times 10^{-3}\,\mathrm{T}^{-3}$); (**h**) mixture to 7 µm thick Kapton foils ($4 \times 10^{-3}\,\mathrm{T}^{-3}$); (**i**) mixture to Teflon tubing with 0.1 mm wall thickness ($2 \times 10^{-3}\,\mathrm{T}^{-3}$); (the data in brackets are AR_{K}) [4.73, this paper gives listings of the original works]

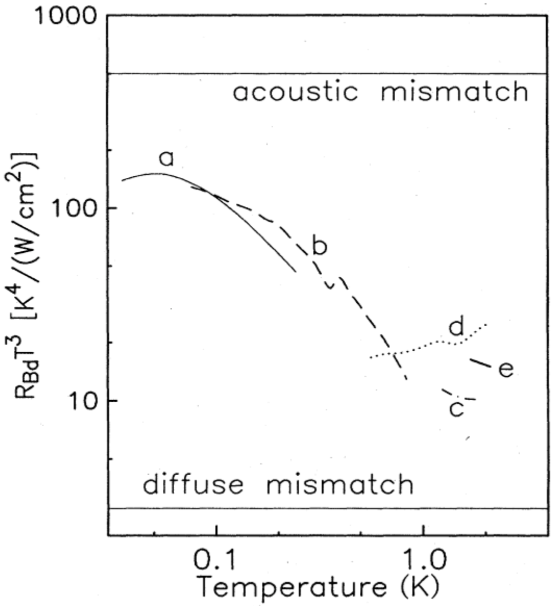
m2008.05.07
7g, D 25 mm x 3.3 mm, 500 kg
--- 200 C, 20 min --->
D 23.8 mm x 3.1 mm
0.49 m^2/g

Bad area/vol; good conductivity



Good area/vol; enough cond?

m2008.05.13
7g, D 25 mm x 3.3 mm, 500 kg
--- 155 C, 10 min --->



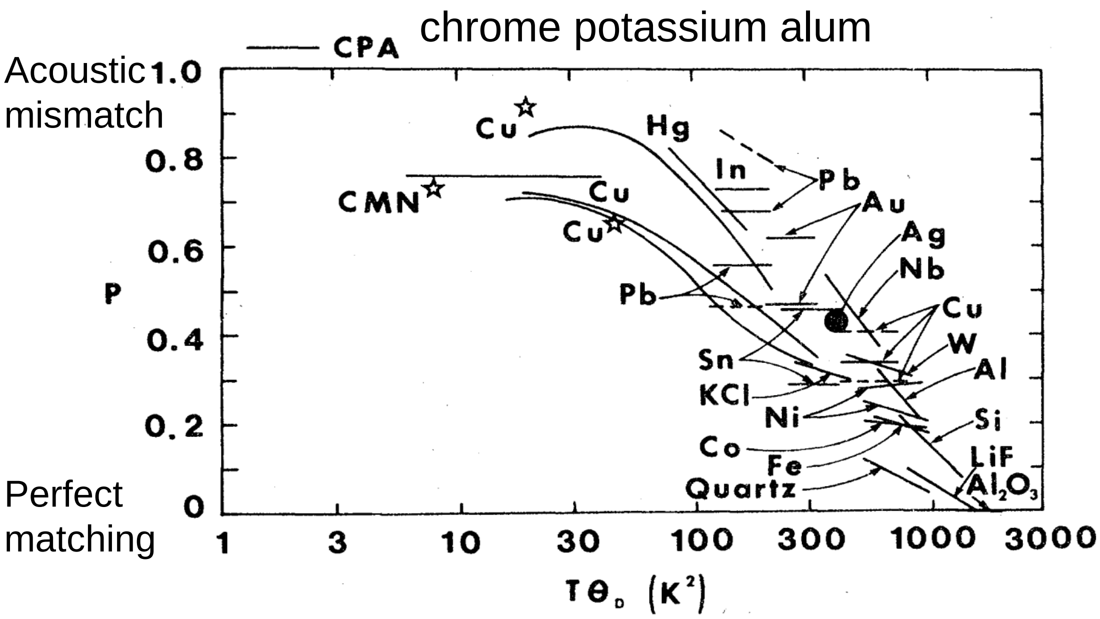
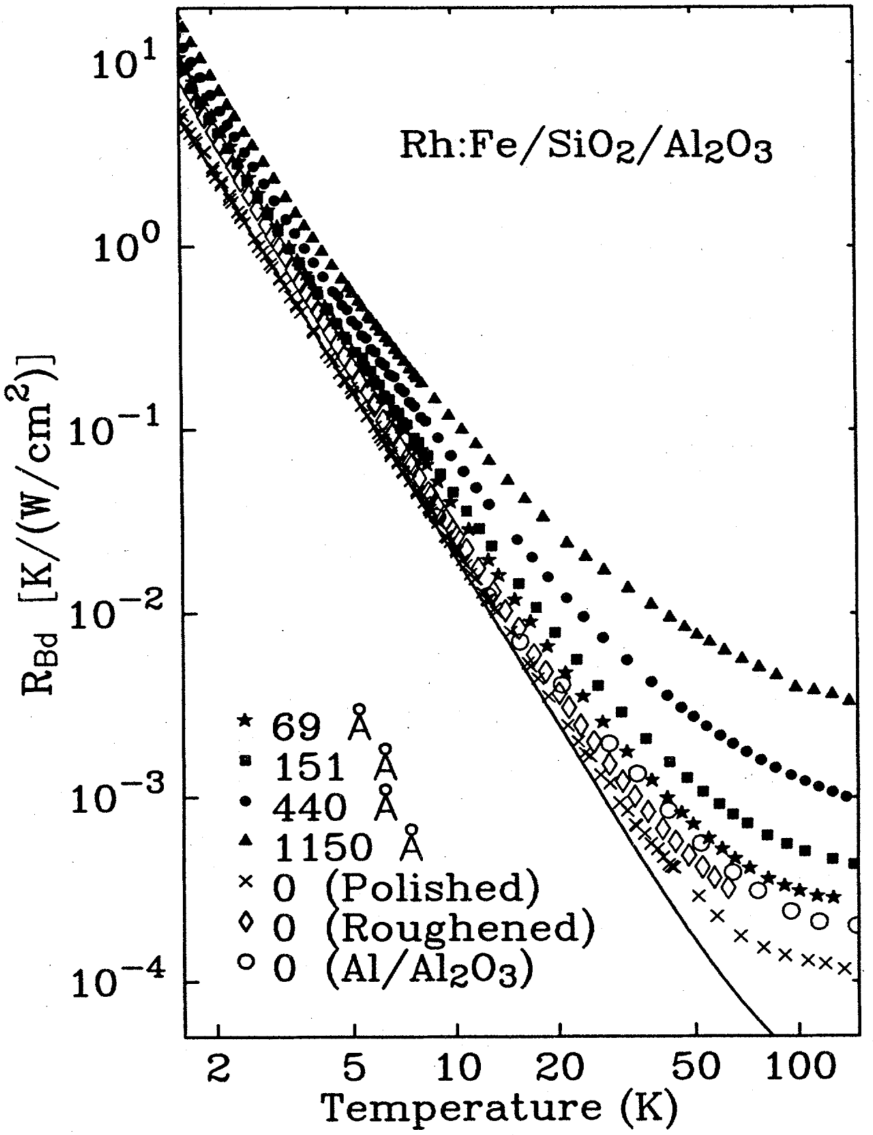


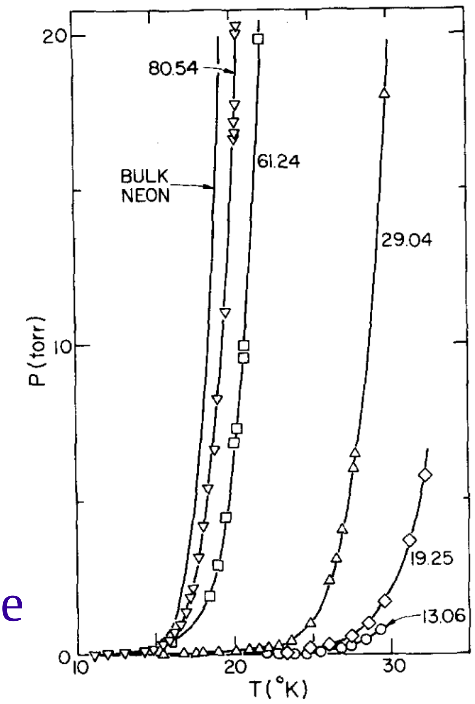
FIG. 18. Plot of the reduced thermal boundary resistance P as a function of $T\theta_D$. The stars indicate measurements between the solid and liquid ^{3}He. From Harrison (1974). The data are from: CPA (Vilches and Wheatley, 1966), CMN (Harrison and Pendrys, 1973), Cu (Challis, Dransfeld, and Wilks, 1961; Johnson and Little, 1963, Anderson, Connolly, and Wheatley, 1964; Anderson and Johnson, 1972); Hg (Neeper, Pearce, and Wasilik, 1967), In and Al_2O_3 (Gittleman and Bozowski, 1962), Pb (Challis, 1962; Guan, 1962; Challis and Cheeke, 1965), Au (Johnson and Little, 1963; Whelan and Osborne, 1968), Ag (Clement and Frederking, 1966), Sn (Gittleman and Bozowski, 1962; Guan, 1962), Nb and Al (Mittag, 1973), Ni (Guan, 1962; Cheeke, Hebral, and Richard, 1973), Co and Fe (Cheeke, Hebral, and Richard, 1973), quartz (Challis, Dransfeld, and Wilks, 1961), W, Si, and LiF (Johnson and Little, 1963), KCl (Johnson, 1964).



Thermal boundary resistance
E. T. Swartz and R. O. Pohl
Rev. Mod. Phys. **61**, 605 (1989)

Boundary resistance to solids with high Debye temperatures, like sapphire or LiF, will drop relative to the acoustic mismatch model at lower temperatures than will the boundary resistance to softer solids, such as lead or mercury.

Heat exchange gases



Used in the inner vacuum space during cooling from RT to ~ 10 K

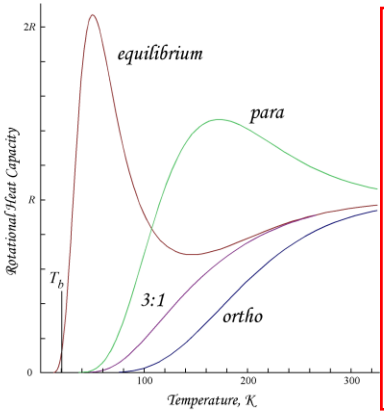
RT –> LN2, neon is good choice

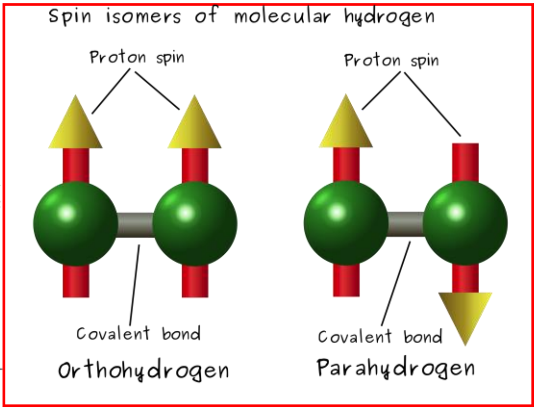
- easy to pump at liquid nitrogen temperature (liquefies only at 27 K)
- ~ 1 mbar is sufficient to achieve best possible thermal exchange
- possible residual gas solidifies well before liquid helium temperature

LN2 –> 10 K, no perfect solution, some available options:

- **H_2 is not good**; solidifies already at 20 K, residual hydrogen would give rise to time dependent heat leak because of ortho–para conversion (see Pobell's book)
- **^{4}He is not good**; helium is used for leak testing and no ^{4}He should be in the VJ. Also, residual He can form a superfluid film with extremely high conductivity
- **^{3}He will do**; critical point < 4 K, so it can be pumped out more efficiently than anything else, ~ no harm to leak testing. Very expensive, but the amount is very small (~ 0.1 mbar @ 80 K × ~ 10 l × ~ 2000 €/l/bar @ 300 K => ~ 10 €)

At 10 K internal refrigeration by He circulation can be started (most enthalpy removed)





Superconducting heat switch

Thermally connect/disconnect nuclear demag. stage and dilution fridge for precool

Thermal conductivity of metals in the **superconducting state** is poor. Only phonons remain to carry heat, when $T < T_c/10$ – $\kappa_s \propto (T/T_D)^3$

Normal state can be switched on by applying magnetic field $B > B_c$

– $\kappa_n \propto T/T_F$

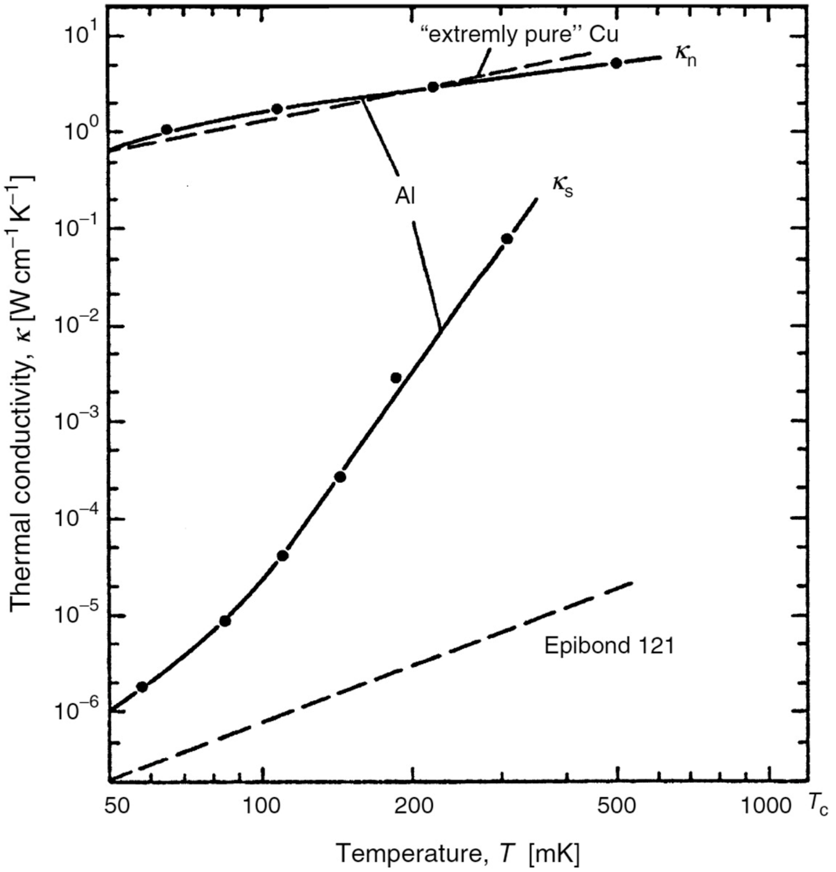
switching ratio $s = \kappa_n/\kappa_s \sim \mathbf{0.05\ (\boldsymbol{T_D/T})^2}$ applies for pure metals at $T < T_c/10$

Material candidates: – Sn, In, Zn, Pb, ...

Best results have been achieved using **aluminum**

- good conductivity in normal state (high purity, easily RRR > 1000)
- high $\boldsymbol{T_D}$ **= 430 K** – poor (*phonon*) conductivity in the SC state
- convenient magnitude of critical magnetic field, $\boldsymbol{B_c}$ **= 10.5 mT**
- $T_c = 1.2$ K, thus can be used below $T < 100$ mK efficiently
- PROBLEM: difficult to join with other metals, hard oxide layer develops fast;
  diffusion welding Al–Cu works reasonably well (~ 500 °C for 15 min)
- use thin foils (~ 0.1 mm) (phonon mean free path & eddy currents due to dB/dt)
- must acknowledge **trapping of magnetic field in superconductors**

Fig. 4.1. Thermal conductivity κ of Al in the normal–conducting state (compared to κ of Cu) and in the superconducting state (compared to κ of the dielectric Epibond 121) at $T < 50\,\mathrm{mK}$ [4.19]



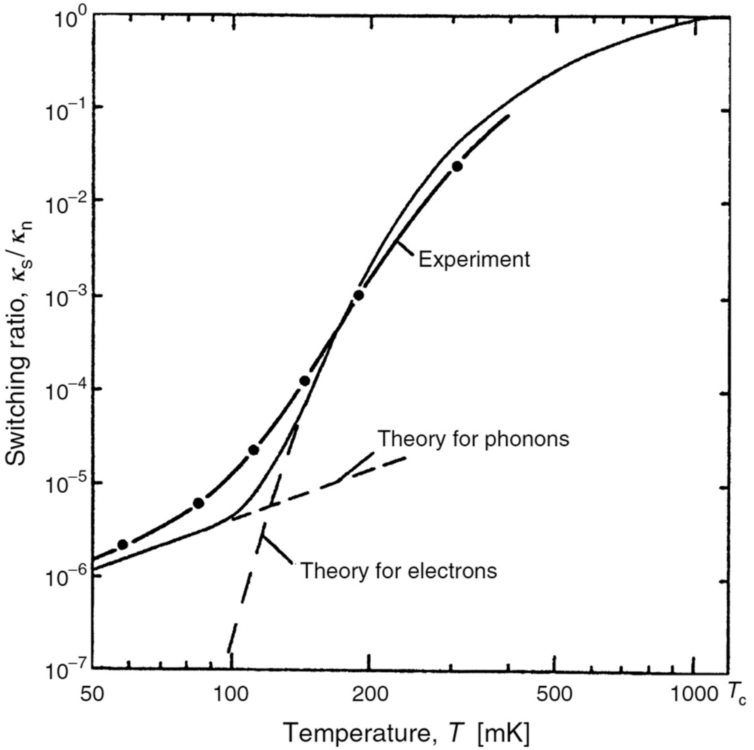


Fig. 4.2. Switching ratio κ_s/κ_n of the thermal conductivity of Al compared to theoretical predictions [4.19]

Summary

Thermal contraction: to be accounted in supports & design

– metals typically contract 0.2 ... 0.4 % (RT–LT)

– plastics and most dielectrics 1 ... 2 % (RT–LT)

Heat load from electrical wiring: resistivity & superconductivity

Residual gas < 10^{-6} mbar: charcoal pumps in vacuum & superinsulation

Thermal contact: copper & annealing & area & joints between two materials

Kapitza resistance: acoustic mismatch theory (also diffusive scattering used)

$$ft = \frac{2\rho_h v_h^3}{\rho_s v_s^3} \qquad R_K = \frac{\Delta T}{\dot{Q}} = \frac{15\hbar^3 \rho_s v_s^3}{2\pi^2 k_B^4 T^3 A \rho_h v_h}$$

– compensate Kapitza resistance with surface area using sinter; large T_D

Heat exchange gas: high T – Ne; low T – ^{3}He works; H_2 has problems

Superconducting heat switches: Al good – high T_D & convenient B_c ~10 mT