Pressure cell for SANS: an update

→ Removable pressure cell windows in metallic alloys

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Aim of the study

✓ Pressure cell for SANS measurements

- pressure range: 2,500 – up to 6,000 bar
- biophysics: low signal < low concentration (*e.g.* few g/L for a protein)

✓ Our strategy: use materials that are stronger than single crystals of sapphire

→ Thick **windows** in **metallic alloys**, which display:

- good mechanical properties
- reasonable transmission
- reasonable q-scattering

and which are:

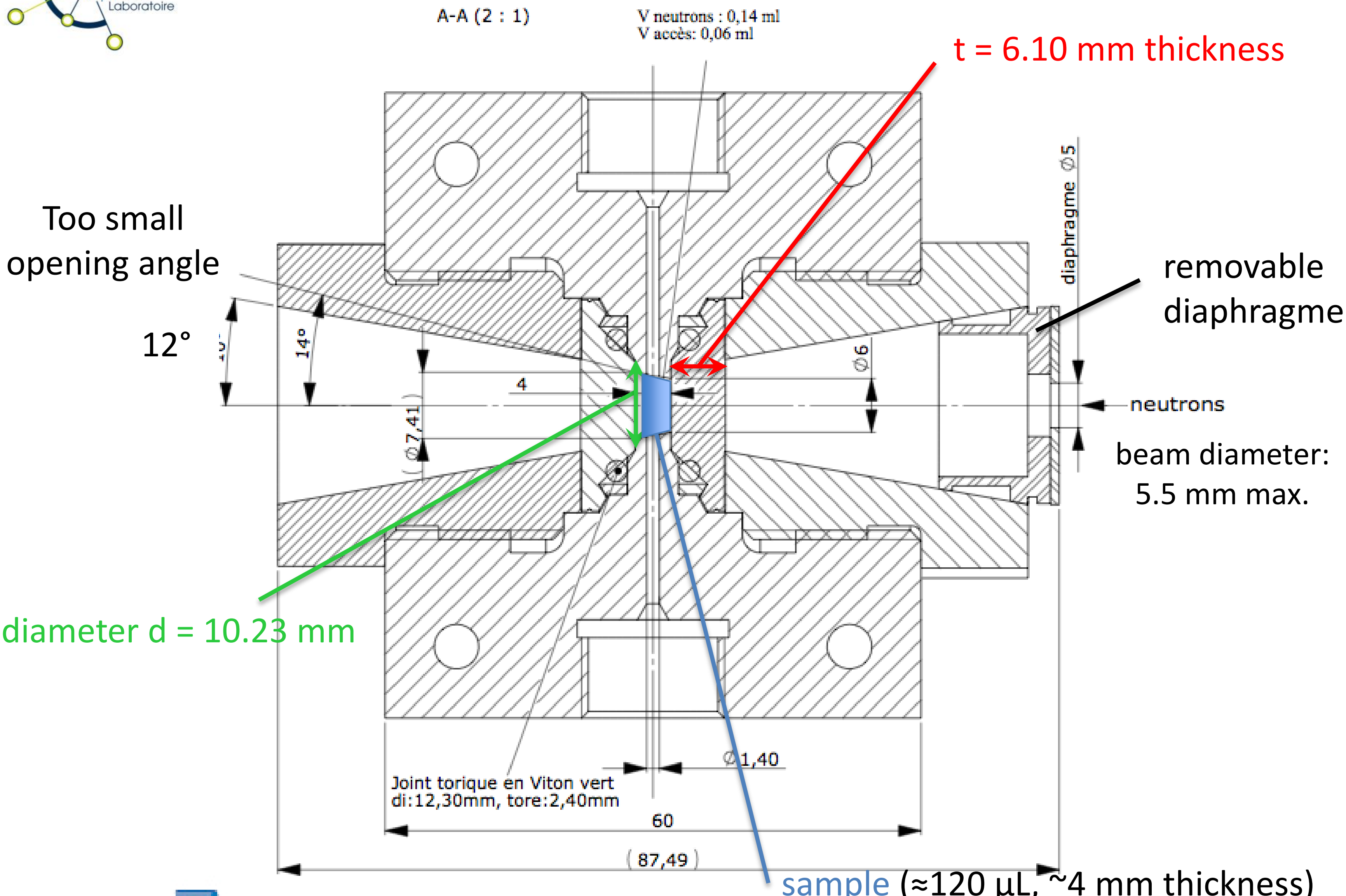
- non magnetic for a possible application in NSE
- not much activation under neutron beam

A SANS pressure cell with removable windows

SANS: small beam, small angle → flat cell with windows:

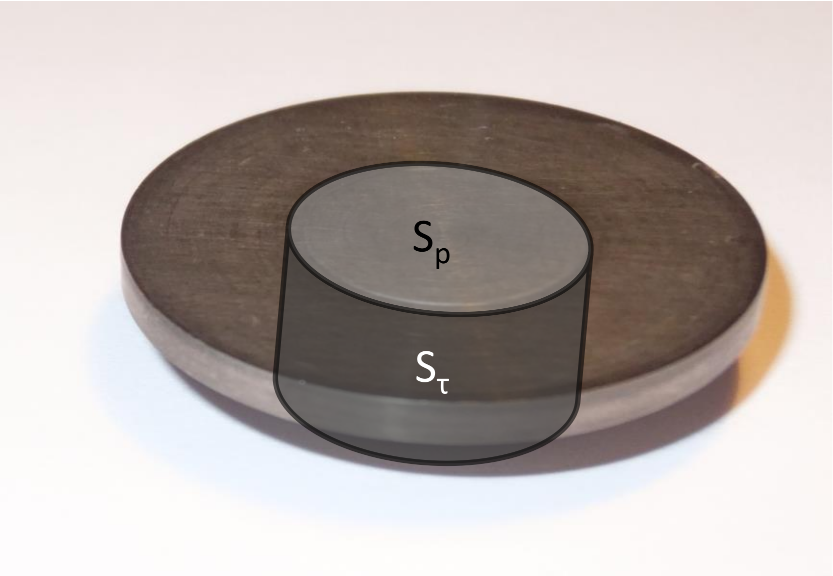
- difficulties: machining, sealing
- advantages: thick body in resistant material (stainless steel)
- **removable windows** in suitable material

Windows parameters



Pressure at shear strength

Nb window (back) after a pressure experiment



Pressure section: $S_p = \pi * d^2/4$ with d = 10.23 mm
→ $\mathbf{S_p = 82.2\ mm^2}$

Shear section: $S_\tau = \pi * d * t$ with d = 10.23 mm and t = 6.10 mm → $\mathbf{S_\tau = 196.0\ mm^2}$

Shear force: $F_\tau = S_\tau * y * UTS$
$= 196.0 * y * UTS$

Pressure at shear strength

Alloy	Shear strength factor (y)	Ultimate tensile strength (UTS) [MPa]	Calculated shear strength [MPa]	Pressure @ shear strength [bar]	Elongation at rupture [%]
TiZr	0.6	840	504	12 018	9

Calculated shear strength = y*UTS

Pressure at shear strength

Alloy	Shear strength factor (y)	Ultimate tensile strength (UTS) [MPa]	Calculated shear strength [MPa]	Pressure @ shear strength [bar]	Elongation at rupture [%]
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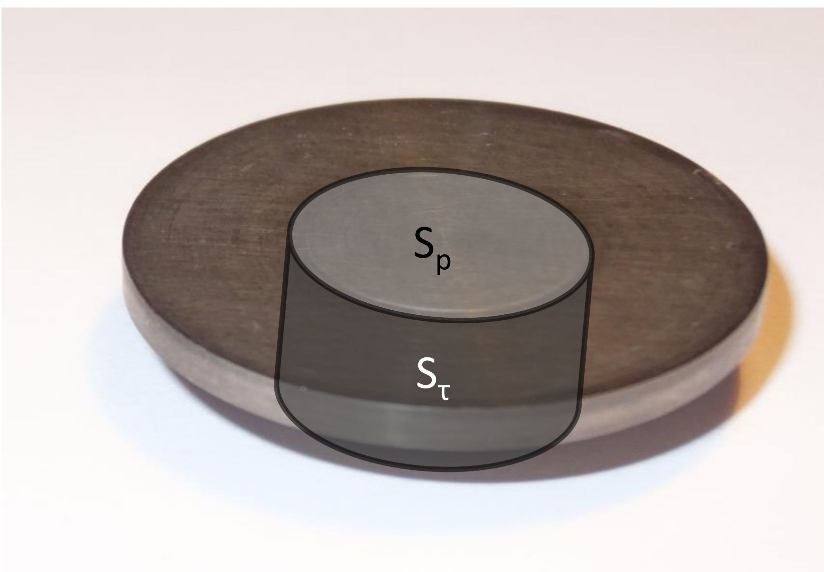


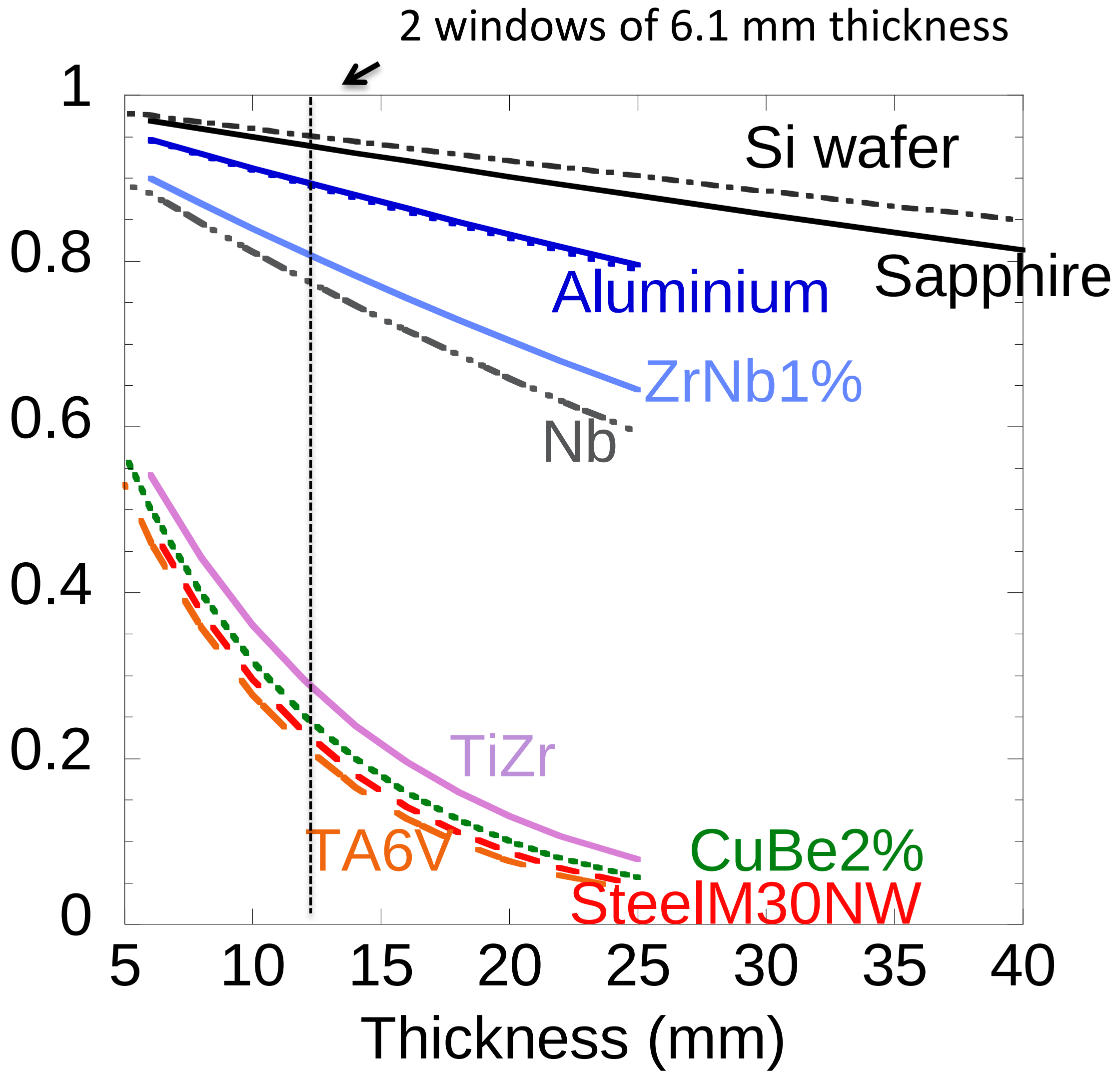
Pressure $P = F_\tau / S_p = 196 * y * UTS / 82.2$
$= 2.4 * y * UTS$

Pressure at shear strength

Alloy	Shear strength factor (y)	Ultimate tensile strength (UTS) [MPa]	Calculated shear strength [MPa]	Pressure @ shear strength [bar]	Elongation at rupture [%]
TiZr	0.6	840	504	12 018	9
Ti-Al6-V4	0.6	1100	660	15 742	10
Ti-Al6-V4 ELI	0.6	860	516	12 307	10
Ti-Al6-Nb7	0.6	900	540	12 880	10
Pure Niobium	0.7	195	137	3 255	30
Aluminium 7049A	0.6	650	390	9 300	10
Aluminium 2017A	0.6	420	252	6 009	18
Steel M30NW	0.6	935	561	13 378	42
CuBe2	0.6	1303	782	18 643	9
Sapphire	0.6	190 - 400			

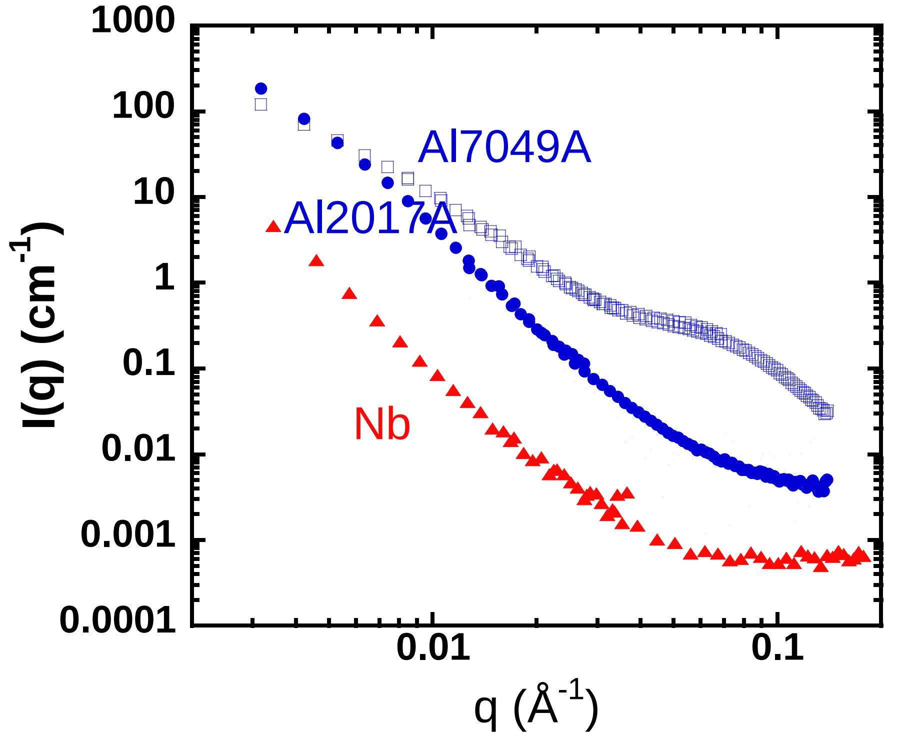
P_{max}: pressure/safety factor (= 1.5, 2, …)

Transmission@6Å



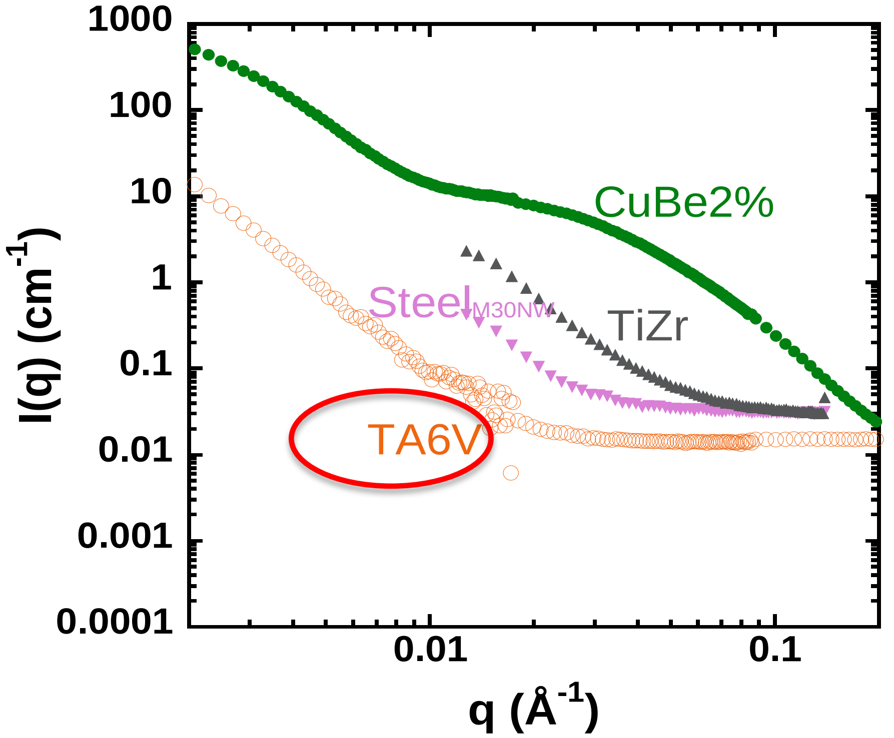
Scattering of high transmission alloys

Normalized to the sample thickness and transmission



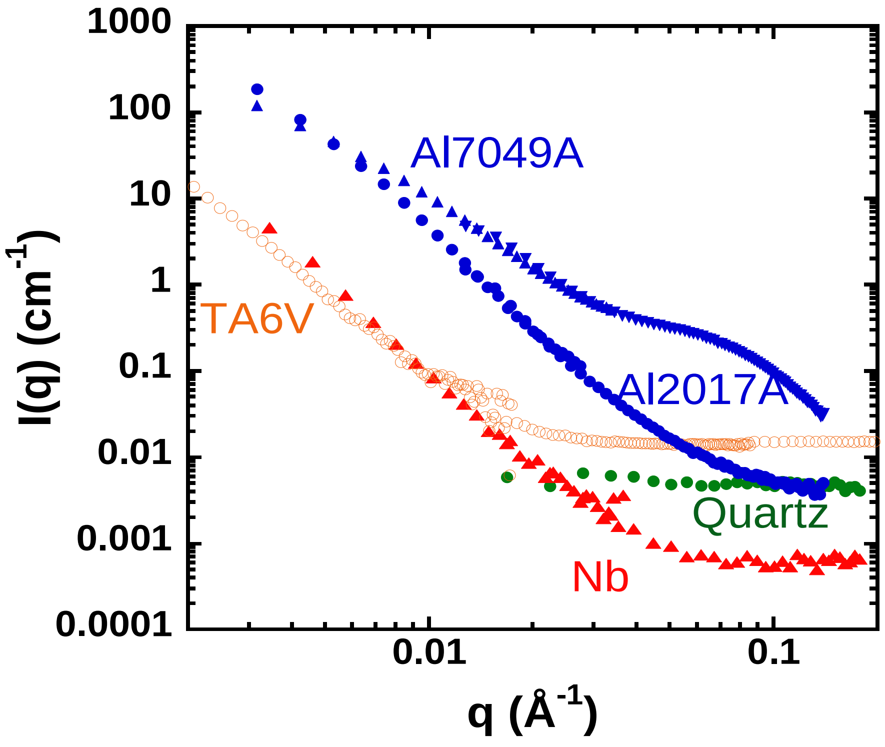
Scattering of low transmission alloys

Normalized to the sample thickness and transmission

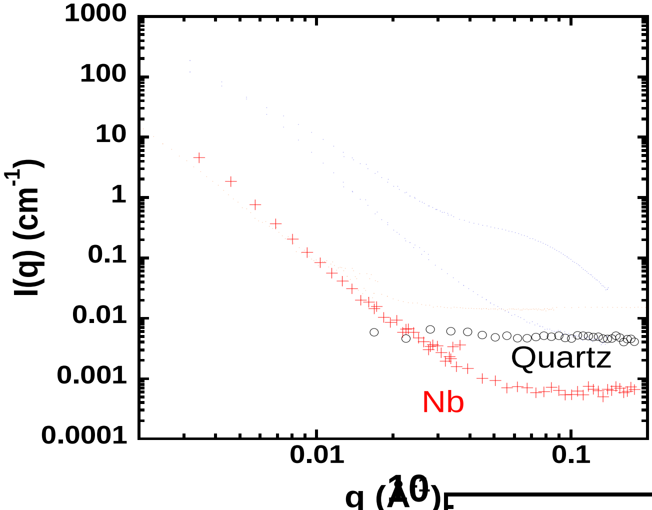


Scattering of the tested materials / Quartz

Normalized to the sample thickness and transmission

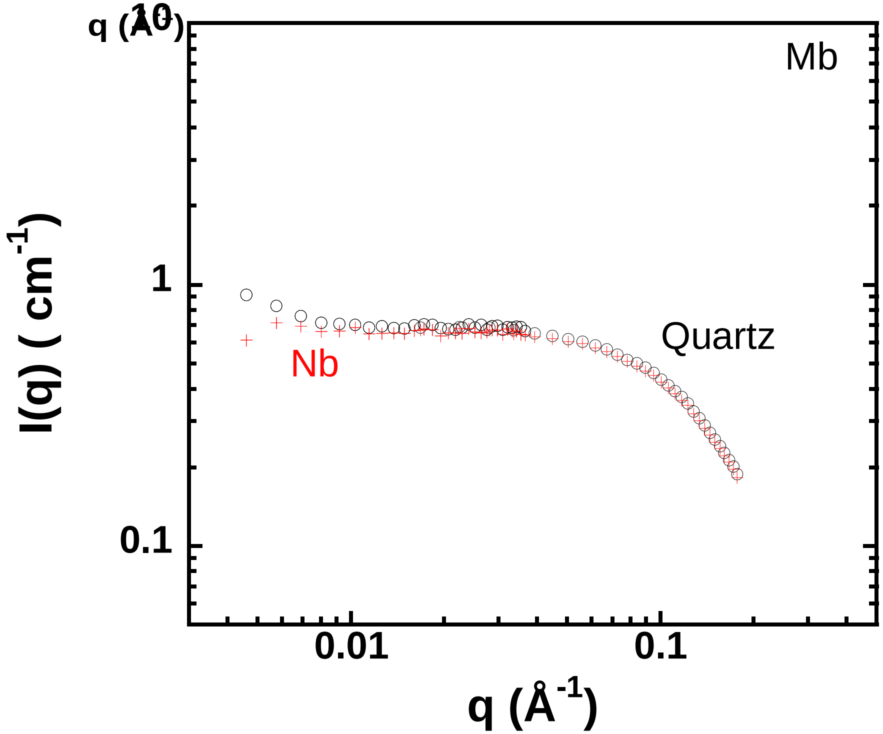


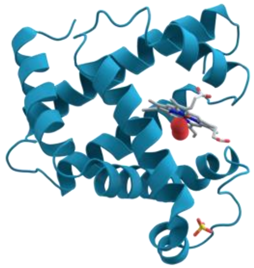
Nb vs. Quartz



Transmission 12.2mm@6Å=.712

P_{max}~3000bars

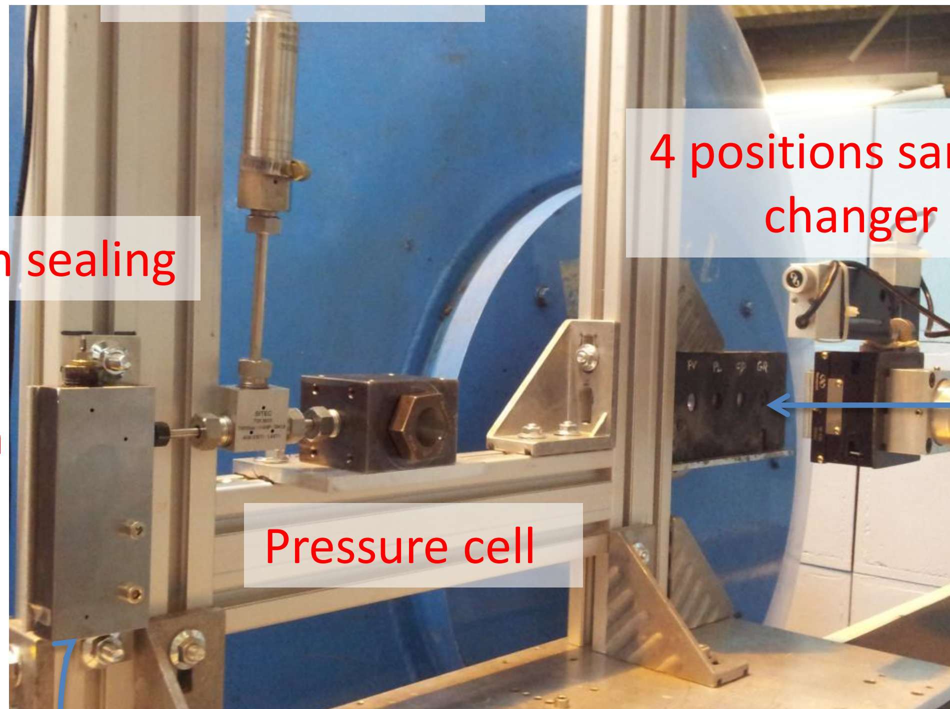




Myoglobin protein
(20 g/L)

Pressure Experiments Device

To fill and empty the cell: quite easy, not much problem of air bubbles (so, no loss in pressure due to change of volume)

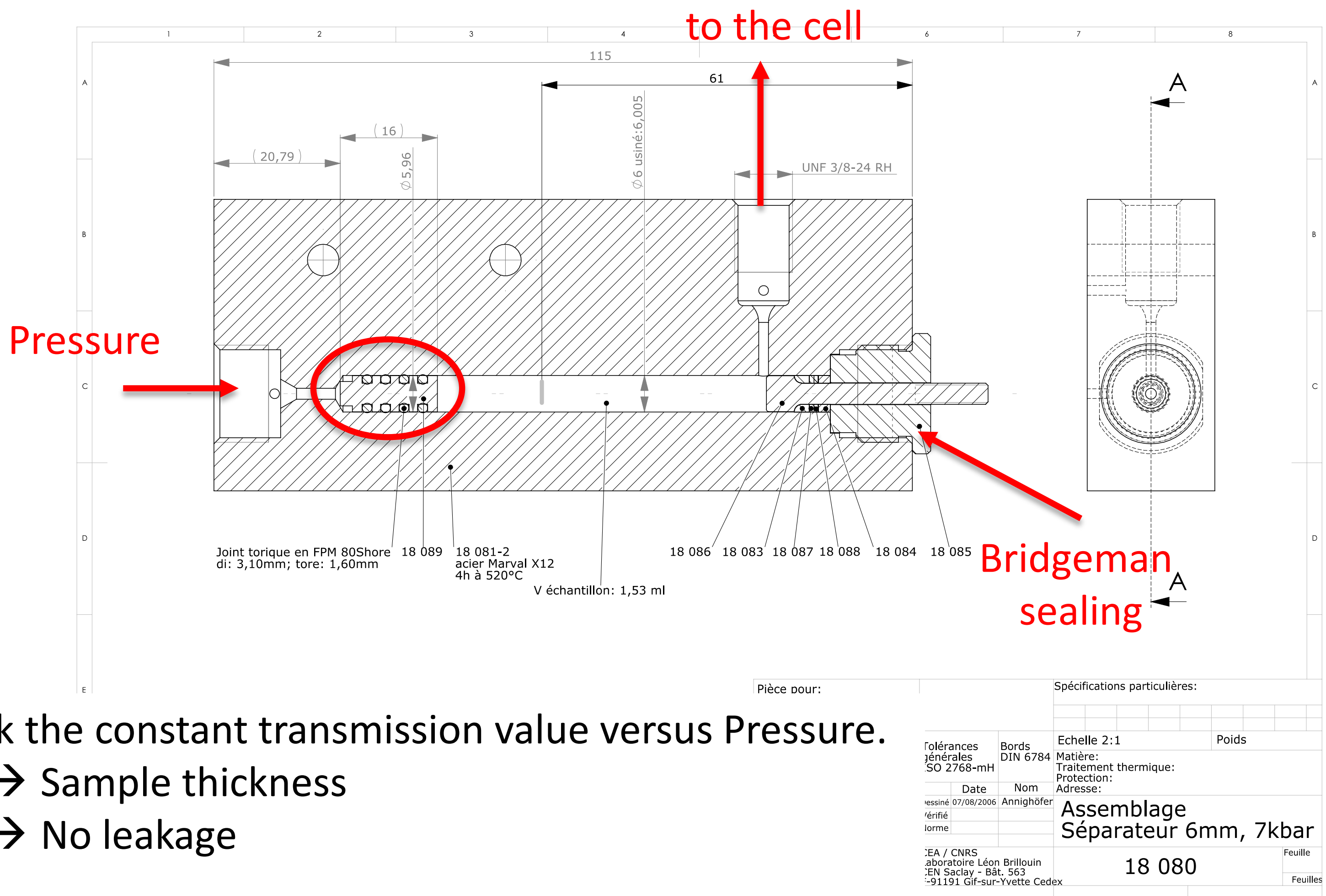


Volume~$3cm^3$

Separating piston

Separation piston: good sealing between the buffer (D_2O) in the sample room.

→ no problem of buffer leakage or mixing with the sample

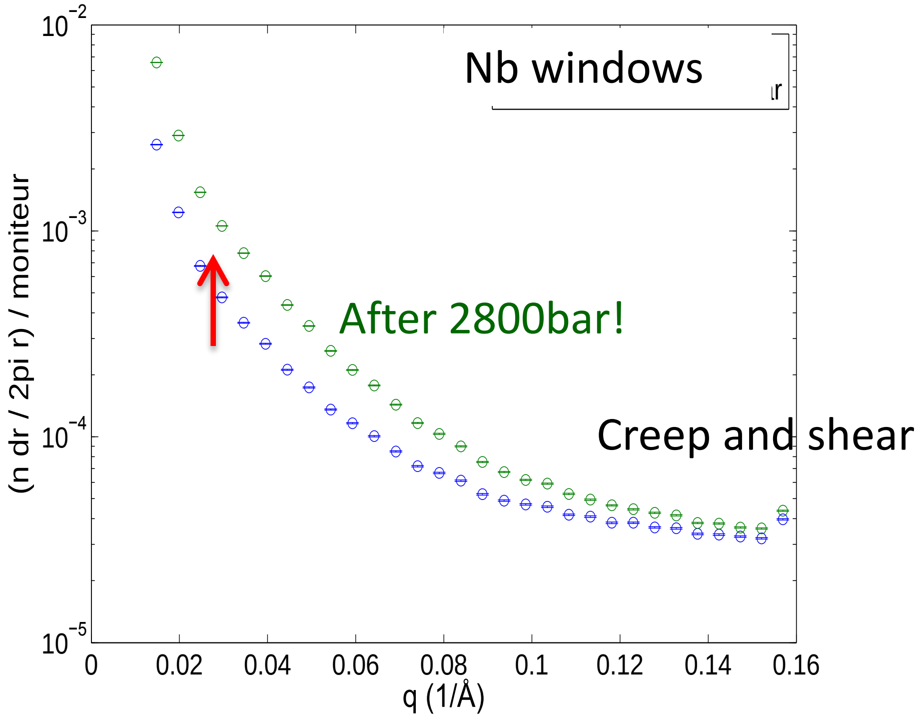


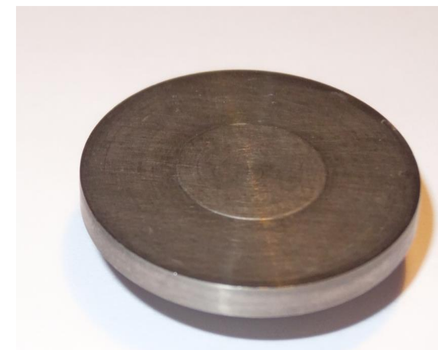
Check the constant transmission value versus Pressure.

→ Sample thickness

→ No leakage

Effect of pressure on windows



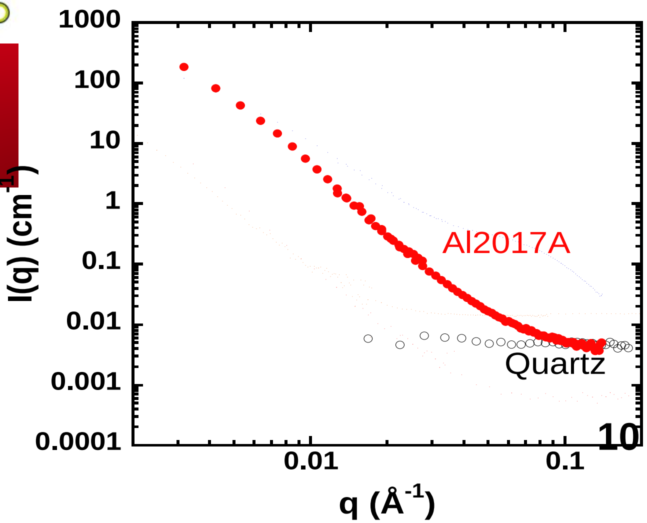


Nb windows after 3kbar

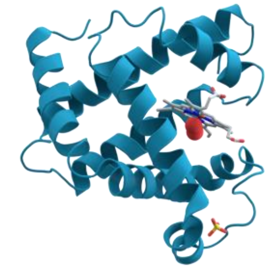
→ Solution:

Plastification of Nb windows at P_{max} before the pressure experiments

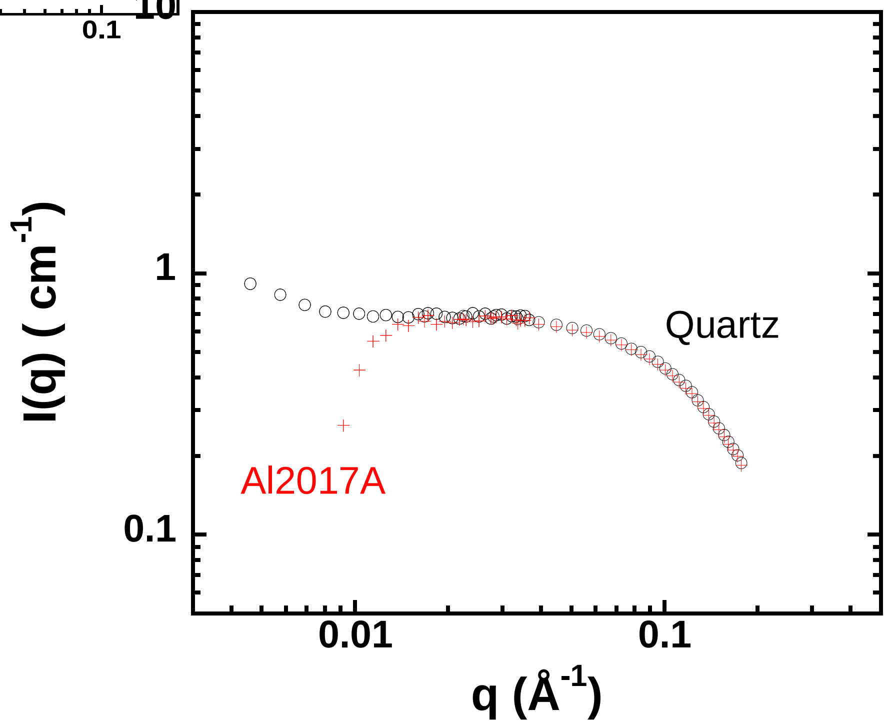
Al2017A vs. quartz



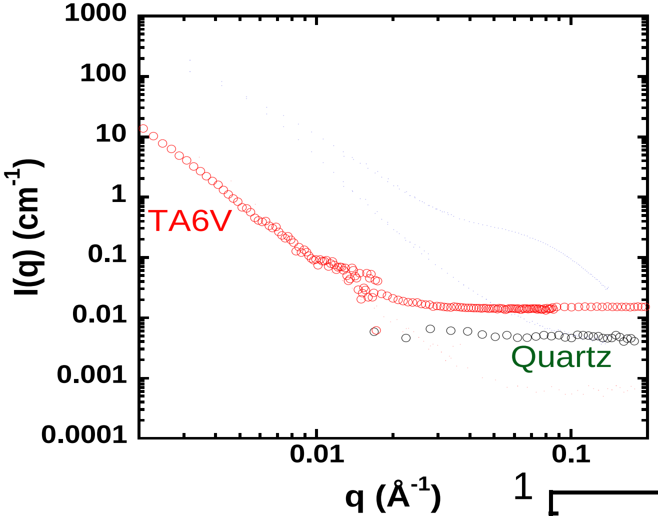
Transmission 12.2mm @6Å=.876
P_{max}~4000bars



Myoglobin protein
(20 g/L)



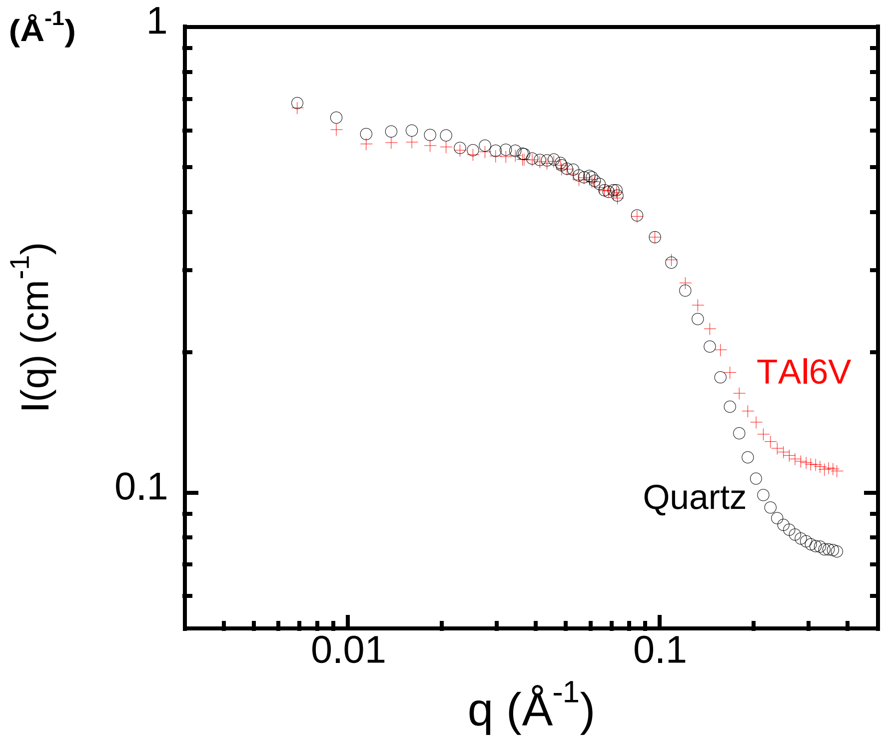
TiAl6V4 vs. quartz



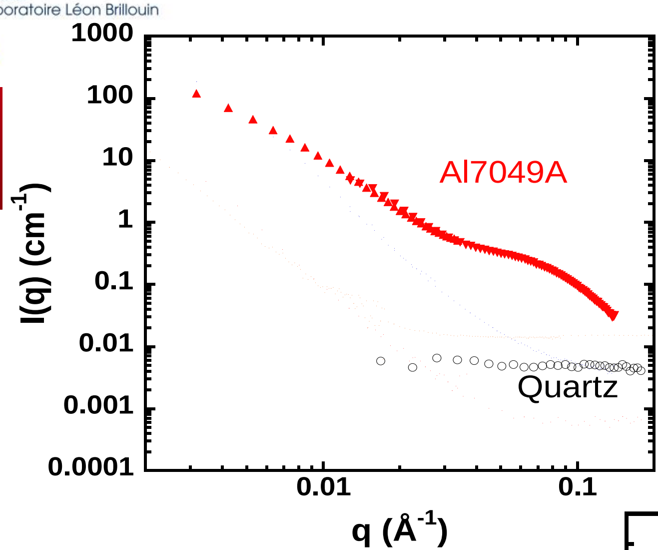
Transmission 9.53mm @6Å=0.395

P_{max}~7-8000bars

Apomyoglobin
(14 g/L)

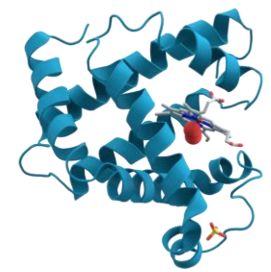


Al7049/ Quartz

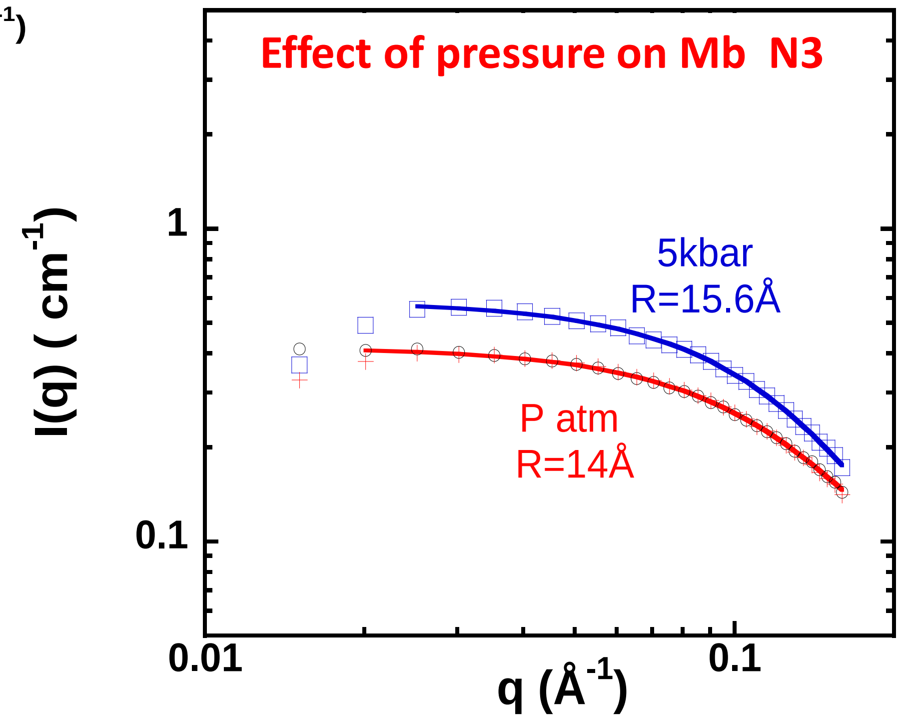


Transmission 12.2mm @6Å=0.898

P_{max}~6000bars



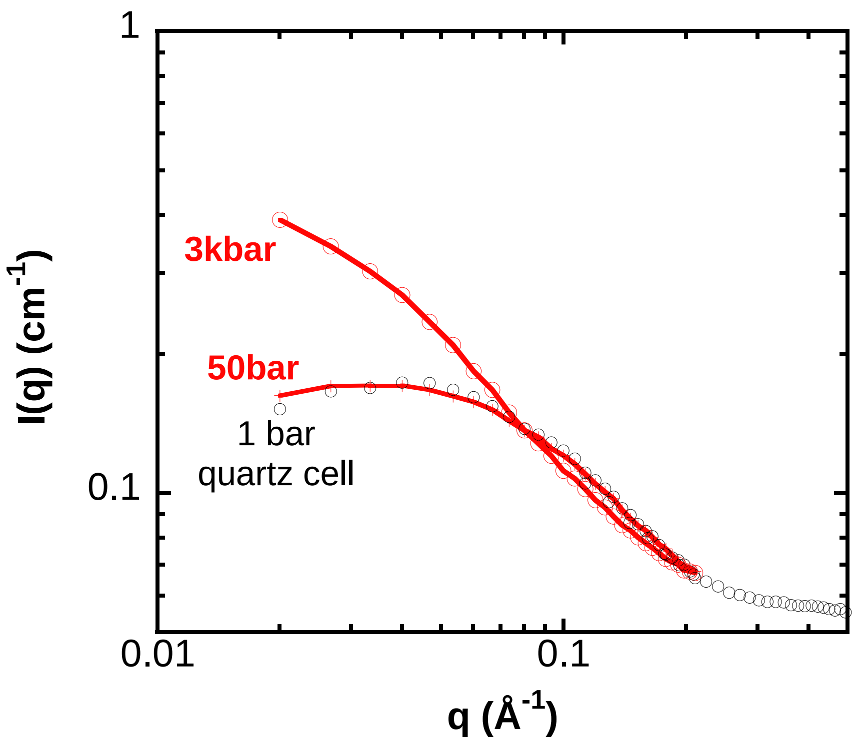
Myoglobin N3
(20 g/L)



TiAl6V4 Pressure experiments

Thin (2*2.97mm) TiAl6V4 windows
Plastification at 3.6kbar before P experiments
Transmission @6Å=0.489
P_{max}~3kbars

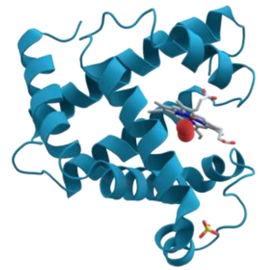
Effect of pressure on Apomyoglobin @ pH=6



Apomyoglobin
pH=6
(5 g/L)

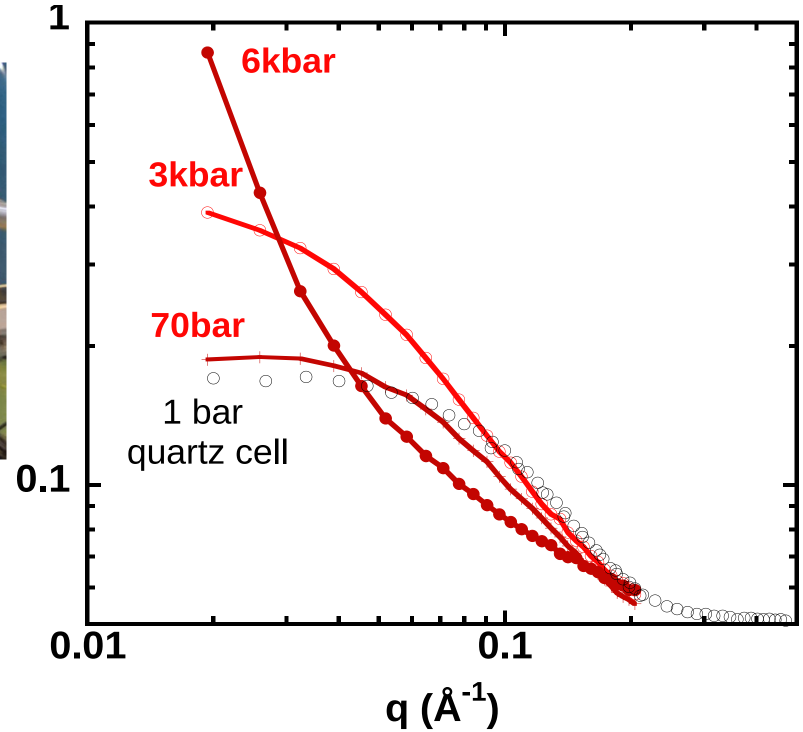
TiAl6V4 Pressure experiments

Thick 2*4.76mm TiAl6V4 windows
Plastification before P experiments at 6.05kbar
Transmission @6Å=0.316
P_{max}~6kbars



Myoglobin
(7g/L)

Effect of pressure on Mb @ pH=6 , 7mg/ml



Which metallic alloy?

- ✓ The strongest materials (TiZr, CuBe2%, SteelM30MN): low transmission and high scattering → not good candidates for pressure cell windows except if the sample is a very good scatterer! Care with multiple scattering for CuBe2%.

- ✓ **Niobium**: good transmission and does not scatter much → a good candidate! It allows SANS measurements even on weak scatterers BUT at a pressure up to about **2.8 kbar**.
- ✓ Plastification at $P_{max\ experiments}$ before the experiments mandatory.

- ✓ **Aluminium** (Al7049A): very good transmission but quite high scattering → for a sample which scatters; can reach about **6 kbar**. Successful at 5kbar with low scatterer (I(q) ~ $0.5cm^{-1}$)

- ✓ **TiAl6V4**: low transmission, does not scatter much, no corrosion

  tested at 3kbar with 2*2.97mm windows and plastification P_{max}=3.6kbar before P experiments (no visible shear deformation)

  tested at **6kbar** with 2*4.76mm windows and plastification at 6.05kbar before P experiments.

  Works rather well with very low scatterers (I(q) ~ $0.2cm^{-1}$!!!) Biological molecules

P max. achieved with dilute solutions of biological molecules

Alloy	Shear strength factor (y)	Ultimate tensile strength (UTS) [MPa]	Calculated shear strength [MPa]	Pressure@ shear strength [bar]	P_{max} tested	Elongation at rupture [%]
Ti-Al6-V4	0.6	1100	660	15 742	**6000**	10
Pure Niobium	0.7	195	137	3 255	**2800**	30
Aluminium 7049A	0.6	650	390	9 300	**5000**	10

P_{max}: pressure/safety factor (= 1.5, 2, …)

General remarks

- ✓ The **sample scattering** has to be **above ~10-20% the one of windows**
  - for samples with low scattering -> sapphire should be better

- ✓ Usable **neutron wavelength** range: **6-10Å**
  - not < 5Å: huge Bragg diffraction (due to disordered polycrystalline domains)
  - not > 10Å: multiple scattering (due to nanometer scale grain boundaries of polycrystalline materials)

- ✓ **Plastification**: mandatory for Nb (maybe for TiAl6V4) before the pressure experiments.

- ✓ **Change of the windows** during the experiment: possible only with tin-platted copper, not with lead. But not easy!
  Have two identical P devices (one « running », one under preparation cleaning…)

- ✓ Transmissions have to be measured as well as sample thickness at each pressure (double check).