Status Report
Task2 Prototype of pressure cell for NSE and SANS

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Task 20.2.3

SANS measurements under pressure were performed
Part I:

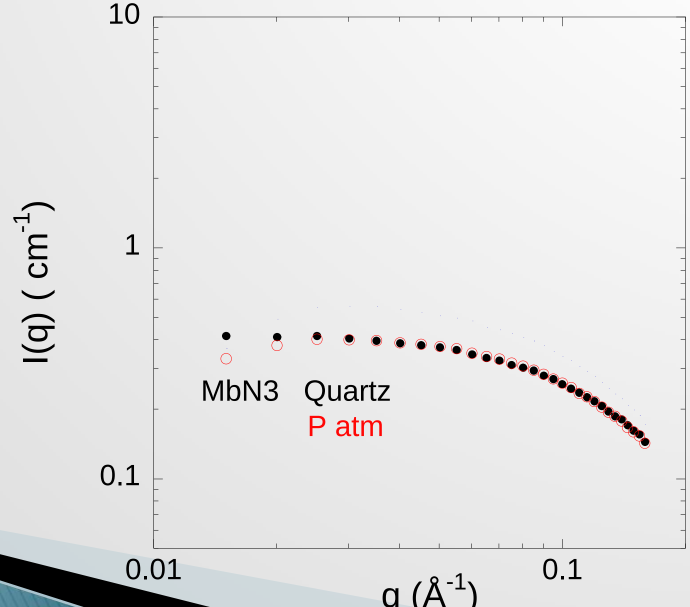
- Myoglobin was studied at 5000bar using our pressure cell with metallic windows

Windows: aluminium alloy 7049A T6, total thickness 12.2mm,

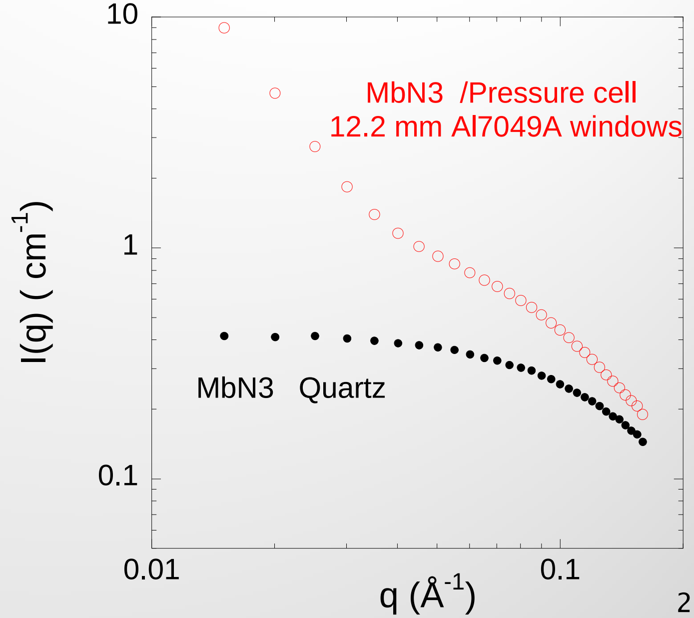
simple window thickness : 6.1mm

Sample size: Ø6mm e=4mm

Closing pressure 50bar
with data treatment



5000bar
without data treatment



Task 20.2.3

SANS measurements under pressure were performed – Part II:

- Apo-myoglobin was studied at 2000bar using our pressure cell with metallic windows

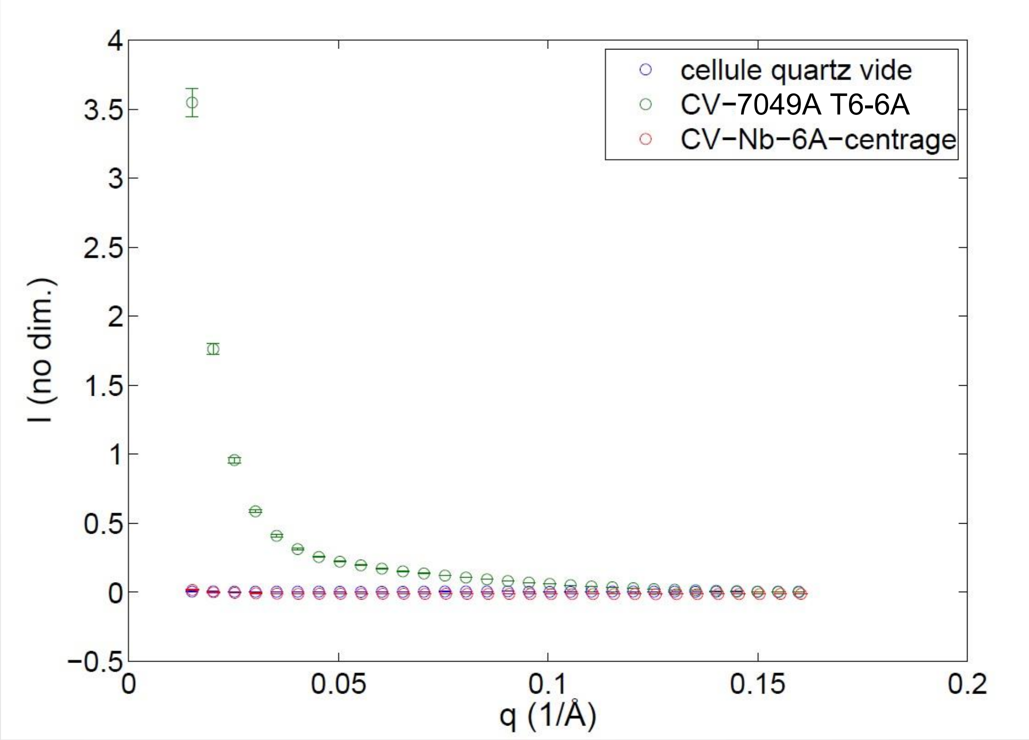
Windows: pure niobium 2 x 6.1mm, sample size Ø 6mm e=4mm

The unfolding process starts between 2000 and 2500bar.

A first measurement was conducted at 4.7Å.

The sample signal was drowned out by the diffraction signal.

More information on page 6.



Used HP Cell

Cell design:

Symmetric metallic windows with metallic seals

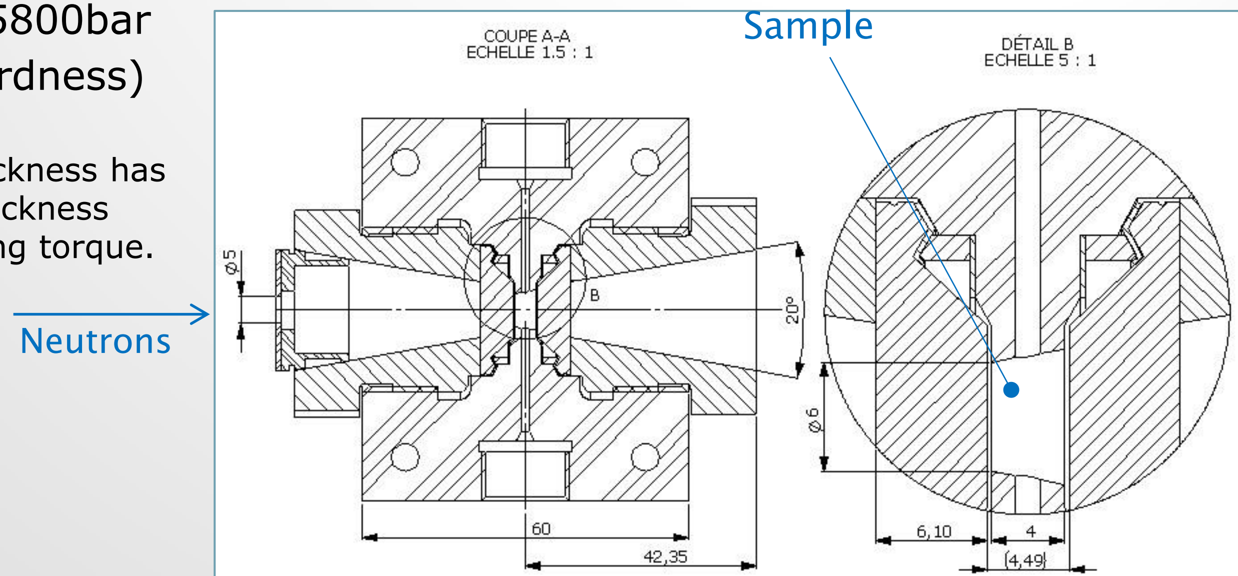
Maximum pressure is window material dependent:

- Pure niobium: 2700bar
- Aluminium 2017A T4: 4500bar
- Aluminium 7049A T6: 6300bar

Achievable pressure with following seal material:

- Lead: 6300bar
- Tin-platted copper: 5800bar
  (due to its higher hardness)

For data treatment the sample thickness has to be measured due to the seal thickness dependence with the applied closing torque.



Cell design
Pros and Cons

Pros:

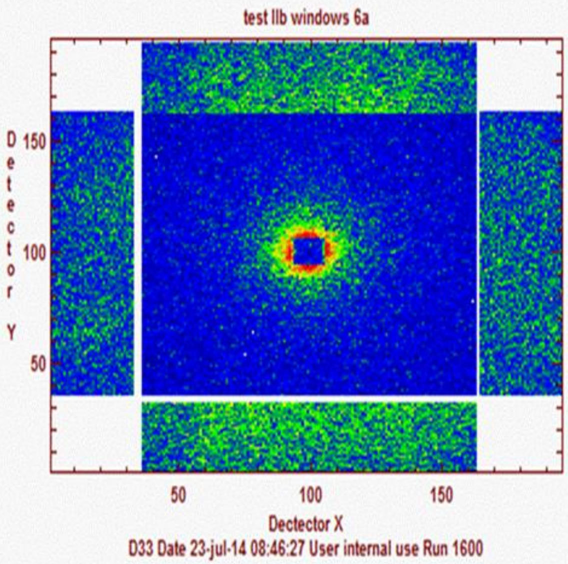
- The cell design is reliable
- Windows are not expensive
- Windows are from metallic materials therefore not fragile
- High pressure is easy to achieve

Cons:

- The cutting edge shape of the anti-extrusion ring is limiting the maximum pressure
- The metallic seals are sticky, especially lead, therefore difficult to take off
- Metallic windows are limiting the usable neutron wavelength from 6Å to 10Å

Problems with metallic alloys for pressure cell windows

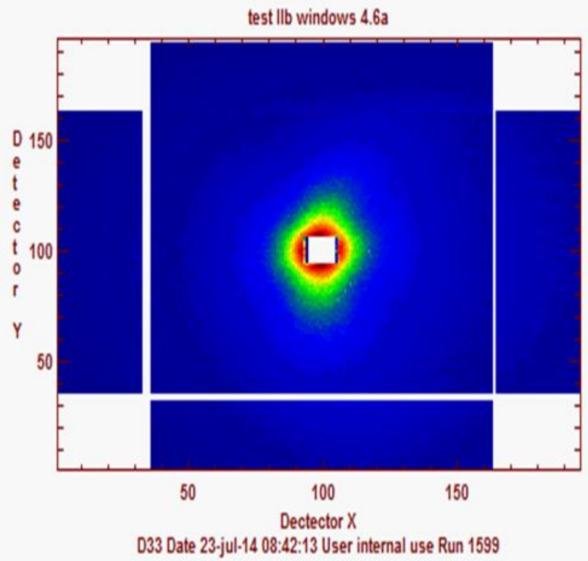
At 6Å



Counting rate =737 c/s

Transmission 0.75

At 4.6Å



Counting rate=34000 c/s

Transmission = 0.572

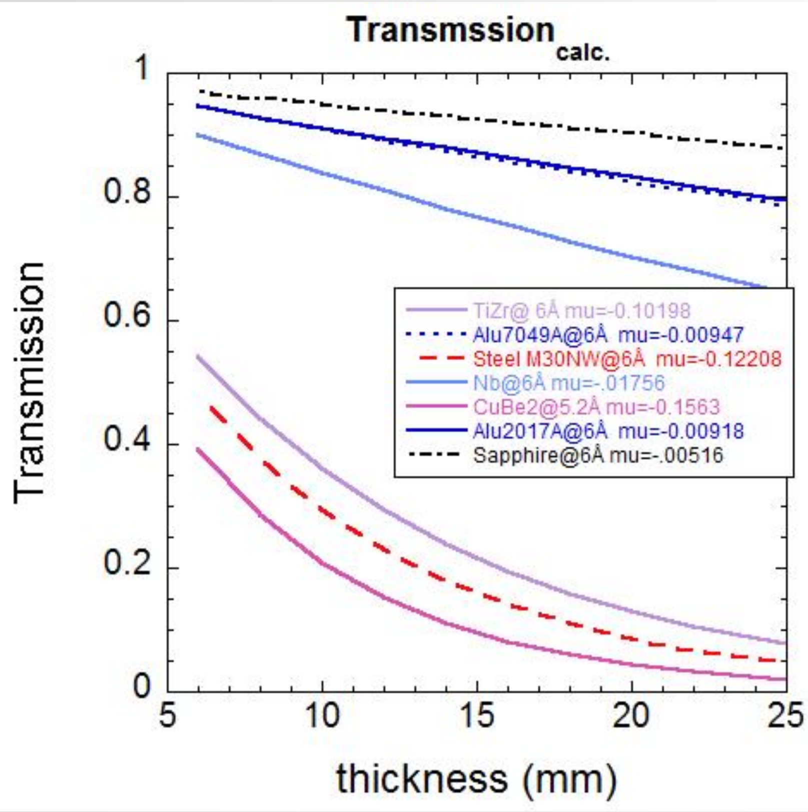
Below ~ 5.5Å huge Bragg diffraction signal from disordered polycrystalline domains !

Above 10Å

Multiple scattering from nanometer scale grain boundaries of polycrystalline materials.

Conclusion : use of alloy windows limits the wavelength range from 6 to 10Å

Neutron Transmission vs Material thickness

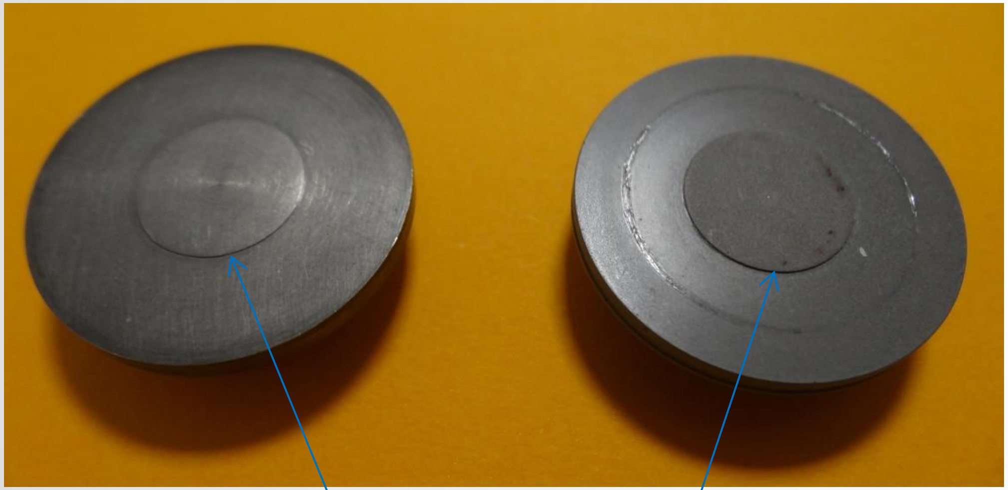


Alloy	Shear strength Factor y	Ultimate tensile strength [MPa]	Calculated shear strength [MPa]	Pressure p at shear strength [bar]	Tensile yield strength [MPa]	Elongation at rupture [%]
TiZr Sample I	0.6	840.0	504.0	12018	690.0	9.0
TiZr Sample II	0.6	706.0	423.6	10101	555.0	14.0
Niobium pur CABOR Type 1 reactor grade annealed, 90% minimum recrystallized	0.7	125.0	87.5	2086	73.0	25.0
Niobium pur REMBAR annealed 60HV	0.7	195.0	136.5	3255	105.0	30.0+
Niobium pur REMBAR Cold worked 150HV	0.6	585.0	351.0	8370	-	5.0
Aluminium 7049A T6 AW-AlZn8MgCu	0.6	650.0	390.0	9300	580.0	10.0
Steel M30NW AUBERT & DUVAL X4CrNiMoN21-9-4 Hyper tempered and cold worked	0.6	935.0	561.0	13378	608.0	42.0
CuBe2 TF/TH2 C17200	0.6	1303.0	781.8	18643	1215.0	8.6
Aluminium 2017A T4 AW-AlCu4MgSi	0.6	420.0	252.0	6009	280.0	18.0
Sapphire		190 - 400				

Shear section S_T: π*d*t; with d=10.23mm and t=6.1mm
Shear force F_T: S_T*y*UTS

Pressure section S_p: π*d²/4; with d=10.23mm
Pressure p: F_T/S_p

Material limits for the windows



On the left:
Pure niobium after 2000bar
On the right:
Anodized aluminium 7049A T6 after 5800bar

Visible shearing

Shear strength of ductil metallic materials:

Low-strength, tough steel: ~0,8 x UTS (Ultimate tensile strength)
High-strength steel: ~0.6x UTS
Aluminium 7075 T6: ~0.6x UTS

Outlook

The cell design is working well

The limiting factor is the seal design.
We will integrate modifications.
Metallic windows are limiting the wavelength from 6Å to 10Å.
If no scattering and transparency is asked use sapphire, but it is fragile, our partners from the ILL will tell you more…