CRYOGENIC CS

590B F08

Sergey L. Bud’ko

Motivations
History
Materials
Design
Cryogenics
The branches of physics and engineering that involve the study of very low temperatures, how to produce them, and how materials behave at those temperatures.

Cryonics
The emerging medical technology of cryopreserving humans and animals with the intention of future revival.
cryo-surgery and veterinary medicine

cryo-transport of natural gas

100 t LH₂ + 600 t LO₂

research - physics

medicine

space

Novel Materials and Ground States
Historical cryogenics events

1850  Mechanical refrigeration first applied
1877  Cailetet & Pictet liquefied O₂ (90 K).
1892  James Dewar developed the vacuum flask
1908  Kamerlingh Onnes liquefied He (4.2 K).
1911  K. O. discovered superconductivity.
1926  Giauque reached 0.25 K by magnetic cooling.
1946  Collins developed commercial He liquefier.
1995  Connell & Wiemann achieved Bose-Einstein condensation at 2×10⁻⁸ K.
Some typical low temperatures

<table>
<thead>
<tr>
<th>Temperature</th>
<th>Celsius</th>
<th>Absolute</th>
</tr>
</thead>
<tbody>
<tr>
<td>Tropics</td>
<td>45</td>
<td>318</td>
</tr>
<tr>
<td>Human body</td>
<td>37</td>
<td>310</td>
</tr>
<tr>
<td>Room temperature</td>
<td>20</td>
<td>293</td>
</tr>
<tr>
<td>Ice point</td>
<td>0</td>
<td>273</td>
</tr>
<tr>
<td>Salt + water (cryogen)</td>
<td>-18</td>
<td>255</td>
</tr>
<tr>
<td>Antarctic winter</td>
<td>-50</td>
<td>223</td>
</tr>
<tr>
<td>Solid carbon dioxide</td>
<td>-78</td>
<td>195</td>
</tr>
<tr>
<td>Liquid oxygen</td>
<td>-183</td>
<td>90</td>
</tr>
<tr>
<td>Liquid nitrogen</td>
<td>-196</td>
<td>77</td>
</tr>
<tr>
<td>Liquid helium</td>
<td>-269</td>
<td>4</td>
</tr>
<tr>
<td>Absolute zero</td>
<td>-273</td>
<td>0</td>
</tr>
</tbody>
</table>
Logarithmic temperature scale

LOWEST TEMPERATURES RECORDED

CRYOGENICS

ATMOSPHERIC TEMPERATURES

HIGH TEMP.

PLASMAS

FUSION

HIGH ENERGY PARTICLES

LOWEST SUPERFLUID $T_c$

$^3$He

$^4$He

$^4$He

N$_2$

H$_2$O

W

BOILING POINTS

$K$

$10^{-8}$ $10^{-6}$ $10^{-4}$ $10^{-2}$ $1$ $10^2$ $10^4$ $10^6$ $10^8$ $10^{10}$ $10^{12}$

Novel Materials and Ground States
### Characteristic temperatures of low-energy phenomena

<table>
<thead>
<tr>
<th>Phenomenon</th>
<th>Temperature</th>
</tr>
</thead>
<tbody>
<tr>
<td>Debye temperature of metals</td>
<td>few 100 K</td>
</tr>
<tr>
<td>High-temperature superconductors</td>
<td>~ 100 K</td>
</tr>
<tr>
<td>Low-temperature superconductors</td>
<td>~ 10 K</td>
</tr>
<tr>
<td>Intrinsic transport properties of metals</td>
<td>&lt; 10 K</td>
</tr>
<tr>
<td>Cryopumping</td>
<td>few K</td>
</tr>
<tr>
<td>Cosmic microwave background</td>
<td>2.7 K</td>
</tr>
<tr>
<td>Superfluid helium 4</td>
<td>2.2 K</td>
</tr>
<tr>
<td>Bolometers for cosmic radiation</td>
<td>&lt; 1 K</td>
</tr>
<tr>
<td>Low-density atomic Bose-Einstein condensates</td>
<td>~ μK</td>
</tr>
</tbody>
</table>
Materials change at low temperatures

Structural phase transition (brittle-ductile - Ti alloys)

CW paramagnetism, long range magnetic order, spin glass

Superconductivity (annoying traces of it - phosphor bronze, Ti-alloys, many solders)

Freezing/glassing (liquids, oils, greases)

Condensation/liquefaction (gases)

Change of mechanical properties (rubber, plastics)

see appendix
Cooling with cryogenic liquids

Useful range of cryogens

- Helium
- Hydrogen
- Neon
- Nitrogen
- Argon
- Oxygen

T [K]
Cooling with cryogenic liquids

Vapour pressure at cryogenic temperatures
### Cooling with cryogenic liquids

#### Normal boiling point and latent heat of fluids

<table>
<thead>
<tr>
<th>Fluid</th>
<th>Normal Boiling Point (K)</th>
<th>Latent Heat (kJ/kg)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Water</td>
<td>373</td>
<td>2256</td>
</tr>
<tr>
<td>Ethylene</td>
<td>169</td>
<td>481</td>
</tr>
<tr>
<td>Krypton</td>
<td>120</td>
<td>116</td>
</tr>
<tr>
<td>Methane</td>
<td>111</td>
<td>512</td>
</tr>
<tr>
<td>Xenon</td>
<td>110</td>
<td>99</td>
</tr>
<tr>
<td>Oxygen</td>
<td>90</td>
<td>213</td>
</tr>
<tr>
<td>Argon</td>
<td>87</td>
<td>162</td>
</tr>
<tr>
<td>Nitrogen</td>
<td>77</td>
<td>199</td>
</tr>
<tr>
<td>Neon</td>
<td>27</td>
<td>86</td>
</tr>
<tr>
<td>Hydrogen</td>
<td>20</td>
<td>443</td>
</tr>
<tr>
<td>Helium</td>
<td>4.2</td>
<td>21</td>
</tr>
</tbody>
</table>
**Cooling with cryogenic liquids**

Amount of cryoliquids (in liters) to cool 1 kg of metal if only latent heat (latent heat + enthalpy of the gas) is used.

<table>
<thead>
<tr>
<th>Cryoliquid</th>
<th>Temperature change (K)</th>
<th>Al</th>
<th>SS</th>
<th>Cu</th>
</tr>
</thead>
<tbody>
<tr>
<td>$N_2$</td>
<td>300 - 77</td>
<td>1.0 (0.63)</td>
<td>0.53 (0.33)</td>
<td>0.46 (0.28)</td>
</tr>
<tr>
<td>$^4$He</td>
<td>77 – 4.2</td>
<td>3.2 (0.2)</td>
<td>1.4 (0.1)</td>
<td>2.2 (0.16)</td>
</tr>
<tr>
<td>$^4$He</td>
<td>300 – 4.2</td>
<td>66 (1.6)</td>
<td>34 (0.8)</td>
<td>32 (0.8)</td>
</tr>
</tbody>
</table>

Use LN$_2$ to precool the equipment (caution and patience needed)

Make use of the enthalpy of the cold He gas (very slow initial transfer, end of the transfer tube close to the bottom)
Heat sources

(i) Heat conduction

Thermal conductivity integrals of selected materials [W/m]

<table>
<thead>
<tr>
<th>From vanishingly low temperature up to</th>
<th>20 K</th>
<th>80 K</th>
<th>290 K</th>
</tr>
</thead>
<tbody>
<tr>
<td>OFHC copper</td>
<td>11000</td>
<td>60600</td>
<td>152000</td>
</tr>
<tr>
<td>DHP copper</td>
<td>395</td>
<td>5890</td>
<td>46100</td>
</tr>
<tr>
<td>1100 aluminium</td>
<td>2740</td>
<td>23300</td>
<td>72100</td>
</tr>
<tr>
<td>2024 aluminium alloy</td>
<td>160</td>
<td>2420</td>
<td>22900</td>
</tr>
<tr>
<td>AISI 304 stainless steel</td>
<td>16.3</td>
<td>349</td>
<td>3060</td>
</tr>
<tr>
<td>G-10 glass-epoxy composite</td>
<td>2</td>
<td>18</td>
<td>153</td>
</tr>
</tbody>
</table>
Heat sources

(ii) Heat radiation

- Wien’s law
  - Maximum of black body power spectrum
    \[ \lambda_{\text{max}} T = 2898 \, [\mu\text{m.K}] \]
- Stefan-Boltzmann’s law
  - Black body
  - "Gray" body
  - "Gray" surfaces at \( T_1 \) and \( T_2 \)

Emissivity of technical materials at low temperatures

<table>
<thead>
<tr>
<th>Material</th>
<th>Radiation from 290 K Surface at 77 K</th>
<th>Radiation from 77 K Surface at 4.2 K</th>
</tr>
</thead>
<tbody>
<tr>
<td>Stainless steel, as found</td>
<td>0.34</td>
<td>0.12</td>
</tr>
<tr>
<td>Stainless steel, mech. polished</td>
<td>0.12</td>
<td>0.07</td>
</tr>
<tr>
<td>Stainless steel, electropolished</td>
<td>0.10</td>
<td>0.07</td>
</tr>
<tr>
<td>Stainless steel + Al foil</td>
<td>0.05</td>
<td>0.01</td>
</tr>
<tr>
<td>Aluminium, as found</td>
<td>0.12</td>
<td>0.07</td>
</tr>
<tr>
<td>Aluminium, mech. polished</td>
<td>0.10</td>
<td>0.06</td>
</tr>
<tr>
<td>Aluminium, electropolished</td>
<td>0.08</td>
<td>0.04</td>
</tr>
<tr>
<td>Copper, as found</td>
<td>0.12</td>
<td>0.06</td>
</tr>
<tr>
<td>Copper, mech. Polished</td>
<td>0.06</td>
<td>0.02</td>
</tr>
</tbody>
</table>

polished surfaces radiation shields
Heat sources

(iii) Residual gas conduction

getters on cold walls

- Molecular regime
  - At low gas pressure $\lambda_{\text{molecule}} >> d$
  - Kennard's law
  - Conduction heat transfer proportional to pressure, independant of spacing between surfaces
    $\Omega$ depends on gas species
  - Accommodation coefficient $\alpha(T)$ depends on gas species, $T_1$, $T_2$, and geometry of facing surfaces

(iv) Thermoacoustic (Taconis) oscillations

standing waves in gas filled tubes with temperature gradient and closed warm end

change in geometry
introduction of damping element

use of Taconis oscillations
Superinsulation

Multi-layer insulation (MLI)

- Complex system involving three heat transfer processes
  - $Q_{MLI} = Q_{rad} + Q_{sol} + Q_{res}$
  - With $n$ reflective layers of equal emissivity, $Q_{rad} \sim 1/(n+1)$
  - Due to parasitic contacts between layers, $Q_{sol}$ increases with layer density
  - $Q_{res}$ due to residual gas trapped between layers, scales as $1/n$ in molecular regime
Typical heat fluxes at vanishingly low temperature between flat plates [W/m$^2$]

<table>
<thead>
<tr>
<th>Description</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Black-body radiation from 290 K</td>
<td>401</td>
</tr>
<tr>
<td>Black-body radiation from 80 K</td>
<td>2.3</td>
</tr>
<tr>
<td>Gas conduction (100 mPa He) from 290 K</td>
<td>19</td>
</tr>
<tr>
<td>Gas conduction (1 mPa He) from 290 K</td>
<td>0.19</td>
</tr>
<tr>
<td>Gas conduction (100 mPa He) from 80 K</td>
<td>6.8</td>
</tr>
<tr>
<td>Gas conduction (1 mPa He) from 80 K</td>
<td>0.07</td>
</tr>
<tr>
<td>MLI (30 layers) from 290 K, pressure below 1 mPa</td>
<td>1-1.5</td>
</tr>
<tr>
<td>MLI (10 layers) from 80 K, pressure below 1 mPa</td>
<td>0.05</td>
</tr>
<tr>
<td>MLI (10 layers) from 80 K, pressure 100 mPa</td>
<td>1-2</td>
</tr>
</tbody>
</table>
Dewar flask

(a) Glass  
(b) Glass  
(c) Metal
Sir James Dewar, FRS (1842-1923)

Developed *Cordite* (smokeless gunpowder alternative) – with Sir Frederick Abel, 1889

Described several formulae for benzene, 1867

Studied physiological action of light, with Prof. J. G. McKendrick (conditions of retina)

Spectroscopical observations, with Prof. G.D. Living

Public liquefaction of oxygen and air, 1884

Cir. 1892 – vacuum flask

Liquid hydrogen, 1898, solid hydrogen, 1899

Gas-absorbing powers of charcoal, 1905

Surface tension of soap bubbles, during and after WW1

Last publication in 1923 (at 80 yrs age)
Storage dewars

Fig. 5.7. Commercial storage vessel for liquid $^3$He. (A: connection for transfer tube, B: overflow valve, C: safety valve, D: manometer, E: vacuum and safety valves, F: gas valve, G: getter material, H: adsorbent material, I: superinsulation)
Dipper

Fast, cheap (and dirty)

Can use storage dewar

Poor temperature control (easy to improve somewhat)
Continuous flow cryostat

- **Cheap**
- **Fast cooling**
- **Relatively low He consumption**
- **Relatively easy optical access**
- **Base temperature?**
- **Mediocre temperature control**
- **Hard to incorporate high field**
Going below 4.2 K - $^4$He

![Graph 1: Pressure vs. Temperature (K)]

- Pressure (kPa) vs. Temperature (K)
  - Superfluid $^4$He
  - Normal $^4$He
  - $H_2$
  - $Ne$
  - $Ar$
  - $O_2$
  - Triple point

![Graph 2: Remaining fraction of liquid helium (%) vs. Temperature (K)]

- Remaining fraction of liquid helium (%) vs. Temperature (K)
  - From 65% to 100%

![Diagram: Test Dewar System]

- Test dewar
- Mechanical vacuum pump
- Thermal insulating vacuum jacket
- Pressure regulator
- Pump vent to helium recovery system
- Specimen holder
- Liquid helium
- Magnet support tubes
- Radiation baffles
- Fill port
- Measurement probe
- Pressure relief valve

*Novel Materials and Ground States*
4He cryostat with variable temperature insert

- Workhorse for LT measurements
- Good temperature control
- Easy magnet accommodation
- Allows for variety of sample holders
- Not so cheap
- Uses fair amount of cryogens
Helium transfer

He level detectors: acoustic, resistive (SC), capacitive.
How to get there? Closed cycle refrigerators...

Gifford-McMahon (G-M) or Pulse Tube (PT) cycle

To make a refrigeration cycle, need a substance, the entropy of which depends on some other variable than temperature

Pressure of gas: Compression/expansion cycle
Magnetization of solid: magnetic refr. cycle

$\Delta Q_1$: heat absorbed at $T_1$
$\Delta Q_2$: heat rejected at $T_2$

$\Rightarrow$ Refrigeration cycle A B C D
Close cycled refrigerators

**GOOD**
- Turn-key
- Easy optical (etc...) access
- No liquid cryogens
- Small footprint (but remember compressor)
- Sometimes reasonably priced

**NOT SO GOOD**
- “Usually” base $T > 4\ K$
- Noise, vibrations
- “Small” cooling power at low temperatures
- Long term maintenance cost?
- Use in high fields?

Somewhat difficult to tweak
One can buy
14T/100mK measurements platform (combination of PT cryocooler and magnetic refrigeration stage)
SQUID magnetometer with installed liquefaction unit.
Safety

Temperature
Pressure
Energy (SC magnets)

Think
Learn
Use PPE (!?)

Ice blocks
Relief valves
He level - magnet quench
Glass dewars
Liquid oxygen

RECOVER!
Reading materials

*Matter and methods at low temperatures*
Author: Frank Pobell; Springer, 2007

*Experimental techniques in low-temperature physics*
Author: Guy K. White; Clarendon Press, 1979

*Experimental low-temperature physics*
Author: Anthony Kent; American Institute of Physics, 1993

*Experimental techniques in condensed matter physics at low temperatures*
Author: Robert C Richardson; Eric N Smith; Addison-Wesley Pub. Co., 1988

*Experimental techniques for low-temperature measurements: cryostat design, material properties, and superconductor critical-current testing*
Author: J. W. Ekin; Oxford University Press, 2006
Electrical resistivity and heat capacity

Figure 2.1 Specific heat as a function of temperature for several types of material. Typical behaviors are illustrated for metals (aluminum, beryllium, and copper), semiconductors (carbon and silicon), an amorphous inorganic (Pyrex glass) (Corruccini and Gniewew, 1960), and for an organic polymer (polyvinyl chloride) (Chang, 1977). A single point is shown for aluminum in the superconducting state at 1 K.

appendix

Novel Materials and Ground States
Yield strength of engineering materials

(1) 2024-T4 Aluminum
(2) Beryllium copper
(3) K-monel
(4) Titanium
(5) 304 Stainless steel
(6) C1020 Carbon steel
(7) 9% Nickel alloy steel
(8) Teflon
(9) Invar-36

appendix
Variation of Young's modulus with temperature

Fig. 10.1 Variation of Young's modulus with temperature. (From Ledbetter [10.1].)
A; silver 99.999% pure,
B; copper (OFHC),
C; coalesced copper,
D; electrolytic tough pitch,
E; single crystal aluminum,
F; machining copper,
G; aluminum 1100 F,
H; aluminum 6063-T5,
I; phosphorus deoxidized copper,
J; aluminum 2024-T4,
K; brass
Thermal conductivity - low

A; 50-50 Pb-Sn solder
B; steel, SAE 1020
C; beryllum copper
D; constantan
E; monel
F; silicon bronze
G; inconnel
H; 347 stainless steel
I; fused quartz
J; teflon (PTFE)
K; polymethylmethacrylate (PMMA)
L; nylon

appendix
Thermal contraction

appendix

Novel Materials and Ground States