PRESSURE – II-III

590B F09

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(Сергей Леокадьевич Будько)

Research (low temperature) cells
Pressure synthesis
Pressure generation

Media:
gas
liquid
solid
Technical achievements (high pressure) associated with P. W. Bridgman (reminder)

The Bridgman unsupported area high pressure system:

\[ P_1 \times \text{Area}_1 = P_2 \times (\text{Area}_1 - \text{Area}_2) \]

\[ \text{Area}_1 - \text{Area}_2 \leq \text{Area}_1 \]

Hence \( P_2 \geq P_1 \)

Hence viscous material at \( P_2 \) seals \( P_1 \)

Illustrating the general principle of the method for giving external support to the pressure vessel in such a way that support increases automatically with the increase of internal pressure.

Novel Materials and Ground States
Pressure generation - materials

**Tungsten carbide** – somewhat magnetic, very hard, brittle (+A)

**Tool steel** – magnetic, brittle

**Maraging steel** - magnetic

**MP35N** – slightly magnetic

**Ni-Cr-Al (40ХНЮ-ВИ)** – less magnetic, not easy to get

**Cu-Be alloy (C17200)** – low susceptibility but DOE does not like it

**Ti – alloys** – low susceptibility but superconductivity and ductile-brittle transition

**Cu-3%Ti alloy** – very low susceptibility but try to get it

**Sapphire (A)**

**Moissanite (A)**

**Diamond (A)**

* A = anvils
Pressure generation - intensifier

Complex
Requires heavy duty tubes going into dewar
DOE regulations – "pressure vessels"
Can have "true hydrostatic" gas/liquid pressure

A schematic layout of an intensifier system: (B) to experimental cell, (C) low-pressure compressor (70 MPa), (I) intensifier cylinder, (M) resistance manometer, (P) main oil ram and press, (S) separator cylinder, (U) compression cylinder, and (V) valves. Here, $R$ is the multiplication ratio of the cylinders.
Helium pressure cell

General view of high pressure helium gas installation: 1) intensifier, 2) inlet valve, 3) check valve, 4) check valve holder, 5) manganin bomb, 6) connector for high pressure tubing, 7) high pressure tubing 8) relieving valve, 9) oil screw pump, 10) experimental cell.
Pressure generation – ice bomb

Lazarev & Kan, 1944

Water as pressure medium
1.7 kbar (? 1.9 kbar)
Very simple
One pressure point

Some of us thought that the ice bomb belongs to *Kunstkammer* ... but

Body – Ti alloy
Pressure medium – Ga
P ~ 4 kbar
Pressure clamp

Can be very simple (no leads are needed, Teflon capsule filled with liquid medium)

\[ P_{\text{max}} \sim 1 \text{ GPa} \]

Ames Lab (MP35N or CrNiAl)

EasyLab (Be-Cu)

Novel Materials and Ground States
Pressure clamp

For a long time was (and still is) a workhorse for 20 kbar high pressure/low temperature work

Similar units: USA, Europe, USSR

Relatively simple
Compact
Non-magnetic
Teflon capsule

Cir. 1953, 20 kbar

Simple clamp cell for use to 3 GPa. A—compressing shaft, B—piston (tungsten carbide), C—cylinder (Be—Cu), D—thermocouple, E—electrode plug, F—backing plate, G—lock nut, H—holder, I—supporting ring, J—Teflon bucket, K—binding ring, L—leads. (Reproduced by kind permission of the American Institute of Physics.)

Extended support

D>>d

Novel Materials and Ground States
Pressure clamp - seals

Cone!

0.8 mm diameter; ~ 12 wires

Electrical feedthroughs

STYCAST 2850 FT works fine
(do not try 5 min Epoxy)
Pressure clamp - windows

Not easy but doable.

Also – T-shaped cell was in use
Pressure decreases with temperature decrease down to ~100 K

$\Delta P < 4$ kbar, decreases with pressure

Materials/media dependent...
Two-layer cell

30+ kbar, possible optical access

USSR – 1980 (Efim Itskevich et al.) ; later UK, USA, Japan

1–fixing nut, 2-inner cylinder (insert), 3-piston, 4-Bridgman packing, 5-shaft, transmitting force from press, 6-thrust plate, 7-specimen, 8-transmitting medium, 9-stop, 10-support shell, 11-stop packing.

Novel Materials and Ground States
Bridgman cell

- gasket - pyrophyllite
- medium - steatite

$P_{\text{max}} \approx 120$ kbar, quasihydrostatic, resistance, TEP, heat capacity
Modified Bridgman cell

want to have pressure in liquid

Simple adaptation of the Bridgman high pressure technique for use with liquid media

E. Colombier and D. Braithwaite
Département de Recherche Fondamentale sur la Matière Condensée, SPMSM, CEA-Grenoble, 17 rue des Martyrs, 38054 Grenoble, France

**TABLE I.** Total number of tests and typical pressure values and pressure gradient ranges with each medium tested. Values given are for a standard procedure where the cell was loaded first to 30 kN, then to 40 kN. The anvil diameter is 3.5 mm and the inner chamber diameter is 1.4 mm.

<table>
<thead>
<tr>
<th>Medium</th>
<th>Number of tests</th>
<th>$P$ (GPa) ($F = 40$ kN)</th>
<th>$\Delta P$ (GPa)</th>
<th>Remarks</th>
</tr>
</thead>
<tbody>
<tr>
<td>FC84</td>
<td>3</td>
<td>4.4-4.8</td>
<td>0.1-0.35</td>
<td>Sometimes crystallizes</td>
</tr>
<tr>
<td>1:1 FC84:FC87</td>
<td>6</td>
<td>4.9</td>
<td>0.2</td>
<td></td>
</tr>
<tr>
<td>1:1 FC 70:FC77</td>
<td>9</td>
<td>4.9</td>
<td>0.15</td>
<td></td>
</tr>
<tr>
<td>DC 200 1 cS</td>
<td>2</td>
<td>4.4</td>
<td>0.2-0.3</td>
<td>Sometimes crystallizes</td>
</tr>
<tr>
<td>DC 200 20 cS</td>
<td>2</td>
<td>/</td>
<td>/</td>
<td>Always explodes (too high viscosity, low compressibility?)</td>
</tr>
<tr>
<td>DC 200 1:1 1cS: 0.65 cS</td>
<td>2</td>
<td>5.6</td>
<td>0.25</td>
<td></td>
</tr>
<tr>
<td>1:1 pentane: isopentane</td>
<td>4</td>
<td>4.5</td>
<td>0.15</td>
<td></td>
</tr>
<tr>
<td>4:1 Methanol:Ethanol</td>
<td>2</td>
<td>/</td>
<td>/</td>
<td>Wires break (compressible and dissolves epoxy)</td>
</tr>
<tr>
<td>Nitrogen</td>
<td>3</td>
<td>/</td>
<td>/</td>
<td>Wires break (too compressible?)</td>
</tr>
<tr>
<td>Daphne oil</td>
<td>2</td>
<td>/</td>
<td>/</td>
<td>Always explodes (too high viscosity, low compressibility?)</td>
</tr>
</tbody>
</table>

(a) side view and (b) top view of the chamber with sample (right) and lead (left).

Talk to Estelle or watch her working

$P_{\text{max}} \sim 60$ kbar

Novel Materials and Ground States
Modified Bridgman cell

No Teflon ring

Sectional drawing of the pressure cell. The scale of the vertical axis is doubled with respect to the horizontal axis. 1, anvil; 2, gasket formed by lower and upper pyrophyllite rings; 3, epoxy; 4, sample; 5, support for wire; 6, 10 μm gold wire; 7, 25 μm gold wire.

Top view of the pressure cell containing three organic conductors and a lead manometer (in the top left corner).

P_{max} \sim 60 \text{ kbar}
Modified Bridgman cell

A newly developed high-pressure cell by using modified Bridgman anvils for precise measurements in magnetic fields at low temperatures

T. Nakanishi, N. Takehita, and N. Miya
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$P_{max} \sim 60 \text{ kbar}$
“Chechevitsa” (lentil) pressure cell

Larger volume
Possible radial access
Circ. 1950s
HPPI - USSR
Modified toroid cell

(a) liquid-filled ampoule for studies at hydrostatic pressure and high temperatures; (b) assembly for magnetic measurements placed in the top part of an ampoule; (c) sample with the stain gauge bonded;

1-gasket (lithographic stone), 2-teflon ampoule, 3-lid, 4-thermal insulation (asbestos), 5-heater, 6-sample, 7-thermocouple, 8-coil system for magnetic measurements, 9-strain gauge, 10-manganin pressure gauge, 11-electrical leads.

Lev Khvostantsev et al. – 1970s, Vladimir Sidorov et al. 1980s
Diamond Anvil Cell (DAC)

Schematic diagrams of the essential mechanisms of a DAC, showing two alternative layouts. The drive force goes through the alignment mechanisms in (a); in (b) the tilt alignment mechanism is not under load.

Diamond mounting arrangements: (a) a clamp ring; (b) a riveted or staked soft ring (e.g., annealed copper) in a fixed ring; (c) a soft ring in a counterbore; and (d) glue - a ring of epoxy resin around the diamond.
Four tilt and X–Y alignment mechanisms are shown schematically; (a) the traditional hemispherical; and (b) semi-cylindrical rockers; (c) the stand-off screws; and (d) rotating wedges.

alignment – very important

Sectional view of the simple diamond anvil cell.
Many different designs

$P_{\text{max}}$ – few Mbar

Gaskets – usually metal (Re, Inconnel, …)

Media – gas, liquid, solid (NaCl)

Measurements – optics, X-ray, susceptibility, resistance, heat capacity, etc.
High pressure synthesis

- heater
- thermocouple
- container
- pressure medium
- load

Solid (soft) pressure medium (beware of pressure and temperature gradients)

No size restrictions (want to have sample space as large as possible)

Cooling + high T materials

Novel Materials and Ground States
High pressure synthesis – piston-cylinder

35 kbar, 2000°C
High pressure synthesis – multianvil

60 kbar, 2000°C (cubic)
250 kbar, 2000°C (Walker)

Novel Materials and Ground States
High pressure synthesis – belt/girdle

Drickamer cell

Need radial support
High pressure synthesis – toroid

High Pressure Physics Institute (Moscow), cir. 1970
Calibration of HP furnace

SiO$_2$

stishovite
coesite
quartz
Ames Laboratory – Novel Materials and Ground States Group:

100 ton press
Cubic module
Walker module

MgB$_2$ single crystals
NdFeAs(O/F) single crystals

*Ask Matt Tillman to give you a tour*
The "Bridgman method" is based on the invention of Tammann in Göttingen early in the century. He used the "gradient-freeze method" as it is called now, in which crystals can be grown by directional solidification in the temperature gradient region of a furnace whose average temperature is decreased gradually. Bridgman has added mechanical movement of the crucible to this Tammann method. Others, such as Stockbarger, have made important contributions to the Bridgman-method.
High Pressure Furnace Setup

Rockland Research Corporation

Appendix II - courtesy of Matt Tillman
Cubic Anvil

Appendix II - courtesy of Matt Tillman
The Sample Cell

- Pyrophyllite cube ~24mm on a side
- Carbon heater
- BN cup and cap
- Pyrophyllite plugs

- 3.3 GPa
- 0.164 cm³
- 1900 °C

Appendix II - courtesy of Matt Tillman
Calibration (Temperature)

Temperature Calibration Curve for High Pressure Furnace

Slope = 1.09

Measured
Fit
Unity
Calibration (Pressure)

- Bi Transition

3342 PSI = 2.52 GPa
3737 PSI = 2.70 GPa

Appendix II - courtesy of Matt Tillman
Calibration (Pressure)

- Quartz/Coesite Transition

<table>
<thead>
<tr>
<th>Oil Pressure (PSI)</th>
<th>Temperature (°C)</th>
<th>Pressure (GPa)</th>
<th>Result</th>
</tr>
</thead>
<tbody>
<tr>
<td>7500</td>
<td>969</td>
<td>3.05</td>
<td>Coesite</td>
</tr>
<tr>
<td>7500</td>
<td>1212</td>
<td>3.22</td>
<td>Coesite</td>
</tr>
<tr>
<td>7500</td>
<td>1091</td>
<td>3.14</td>
<td>Coesite</td>
</tr>
<tr>
<td>7500</td>
<td>2063</td>
<td>3.81</td>
<td>Quartz</td>
</tr>
<tr>
<td>7500</td>
<td>1577</td>
<td>3.47</td>
<td>Quartz</td>
</tr>
<tr>
<td>7500</td>
<td>1455</td>
<td>3.39</td>
<td>Coesite</td>
</tr>
<tr>
<td>6500</td>
<td>969</td>
<td>3.05</td>
<td>Coesite</td>
</tr>
<tr>
<td>6500</td>
<td>1212</td>
<td>3.22</td>
<td>Coesite</td>
</tr>
<tr>
<td>6500</td>
<td>1455</td>
<td>3.39</td>
<td>Quartz</td>
</tr>
<tr>
<td>6500</td>
<td>1334</td>
<td>3.30</td>
<td>Quartz &amp; Coesite</td>
</tr>
</tbody>
</table>

Calibration (Pressure)

Oil Pressure to Sample Pressure Calibration Curve for The High Pressure Furnace

Quartz/Coesite Transition

Bi Calibration

Appendix II - courtesy of Matt Tillman
Sample Packing (MgB$_2$)

- Distilled Mg
- B Powder (and dopant)
- BN Powder
- Hot Pressed BN

Appendix II - courtesy of Matt Tillman
Informal summary

Pressure work (at least beyond piston-cylinder) cannot be done as a hobby (still one can (i) buy/build a cell and have fun; (ii) use collaboration; (iii) use Users Facilities)

Requires good machine shop, and infrastructure for sample preparation

Research infrastructure for pressure measurements (optics/resistance/susceptibility/x-ray)

Possible non-hydrostaticity should be always on the table

Mere pressure derivatives can tell you a lot if bandstructure is of interest

Sometimes interesting things happen
Reading

PW Bridgman, The physics of high pressure
CA Swenson, Physics at high pressure (*you can talk to the author in our corridors*)
CC Bradley, High pressure methods solid state research
WB Holzapfel, NS Isaacs, High-pressure techniques in chemistry and physics
MI Eremets, High pressure experimental methods
many reviews, some useful