High-Pressure Techniques

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University of Edinburgh
Centre for Science at Extreme Conditions (CSEC)

- A multi-disciplinary Centre opened in 2002.

- Research interests range from fundamental physics, chemistry and biology, through geoplanetary and materials science, to engineering and technology.

- The members of the Centre represent the Schools of Physics, Chemistry, GeoSciences, Engineering, and Biological Sciences.
Outline

- The definition of pressure and the generic types of pressure cells
- Mechanical properties of materials
- Choosing the right materials
- Computer Aided Design software packages
- Testing the designs by Finite Element Analysis
- Examples of high-pressure designs:
  - Neutron and X-ray diffraction
  - Magnetic properties
What is pressure?

Pressure = Force / Area

\[ p = \frac{F}{A} \]

Different surface areas will result in different pressures for the same force.

Therefore, from the engineering point of view the problem of creating pressure splits into two tasks:

1. Generating the **force**, and
2. Defining the **area** over which it is applied
Generating static force

The devices for generating static force fall into two categories:

1) Opposed anvils (uniaxial force) - two surfaces pushing against each other.

2) Other:
   - Hydraulic pumps
   - Gas compressors
   - Hydrothermal

1000-ton hydraulic press

Miniature M-B DAC
Defining the surface area

So, if the opposed anvil is the most common way of creating the force then all the variety of the pressure cells is due to the difference in defining the surface area over which the force is applied.

1) Opposed anvil cells – up to several Mbar

2) Indenter cells – up to 5 GPa

[Diagram of opposed anvil and indenter cells]
Defining the surface area

3) Piston-cylinder cells – up to 3 GPa
4) Multi-anvil cells – up to 16 GPa
Stress in cylinders

- Stresses are maximal at the bore and decrease rapidly with \( r \) leaving most of the cylinder under low stress;
- Increasing wall thickness is no help – the ratio of the bore diameter to the outer diameter of the cylinder that would allow you to achieve the maximum possible pressure is 1:3

\[ \sigma_t \sim \frac{1}{r^2} \]

\[ \sigma_r \sim \frac{-1}{r^2} \]
The first opposed anvil cell

THE RESISTANCE OF 72 ELEMENTS, ALLOYS AND COMPOUNDS TO 100,000 KG/CM²

By P. W. BRIDGMAN

Received November, 1951

FIGURE 1. The apparatus for applying pressure to the resistance specimen. An enlarged section of the cell is shown in the lower part of the diagram.
Various opposed anvil designs…

…and these are just those built in our group!

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Why will you need to design your own pressure cells?

- The need to adapt the pressure cell to
  - your particular sample requirements (solid/liquid/gas sample, weakly/strongly scattering, weak/strong magnetic response, etc)
  - the rest of the sample environment / techniques (heaters, magnets, cryostats, neutron/synchrotron experiments, spectrometers, etc)

- Limited commercial availability of pressure cells
So how do you do it?

Decide on the **pressure limit / sample volume** you require

This will define the **type** of the pressure cell

Select the **materials** for the cell

Draw the **design** of the cell and its components

Test your design using finite-element **analysis**

Hand your drawings to the workshop for **manufacturing**
Materials selection

Some of the key properties to consider:

• Thermal (thermal expansion, thermal conductivity)
• Electrical (resistivity)
• Magnetic (magnetic susceptibility, permeability, etc)
• Optical (transparency)
• Chemical (reactivity, resistance to corrosion)

but most importantly – **mechanical**!
Key mechanical properties

Before we consider them we need to introduce Stress - a measure of the average amount of force exerted per unit area (not quite pressure!)

- Strength (tensile/compressive) - stress applied to a part at the point when it fails
- Elastic modulus – stress-to-strain ratio – the response of the material to stress in the elastic deformation regime
- Toughness – the resistance to fracture of a material when stressed
- Stiffness – force-to-deflection ratio - resistance of an elastic body to deflection or deformation
- Hardness – resistance to elements (indentation, scratch, etc)
- Fatigue resistance – the number of stress-cycles before failure
Stress – Strain Diagrams

1 - Ultimate Strength
2 - Yield Strength (elastic modulus)
3 - Rupture
4 - Strain hardening region
5 - Necking region
The Strength \( \sigma_f (\text{MPa or MN/m}^2) \)

For metals, when stress–strain curve for axial loading deviates by a strain of 0.2% from the linear-elastic line. It is the stress at which dislocations first move large distances, and is the same in tension and compression.

For ceramics and glasses \( \sigma_f \) depends strongly on the mode of loading. In tension, strength means the fracture strength. In compression it means crushing strength, typically 10 to 15 times larger than the fracture strength.
Young’s Modulus – Strength Diagram

- Metals
- Ceramics
- Composites
Metals in High-P Engineering

Good strength, ductility and formability, good electrical and thermal conductivity.

**Maraging steels** – iron-based alloys which are known for possessing superior strength and toughness without losing malleability / shapeability; resist corrosion and crack propagation.

<table>
<thead>
<tr>
<th>Maraging steel</th>
<th>Ultimate tensile strength (N/mm²)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Böhler W720 (Fe-Mo-Ni-Co)</td>
<td>2260</td>
</tr>
<tr>
<td>Aubert &amp; Duval 819AW (Fe-Mo-Ni-Cr)</td>
<td>1900</td>
</tr>
</tbody>
</table>

The yield strength of Böhler W720 (depends on heat treatment)

Austenitizing temperature (holding time 3 hours)

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Metals in High-P Engineering

**Superalloys** – Ni-, Co-, Co/Fe-based alloys with face-centered cubic crystal structure - excellent mechanical strength and creep resistance at high temperatures, good surface stability, and corrosion and oxidation resistance.

CrNiAl (Cr 39-41%, Al 3-4%, Ni balance): Yield strength - 2.3 GPa

**Beryllium-copper alloys** – excellent properties for low-temperatures (its strength even improves at low-T) and for magnetic applications (Cu and Be are diamagnetics => low magnetic susceptibility).

BERYLCO-25 (1.8-2.0% of Be, 0.6% of Co+Ni+Fe and balance copper) Yield strength – 1.6 GPa
Ceramics in High-P Engineering

Strong, electrical and thermal insulators, resistant to high temperature, but brittle.

<table>
<thead>
<tr>
<th>Material</th>
<th>Compressive strength (at room temperature)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Zirconia (ZrO$_2$)</td>
<td>2.1 GPa</td>
</tr>
<tr>
<td>Alumina (Al$_2$O$_3$)</td>
<td>2.9 GPa</td>
</tr>
<tr>
<td>Tungsten Carbide (WC)</td>
<td>7.0 GPa</td>
</tr>
<tr>
<td>Sintered diamond</td>
<td>15.0 GPa</td>
</tr>
</tbody>
</table>
New materials for pressure cells

![Material Tensile Strength](chart1)

![Material Compressive Strength](chart2)
Finding the properties of materials

1. Reference books (for well-known materials)
2. Manufacturers’ websites (especially for new materials)
3. Material property databases (on-line and off-line)
Computer Aided Design

CAD software allows you to
- Make 3D drawings of parts
- Combine individual parts into assemblies
- Convert 3D parts into 2D technical drawings
PPMS pressure cell design: DAC
PPMS pressure cell design: DAC
Computer Aided Design

CAD software is also great for producing drawings for presentations and animations for moving parts.

Small DAC mounted on a rotator

Most common CAD packages:
- Solid Edge
- Solid Works
- AutoCAD

The chances are your university will have a site licence for one of these.
Finite Element Analysis (FEA)

FEA is a computer simulation technique based on the finite element method (FEM), which is used for finding approximate solution of partial differential equations.

We use FEA to calculate:
- Stress distribution
- Deformation under load
- Temperature gradients
- Magnetic field distribution

A continuum with infinite degrees of freedom is represented by assemblage of elements with finite degrees of freedom.
Finite Element Analysis (FEA)

Finite Element Method is a method for finding approximate solutions of partial differential equations. The approximation is based on discretization - replacing infinite dimensional problem with the one with a finite dimensional one.

Visualization of how a car deforms in an asymmetrical crash.

Fracture mechanics of bone.

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Finite Element Analysis: Step-by-Step

1. Create or import a part to be modelled; simplify the model using the symmetry
2. Assign it the properties of a particular material
3. Break down the whole structure into elements – adjust the mesh size according to stress concentration
4. Apply loads and boundary conditions
5. Solve for displacements
6. Postprocess - find reaction forces, stresses, deformations, etc
Finite Element Analysis

FEA can be used to model:
• Stress distribution
• Deformation under load
• Temperature gradients
• Magnetic field distribution
• Fluid dynamics
• etc

Major FEA packages:
• ANSYS
• Abacus
• Nastran (Simulia)

All can be run on a desktop PC!
Specialised workshop equipment for making high-pressure instruments

Laser drilling, cutting and welding centre

Diamond polishing and faceting machine

Gun drilling machine (ружейные сверла)
Piston-cylinder cell for inelastic neutron scattering

- Temperature (K)
- Pressure (GPa)

- Ferromagnetism
- Superconductivity

- $T_C$
- $10 T_{SC}$

- UGe$_2$
Clamped cell for INS studies

Section view of the 3D FEA model of the pressure cell body with maximum principal stress distribution as calculated for the sample pressure of 2 GPa.

Cross-sectional view of the pressure cell assembly
HP DAC for LT XRD: Challenges

- **Dimensions** – the pressure cell should be miniaturised to reduce its thermal mass

- **Geometry** – should comply with the restrictions imposed by the cryogenics (gas flow rate and geometry)

- **Materials selection** – not only mechanical properties (e.g. strength, toughness etc) but also thermal properties (thermal conductivity, thermal expansion) need to be taken into account
Finite Element Analysis:
Equivalent stress distribution at the diamond seat and the bottom plate when the pressure of 5 GPa is applied on the diamond interface.
(Miniature Merrill-Bassett cell)$^2$
(Miniature Merrill-Bassett cell)
(Miniature Merrill-Bassett cell)$^2$

Pressure cell mass: 23 g
High pressure room temperature data: single crystal NaCl (circled), diamond spots and tungsten gasket (faint lines)
(Miniature Merrill-Bassett cell)²

<table>
<thead>
<tr>
<th>LT-XRD DAC</th>
<th>MB DAC</th>
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<tbody>
<tr>
<td>Material</td>
<td>BeCu alloy</td>
</tr>
<tr>
<td>Diamond anvil</td>
<td>Boehler – Almax design</td>
</tr>
<tr>
<td>Diamond culet (µm)</td>
<td>600</td>
</tr>
<tr>
<td>Opening angle</td>
<td>60</td>
</tr>
<tr>
<td>Cell diameter (mm)</td>
<td>23</td>
</tr>
<tr>
<td>Material volume (mm³)</td>
<td>~2900</td>
</tr>
<tr>
<td>Mass (gr)</td>
<td>23</td>
</tr>
</tbody>
</table>
Mini M-B DAC on the Bruker APEX diffractometer

\[ T_{\text{Cryostream}} = 80 \text{ K}, \]
\[ T_{\text{DAC}} = 150 \text{ K} \]

(as measured using NaCl EoS)

Pressure drop on cooling from 300 K to 150 K – 0.1 GPa
The DAC is sensitive to the alignment – the windows are frost-free but the temperature stability can be affected during a omega-scan.
Mini M-B DAC on Station 9.8 at SRS

Powder NaCl

P = 9.8 kbar; T=20°C (293 K)  P = 9.8 kbar; T = -170°C (103 K)

The increased number of spots at low-T is due to ice formation.

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Turnbuckle pressure cell
Equivalent stress in the *Boehler-Almax* diamond anvil and in the beryllium copper support screw under 25 GPa applied to the culet – maximum stress in the steel: 2 GPa.
Equivalent stress in the *Boehler-Almax* diamond anvil and in the beryllium copper support screw under 25 GPa applied to the culet – maximum stress in the steel: 2 GPa.
Turnbuckle pressure cell: Design

Pressure cell mass: 2.7 g
Turnbuckle pressure cell: Operation
Turnbuckle pressure cell – Alignment

Top view - no fringes

Side view - no wedge gap
Turnbuckle pressure cell – Testing with Cryostream

Pressure drop on cooling from 300 K to 120 K: 1.22 to 1.07 GPa (ΔP = 0.11 GPa) (as measured using NaCl EoS)
Turnbuckle pressure cell for MPMS

The cell can generate and hold loads in excess of 6 kN between the end-nuts.
Recommended Reading

High-pressure cells (books)

High-pressure cells (journals)
*High Pressure Research* (T&F): [http://www.tandf.co.uk/journals/titles/08957959.asp](http://www.tandf.co.uk/journals/titles/08957959.asp)

Engineering Textbooks
*Mechanical Engineer’s Data Handbook* by J. Carvill (Butterworth Heinemann, 3rd edition, 2001)
References

Steels / alloys manufacturers
Böhler Steels: http://www.bohler-edelstahl.com/
Aubert & Duval: http://www.aubertduval.com/
NGK Metals: http://www.ngkmetals.com/

CAD packages
Solid Edge: http://www.solidedge.com/
Solid Works: http://www.solidworks.com/
AutoCAD: http://www.autodesk.co.uk/

FEA packages
ANSYS: http://www.ansys.com/
Abacus: http://www.simulia.com/
Nastran: http://www.mscsoftware.com/products/msc_nastran.cfm
Thank you