Pressure cells for soft matter at LLB, Which solutions can be used? Where are the limitations and the problems encountered?

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Task 2: "Kinetics and Dynamics"

Saclay – 28th January 2013

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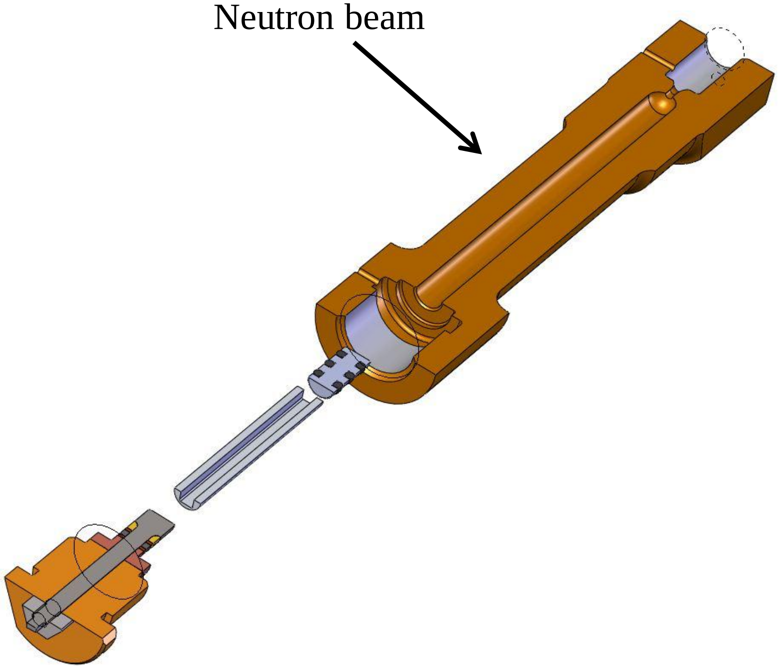
NMI3-FP7-JRA-II-WP20 "Advanced neutron tools for Soft and Bio-Materials"

Pressure cells for soft matter at LLB
Type I (hollow cylinder)



The pros:

- Easy to seal
- Low acquisition costs
- The Bridgman-seal is tight up to very high pressure > 30kbar
- No size problems

The cons:

- Above 10kbar the cone-cone or radius-cone seal becomes to be tricky
- The data analysis for SANS measurements with the cylinder geometry is more complicated to do than a flat geometry

Pressure cells for soft matter at LLB
Type I (hollow cylinder)

Title Drawing number	Maximum allowable pressure [bar]	Volume in the beam [cm³] Ø and H in [mm]	HP cell volume [cm³]	Dimension D_{ex} / D_{int} [mm]	Temperature range [K]	Material	Application area	Q-range [Å⁻¹]	Transmission [%]
High pressure cell 8kbar for inert gases N°: 1001	8000	1,42cm³ Ø 6mm ; 50mm	2,16	Ø18 / Ø6	1-400	CuBe2 AT(TF00)	Diffraction, inelastic and quasi-elastic diffusion		
High pressure cell 10kbar for inert gases N°: 2001	10000	0,99cm³ Ø 5mm; 50mm	1,53	Ø20,5 / Ø5	1-400	CuBe2 AT(TF00)	Diffraction, inelastic and quasi-elastic diffusion		
Cellule HP Mibemol Alu N°: 18400	6500	2,51cm³ Ø 8mm; 50mm	4,45	Ø31,5 / Ø8	1-300	Aluminium 7049A T651	Diffraction, inelastic and quasi-elastic diffusion	TOF: 0,4-2 at 6Å G6.1: 0,25-2,36 at 4,7Å	75% at 6Å
Cellule HP gaz 6kbar N°: 19500	6000	1,42cm³ Ø 6mm; 50mm	1,63	Ø15 / Ø6	1-400	CuBe2 AT(TF00)	Diffraction, inelastic and quasi-elastic diffusion	G4.1: 0,2 – 3,5 at 2,42Å	
Cellule ronde HP Petits Angles N°: 18029	6000	0,21cm³ Ø5,2mm ; 10mm	0,23	Ø18 / Ø5,2	293	Zero scattering alloy TiZr 52.48	SANS Diffraction	Q> 0,07 G6.1 :0,12-2,36 at 4,7Å	38% at 6Å
Cellule ronde HP Petits Angles N°: 18029	2500	0,21cm³ Ø5,2mm ; 10mm	0,23	Ø18 / Ø5,2	293	Niobium pur	SANS Diffraction	Nb : Q≥ 2.10^{-2} above Q = 5.10^{-2} the spectrum is flat	78% at 6Å
Cellule Hélium HP 3 Axes	6000	4,52cm³ Ø12mm ; 40mm	4,52	Ø48 / Ø12	1-300	Aluminium 7049A T651	Diffraction, inelastic and quasi-elastic diffusion		
Cellule HP Mibemol 5mm 3Kbar TOF N°: 18553	3000	0,99cm³ Ø5mm; 50mm	1,71	Ø14 / Ø5	1-300	Aluminium 7049A T651	Diffraction, inelastic and quasi-elastic diffusion		
Cellule HP Mibemol 5mm 5,5Kbar TOF N°: 18551	5500	0,99cm³ Ø5mm ; 50mm	1,71	Ø18,1 / Ø5	1-300	Aluminium 7049A T651	Diffraction, inelastic and quasi-elastic diffusion		
Cellule HP CO2 supercritique N°: 18061	400	0,52cm³ Ø7,15 ; 13mm	0,67	Ø10 / Ø7,15	293-360	Niobium pur	SANS Diffraction		
Cellule HP Mibemol CuBe	7500	3,93cm³ Ø10; 50mm	6,79	Ø28 / Ø10	1-300	CuBe2 AT(TF00)	Diffraction Inelastic and quasi-elastic diffusion	TOF: Q:0,6-2	Calculated: 2Å: 15% 5,2Å: 6%

Type I: Overview Material pressure limits

The fully plastic state $(pi)_{v,pl}$ according to the *von-Mises* criterion, also known as maximum distortion energy criterion, for materials without hardening tendency:

$$(pi)_{v,pl} = \frac{2}{\sqrt{3}} * \sigma_F * \ln k \qquad k = \frac{d_{out}}{d_{in}}$$

Yield strength: σ_F *Diameter ratio*: **k** *Outer diameter*: d_{out} *Inner diameter*: d_{in}

Chosen values: *k=3;* *with d_{out}=18mm and d_{in}=6mm*

Materials suitable for soft matter and SANS experiments:

Alloy	Alloy properties with average values			Calculated values:	
	Yield strength [MPa]	Tensile strength [MPa]	Elongation at rupture [%]	Fully plastic state (burst pressure) [bar]	Yielding at the inner layer [bar]
Aluminium 7049A T6	580	650	8	7358	2977
CuBe2 AT (TF00) C17200	1242	1365	8	15756	6374
Ti52.5Zr47.5 zero-scattering alloy	690	840	9	8753	3541
Stainless steel: M30NW; X4CrNiMnMoN21.9.4 Solution annealed	450	860	40	5709	2309
Stainless steel: M30NW; X4CrNiMnMoN21.9.4 Cold worked	850	900	15	10783	4362
M5™ from AREVA (ZrNb1)	~300	~450	21	3806	1540
Nb, reactor grade,soft	110	226	50	1395	565

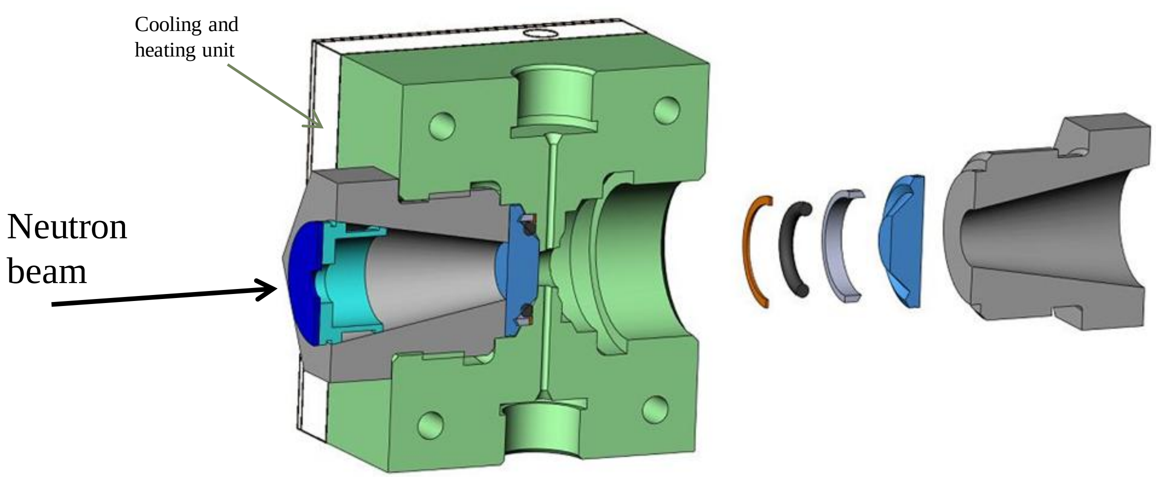
Materials probably not suitable for SANS experiments due to their grain size, too small

Stainless steel PH-type: Custom465®Stainless H1000	1510	1593	15	19155	7749
Russian alloy 40HNU-VI NiCrAl40.4	2000	2100	7	25371	10264

The influence of a changing *k*-value for a given material:

						***k*-value**
CuBe2 AT (TF00)	1242	1365	8	9941	5378	**2**
CuBe2 AT (TF00)	1242	1365	8	13141	6023	**2.5**
CuBe2 AT (TF00)	1242	1365	8	15756	6374	**3**
CuBe2 AT (TF00)	1242	1365	8	17966	6585	**3.5**
CuBe2 AT (TF00)	1242	1365	8	19881	6723	**4**
CuBe2 AT (TF00)	1242	1365	8	23082	6884	**5**

Pressure cells for soft matter at LLB
Type II (window type)



The pros:

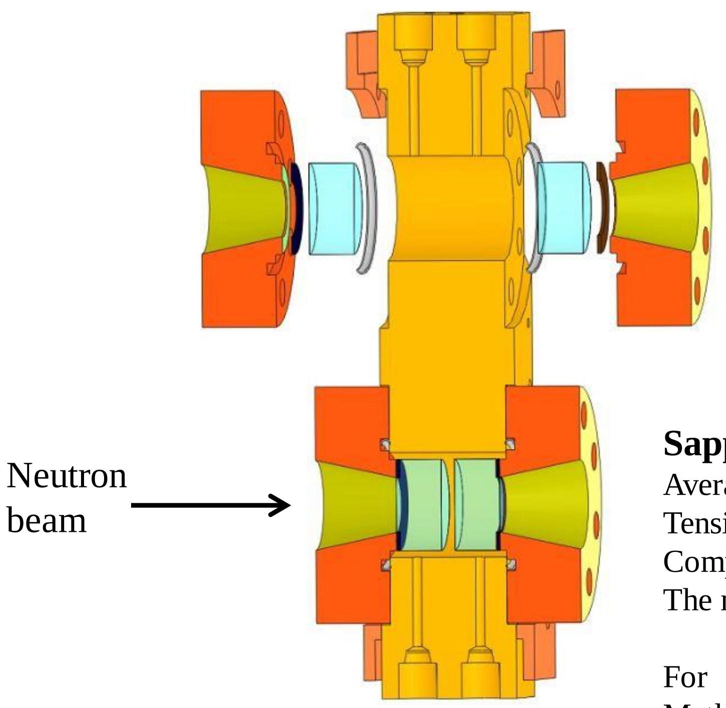
- Easy to clean
- Windows are easy to change
- Low window price
- Windows are from different materials
- Easy data analysis due to flat geometry

The cons:

- Low window opening
- Only for SANS
- Used for metallic windows

Window material	Maximum allowable pressure [bar]	Total window thickness [mm]	Transmission [%]	Q-range [Å^{-1}]
Aluminium 7049A T6	6300	12,2	76 at 13Å	>0,1
Aluminium 2017A	4500	12,2	94,8 at 6Å 78,5 at 13Å	>10^{-2}
Niobium pure	2700	10	80 at 6Å	≥ 2.10^{-2} above Q = 5.10^{-2} the spectrum is flat

Pressure cells for soft matter at LLB
Type III (windows, Poulter type)



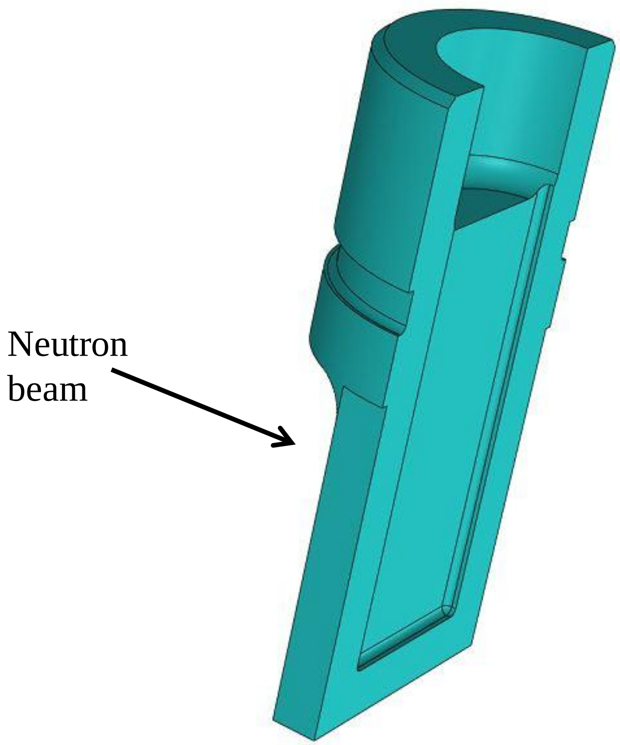
Sapphire
Average values for the crystal:
Tensile strength: 300MPa
Compressive strength: 3000MPa
The mechanical properties are axis dependent

For high pressure application the Heat Exchanger Method (HEM) growing method should be used and not the Verneuil's method since there is too much residual stress inside the crystal.

The pros:
- Transparent windows
- Opening 12mm up to 2000bar
- Easy data analysis due to flat geometry

The cons:
- The sapphires must be optical polished and without residual stress to withstand high pressure
- Only for SANS
- Fragile

Pressure cells for soft matter
Type IV (flat cell)



The pros:

- Large sample size in the beam
- Bridgman sealed
- Easy data analysis due to flat geometry

The cons:

- The resistance is lower than a hollow cylinder with the same wall thickness
- The edges produces local stress concentrations
- Machining is complicated without EDM
- EDM remelted surface layer must be taken off to have crack free material, difficult to do in the edges

Manufacturing difficulties due to electro erosion

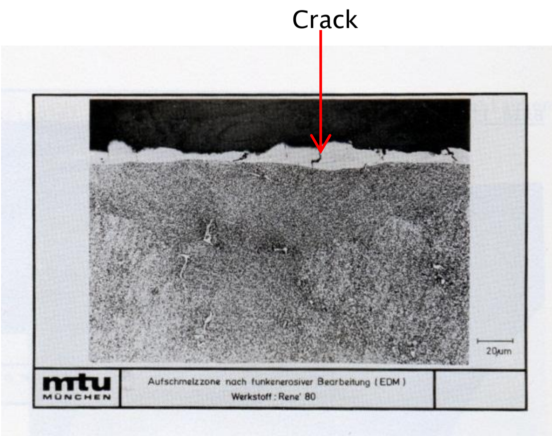


Figure:
Bunk/Hansen: Werkstoffe hoher Beanspruchung am Beispiel der Luftfahrt, Deutsche Gesellschaft für Metallkunde 1982, p,42
ISBN3-88355-055-8

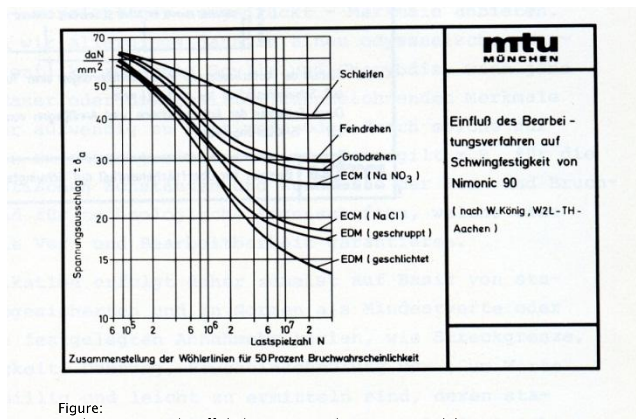


Figure:
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ISBN3-88355-055-8

Influence of the machining process on the cycle fatigue strength, technical processes:

Schleifen = grinding
Feindrehen = precision turning
Grobdrehen = rough turning
ECM = Electrochemical machining

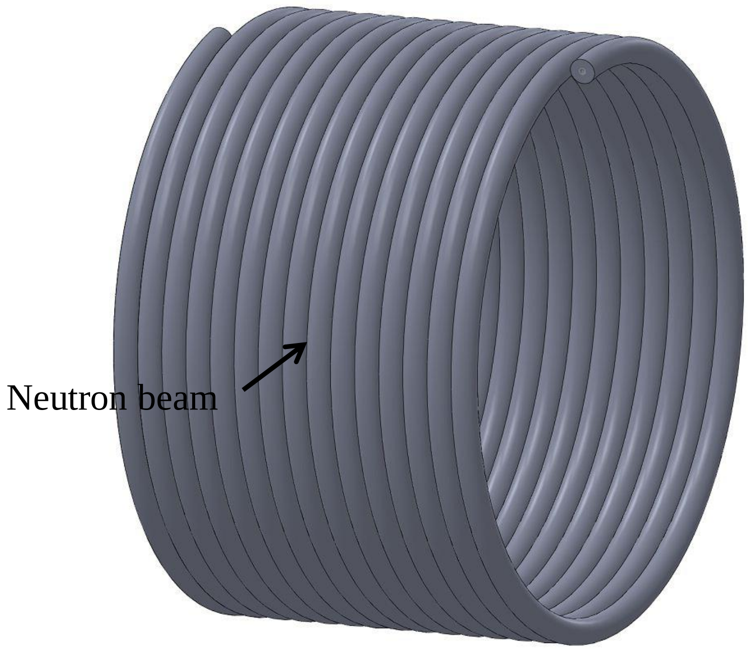
(In the ECM process is no tool wear, no sparks are created, mirror surface finishes can be achieved)

EDM = Electric discharge machining

Spannungsausschlag = stress amplitude
Lastspielzahl = number of load cycles

The EDM process gives the opportunity to realize complicated contours but the surface layer contains defects, cracks, which are reducing the cycle fatigue strength, if not removed.

Pressure cells for soft matter
Type V (high pressure capillary)



The pros:

- The high pressure capillary is easy to obtain
- Various sizes are available
- Up to 14kbar
- Easy to seal

The cons:

- Difficult to clean
- Is made from stainless steel, AISI 304 or 316, HP160, must be Co-free
- Minimum bending radius 5xd

Limitations on belonging material

Valves

Most of the inner parts of the valves are made from 17-4 PH stainless steel, with the outer part made from AISI316, if destilled or heavy water is used, corrosion can occur, therefore the stem material has to be adapted, by choosing another material.

For a long service life of the valve the high pressure should act on the stem and not the seal packing.

Pressure transducers

During the first running of the pressure sensor at high pressure > 8kbar a shift in the zero can often be observed

Fittings

Tubing and fittings which should be used on pressures > 10kbar must be degreased before assembling to seal well. Connecting and disconnecting of the fittings will result in leakage because the material is work hardening.