Pressure cell for SANS: an update

→ Removable pressure cell windows in metallic alloys

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

Aim of the study

✓ Pressure cell:
- for SANS measurements
- pressure range: 2,500 – up to 6,000 bar
- small volumes (≈100 µL)
- biophysics: low signal < low concentration (*e.g.* few g/L for a protein)

✓ Our strategy: to 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

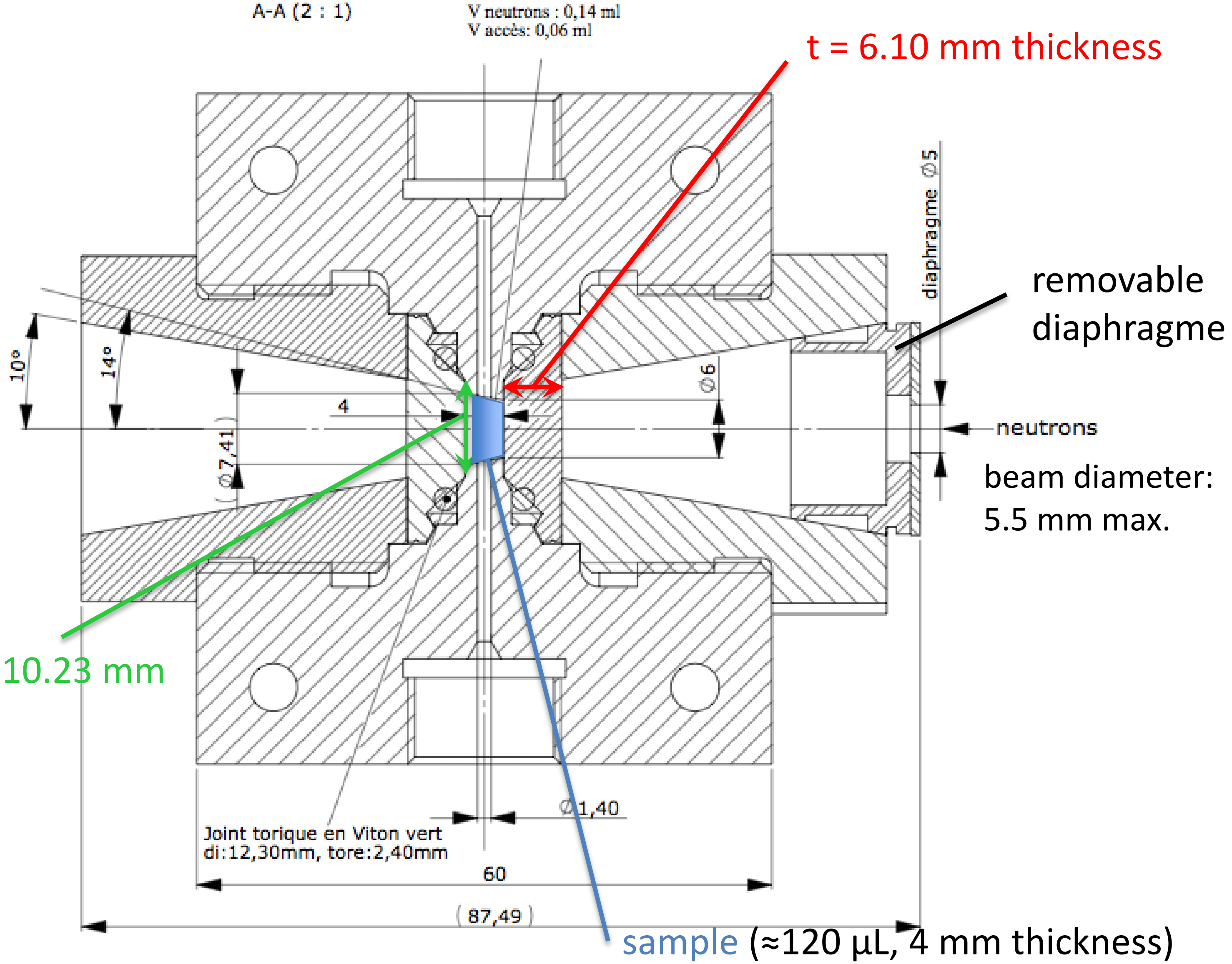
and which are:
- non magnetic for a possible application in NSE
- not much activable

A SANS pressure cell with removable windows

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

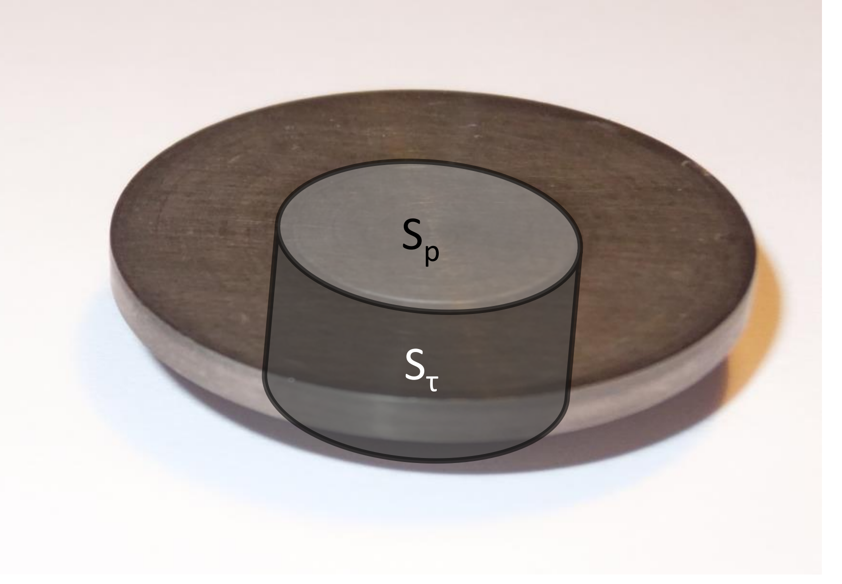
- difficulties: machining, sealing
- advantages: thick body in resistant material (stainless steel)
- **removable windows** in suitable material

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
→ **S_p = 82.2 mm²**

shear section: $S_\tau = \pi * d * t$ with d = 10.23 mm and t = 6.10 mm → **S_τ = 196.0 mm²**

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

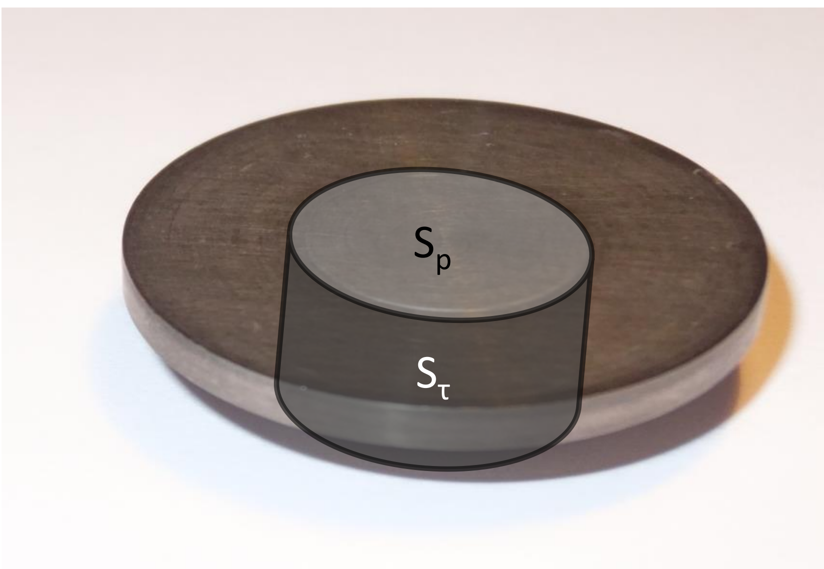
Pressure at shear strength

! 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! [%]!
TiZr!!	0.6!	840!	504!	12,018!	9!



pressure p: $F_\tau/S_p = 196*y*UTS/82.2$

$= 2.4*y*UTS$

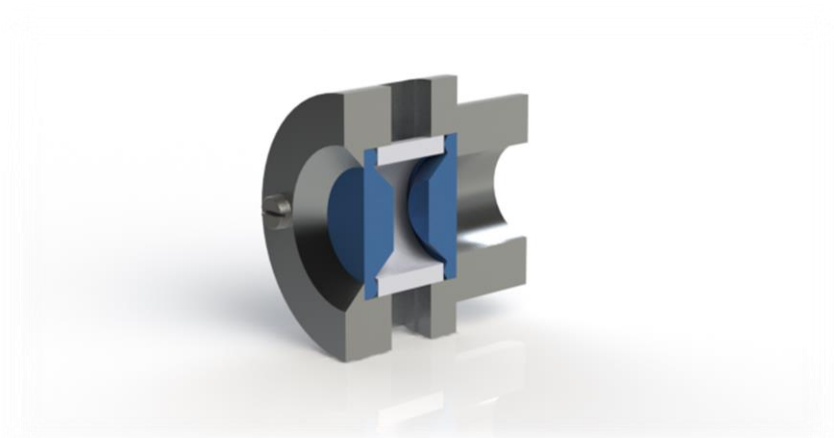
Pressure at shear strength

! 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!
TiDAl6DV4!	0.6!	1100!	660!	15!742!	10!
TiDAl6DV4!ELI!	0.6!	860!	516!	12!307!	10!
TiDAl6DNb7!	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, …)

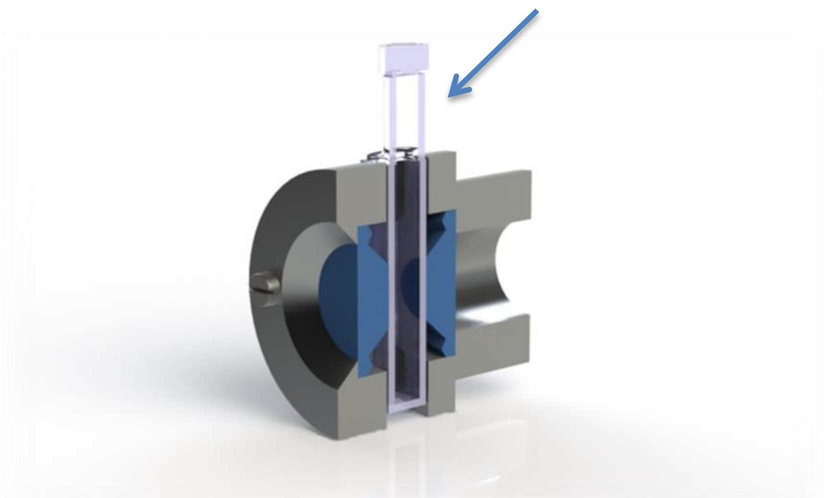
Tests on the windows

Use of a dedicated device for the measurements (without the pressure cell):



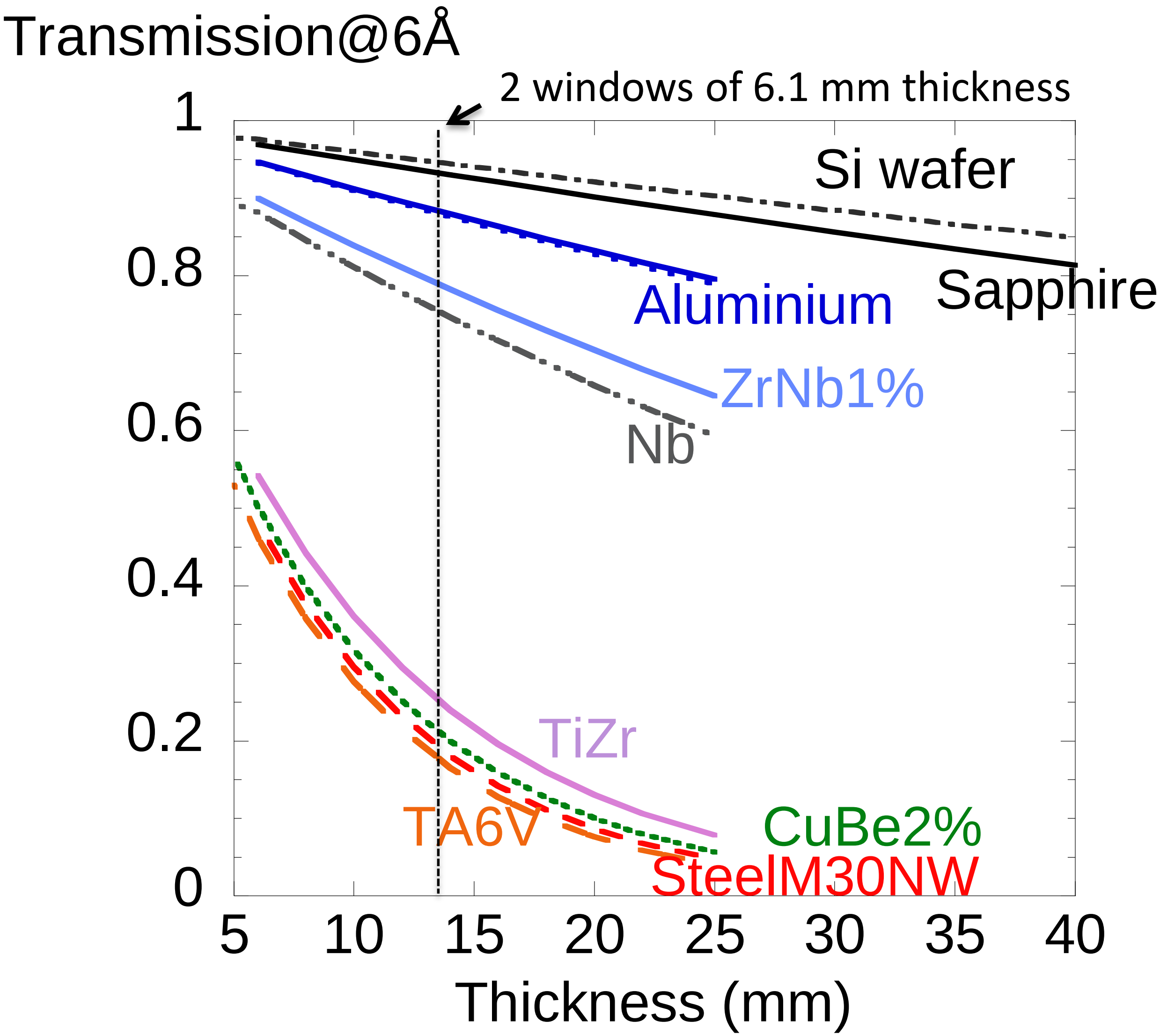
« empty cell » (2 windows)

Quartz cell to test a sample



2 windows + sample in quartz Hellma cell

Transmission

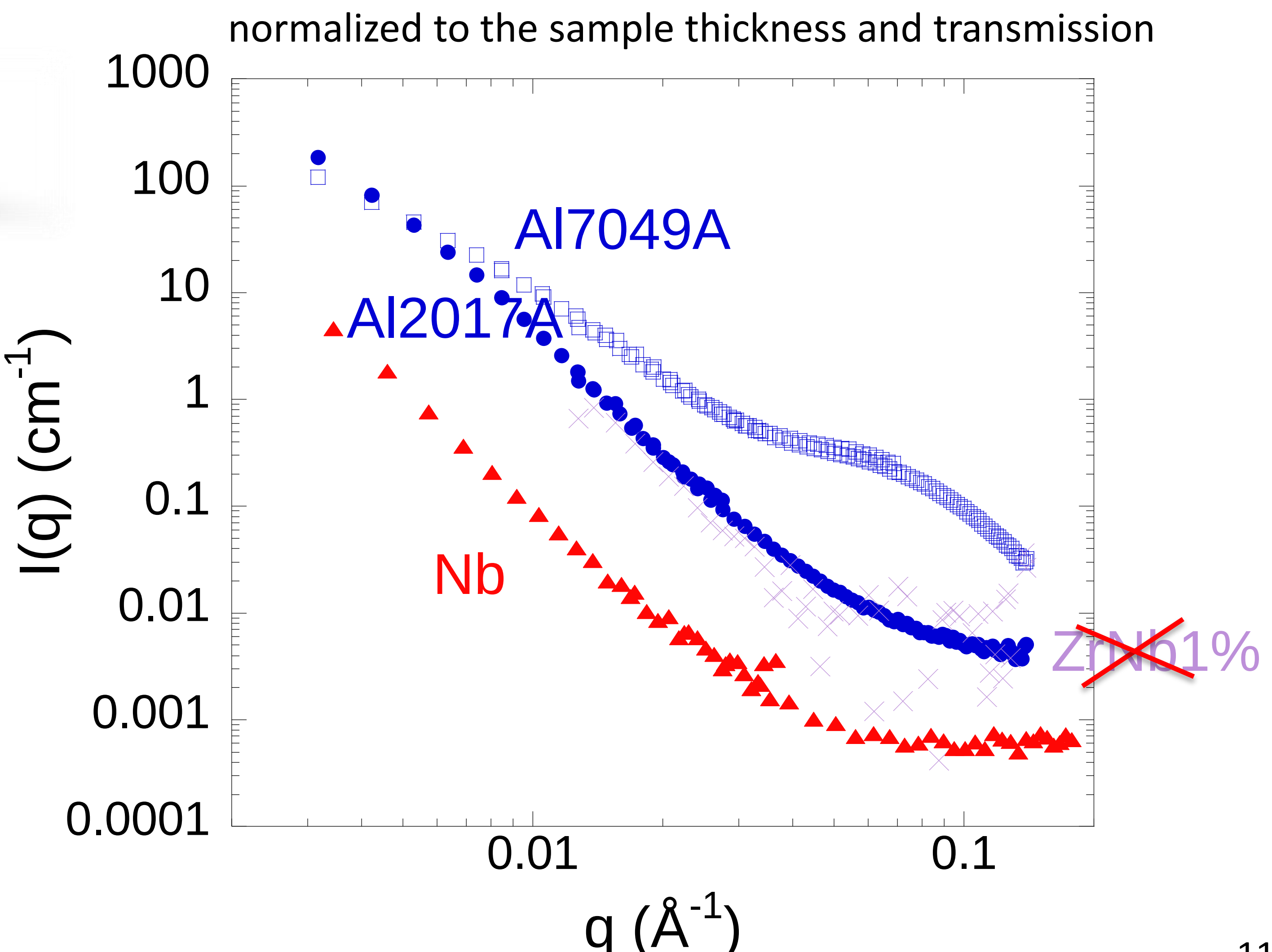


T=exp(-µ.t)

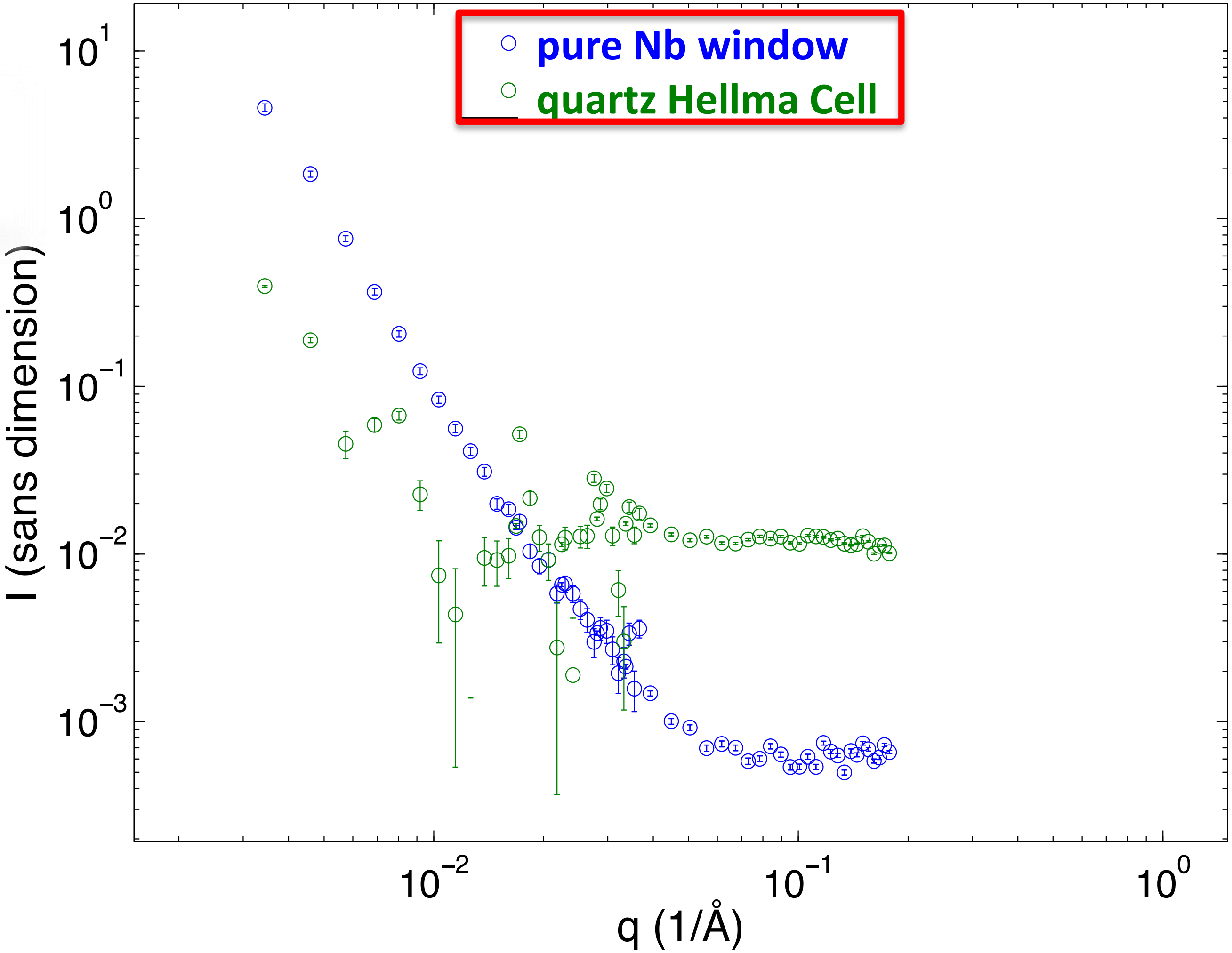
linear absorption coefficient

thickness

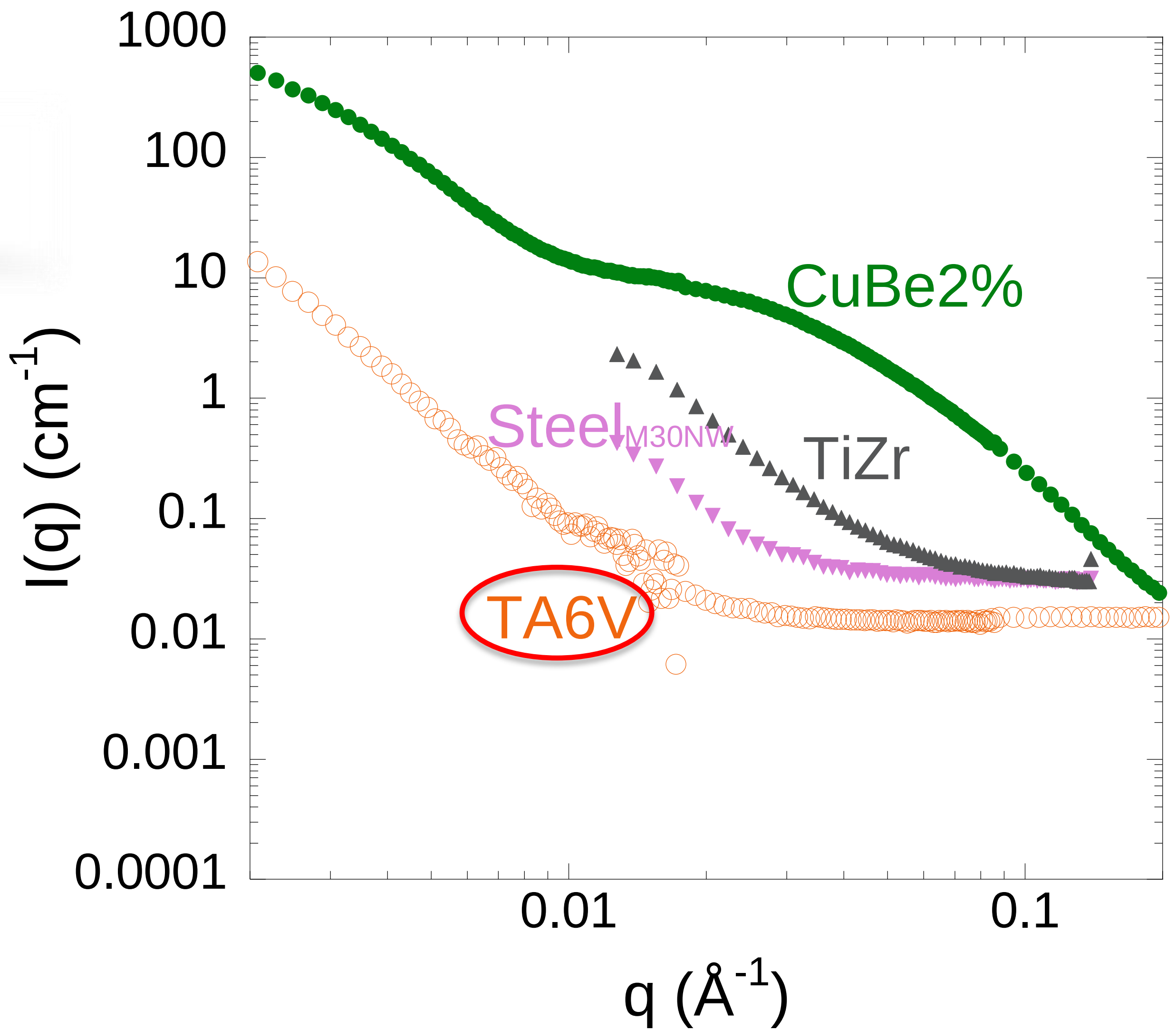
Scattering: good transmission alloys



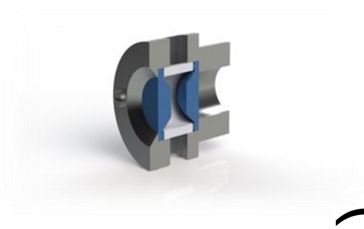
Scattering: Nb vs. quartz

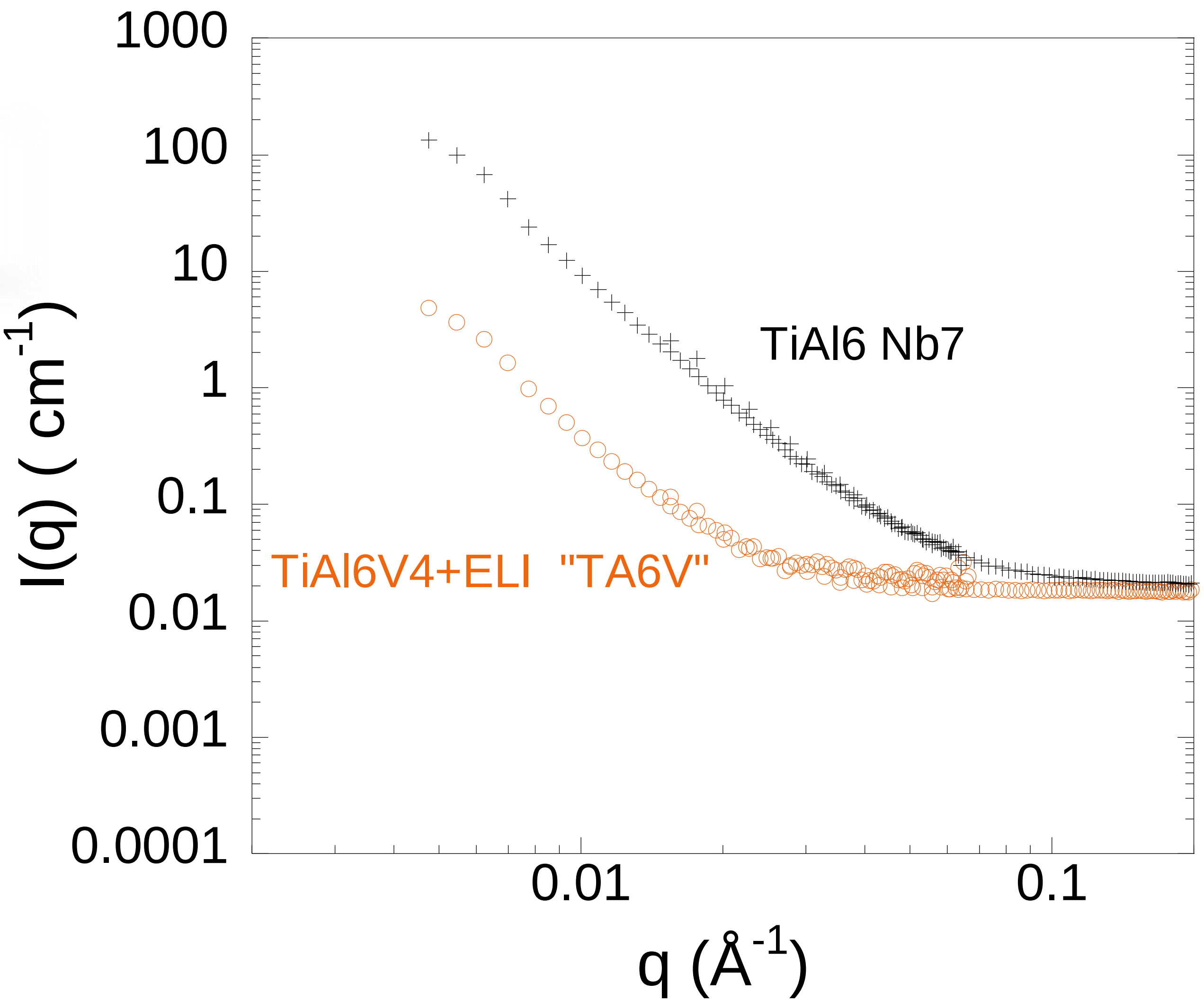


Scattering: bad transmission alloys

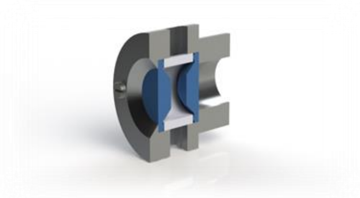


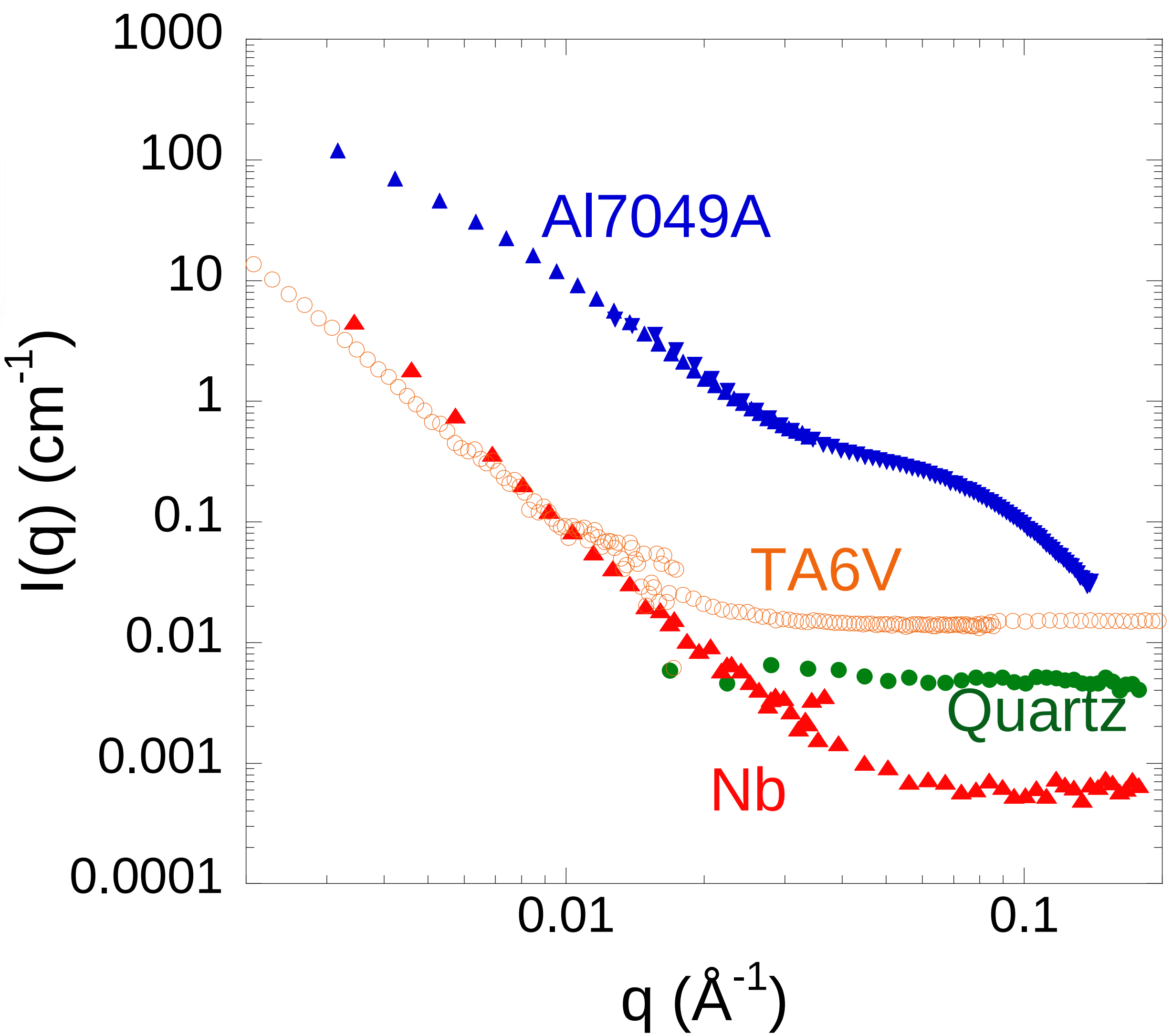
Scattering: TiAl6V4 vs. TiAl6Nb7





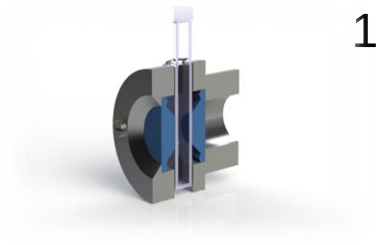
→ the tested alloys

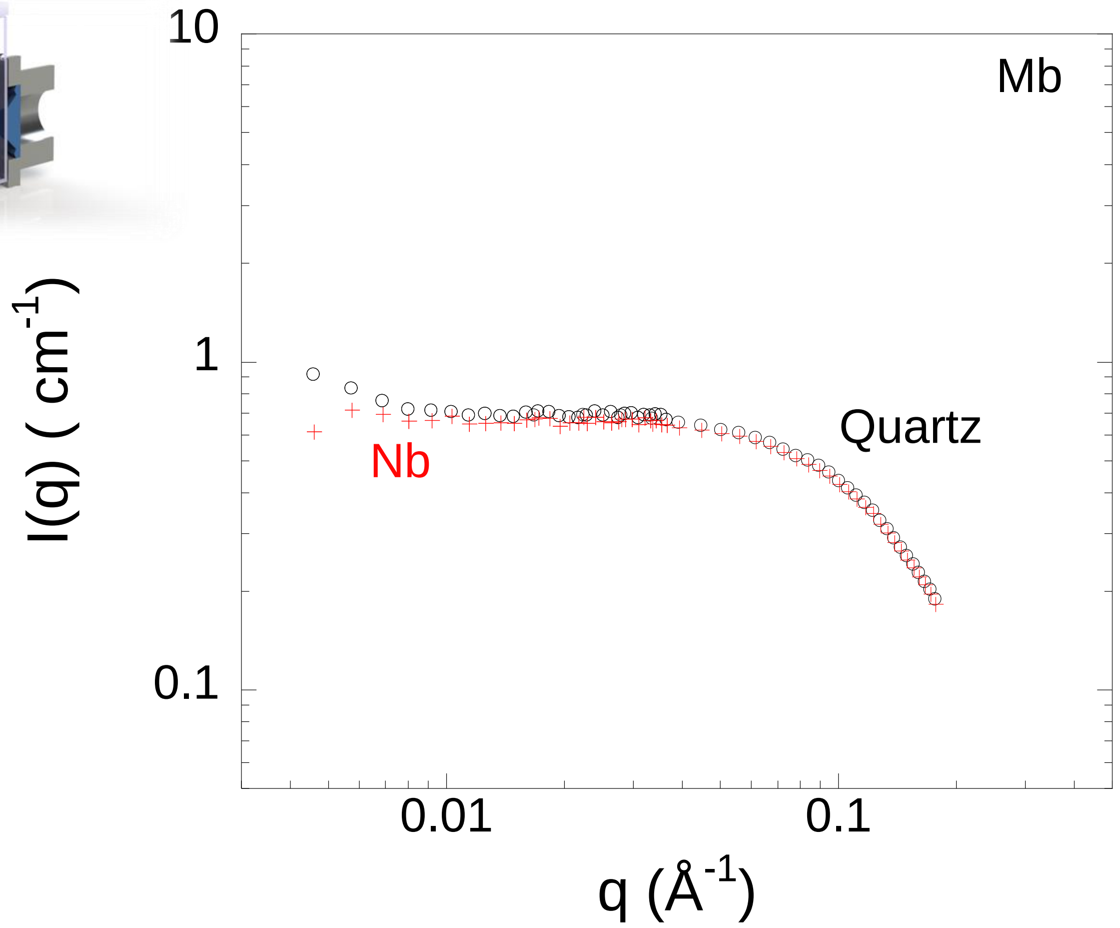


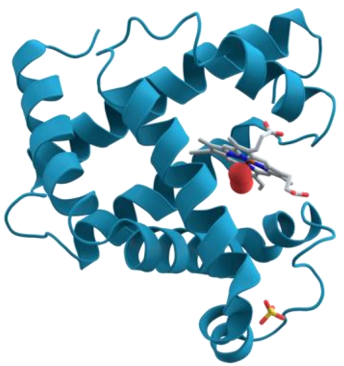


Myoglobin solution: Nb vs. quartz

After subtraction of the windows and normalization by H_2O

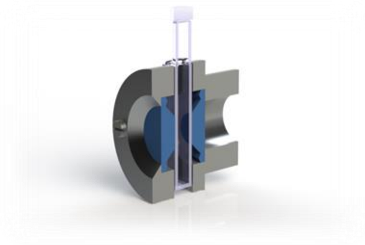


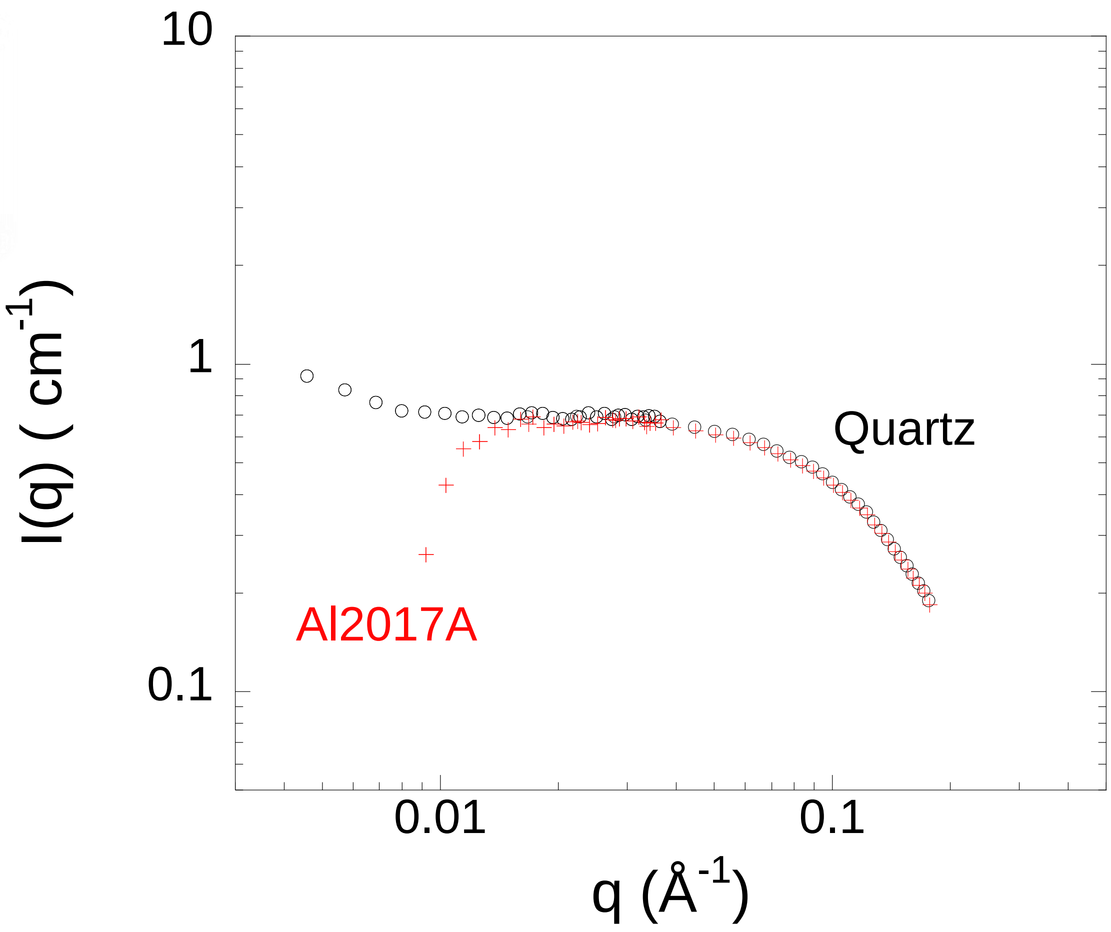




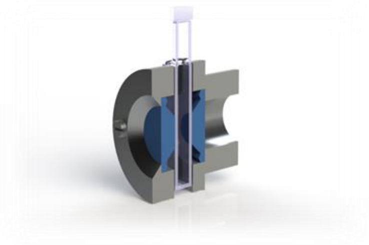
Myoglobin protein
(Mb, 20 g/L)

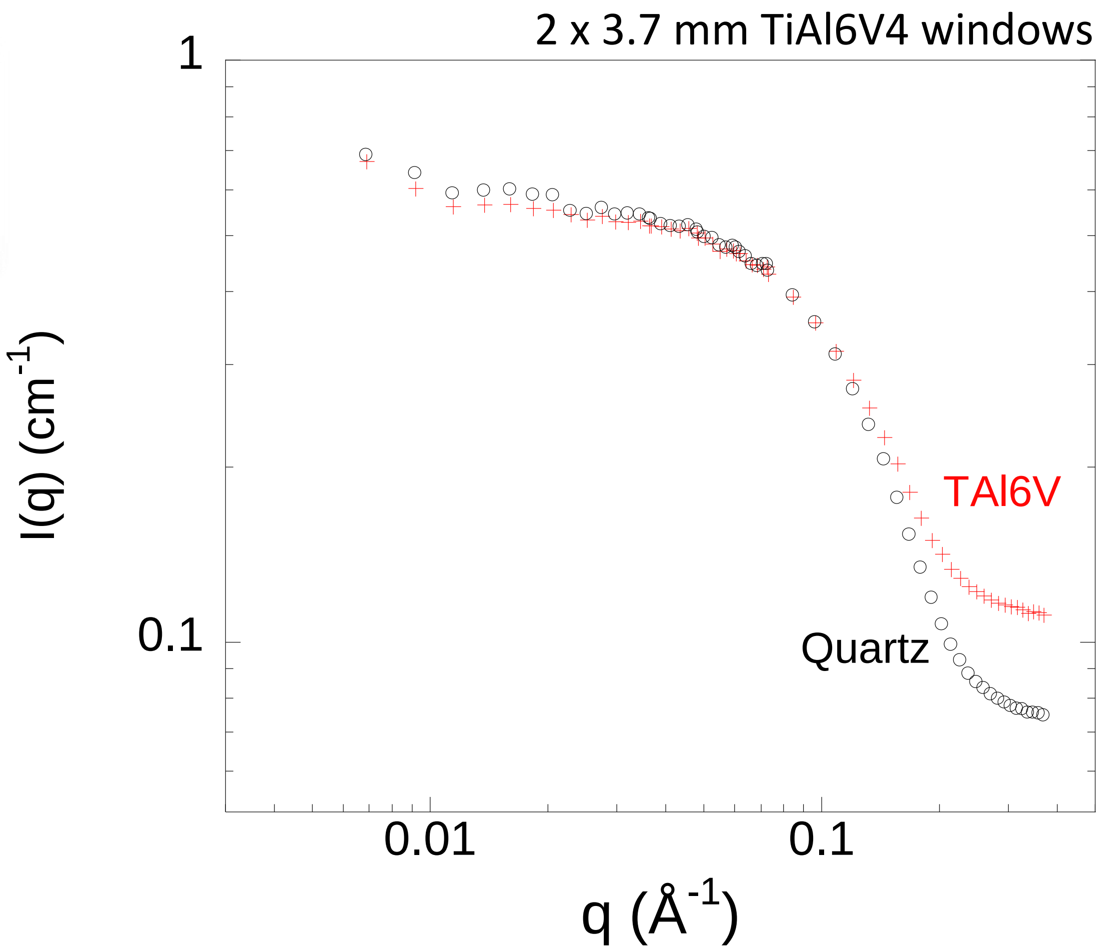
Myoglobin solution: Al2017A vs. quartz





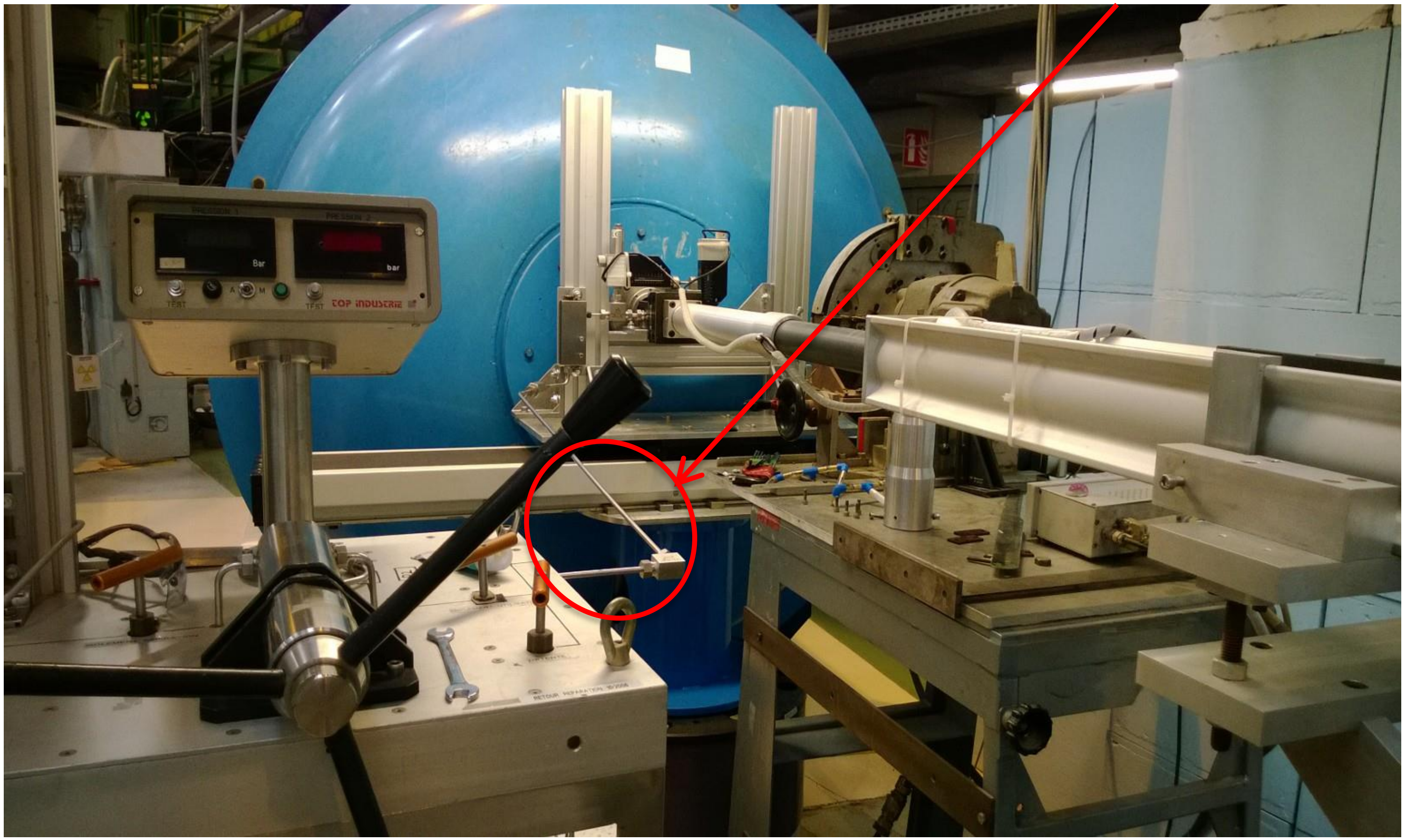
Myoglobin solution: TiAl6V4 vs. quartz





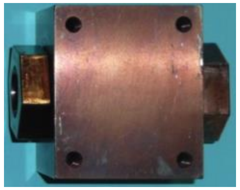
A real experiment with the SANS pressure cell

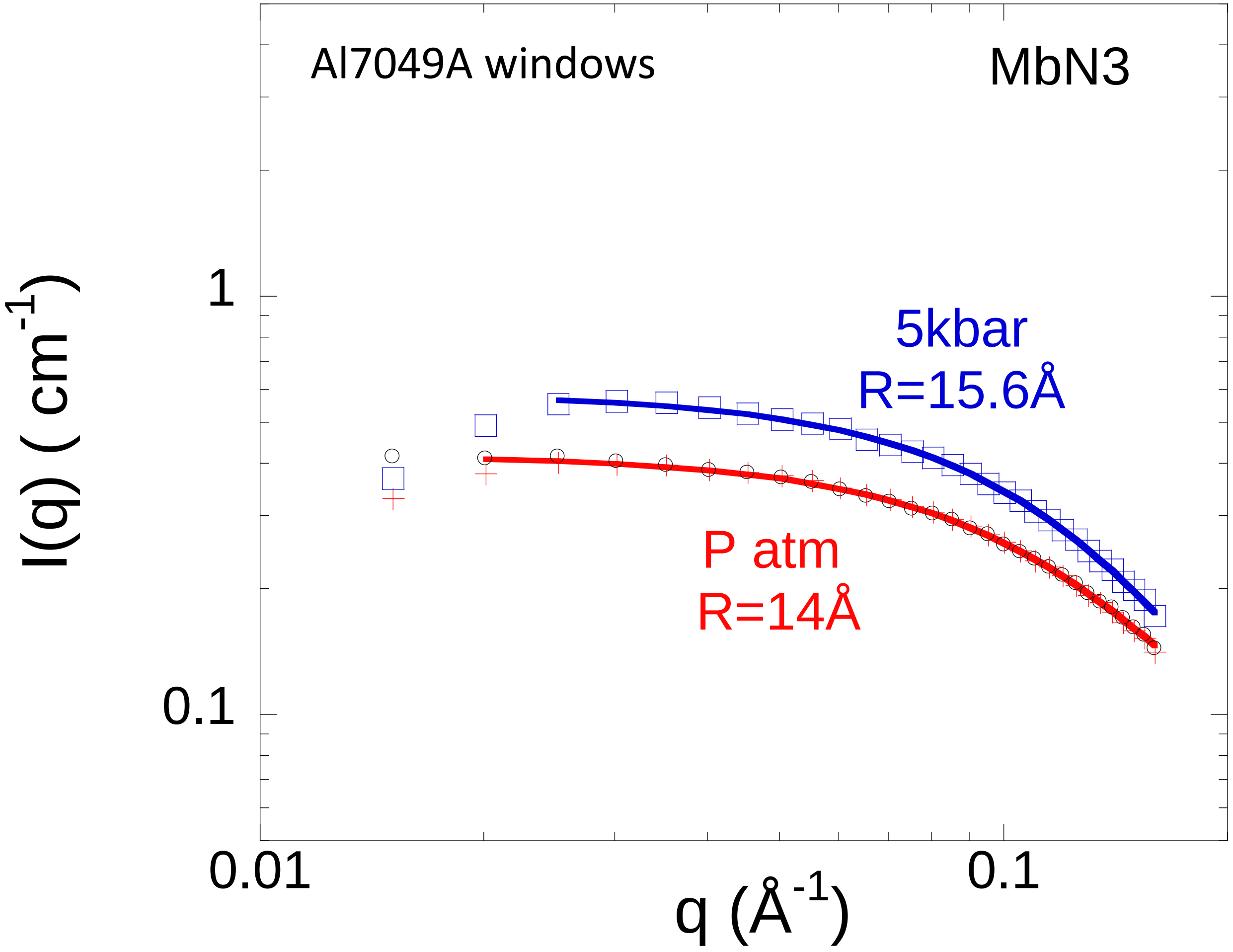
inox capillaries with 1.6 mm external diameter,
10 kbar resistance → a service pressure of 6 kbar



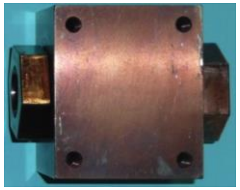
Pressure device on PACE SANS spectrometer (LLB)

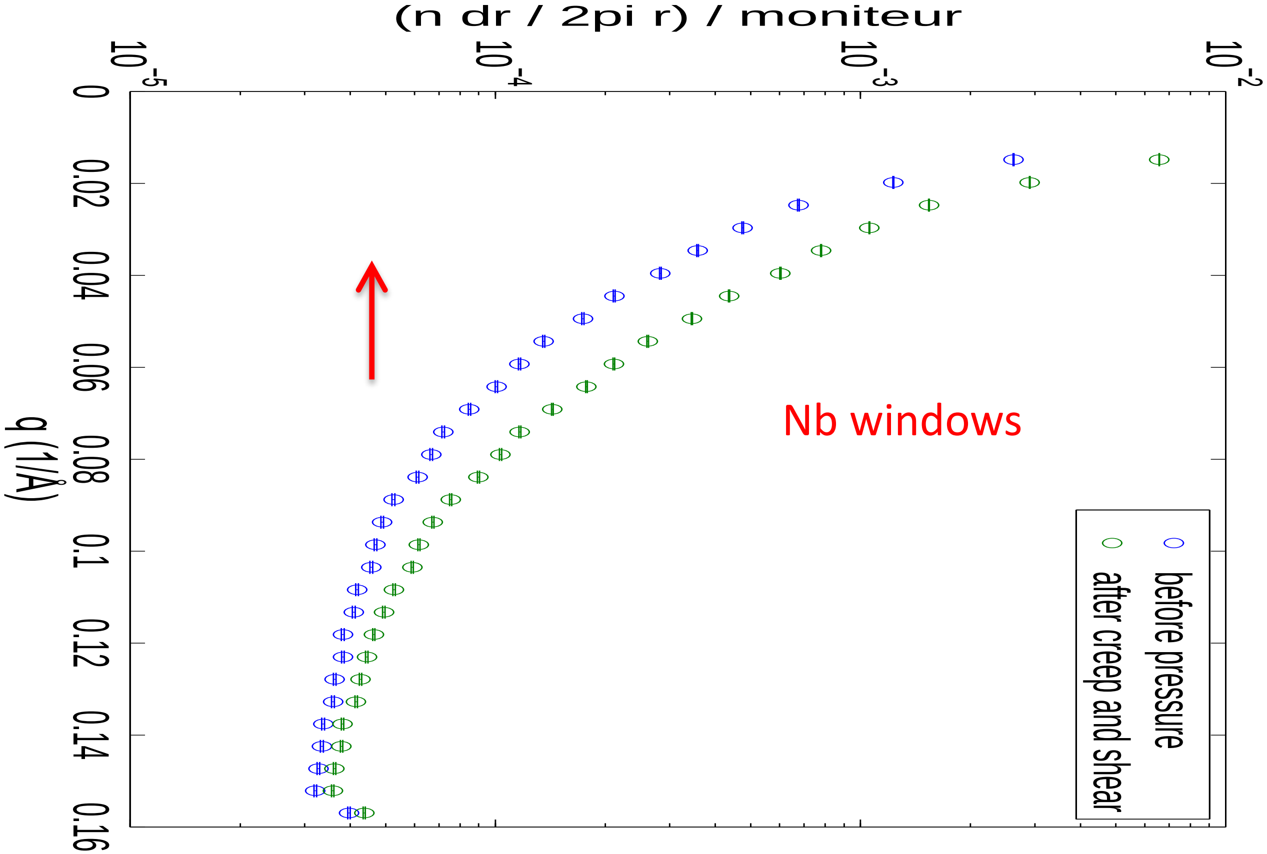
Effect of pressure on Mb





Effect of pressure on… windows





→ the solution: plastification of Nb at P_{max} before the pressure experiment

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!
- ✓ **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 only about **2.8 kbar**.
- ✓ **Aluminium** (Al7049A): very good transmission but quite high scattering → for a sample which scatters; can reach about **6-7 kbar**.
- ✓ **TiAl6V4**: promising (to be tested in a real experiment) despite its low transmission → recommended to decrease the window thickness. Its scattering is weak: in a wide q range it is comparable or a bit higher than the one of Nb. Pressure about **5-6 kbar** up to 10 kbar (depending on the thickness).

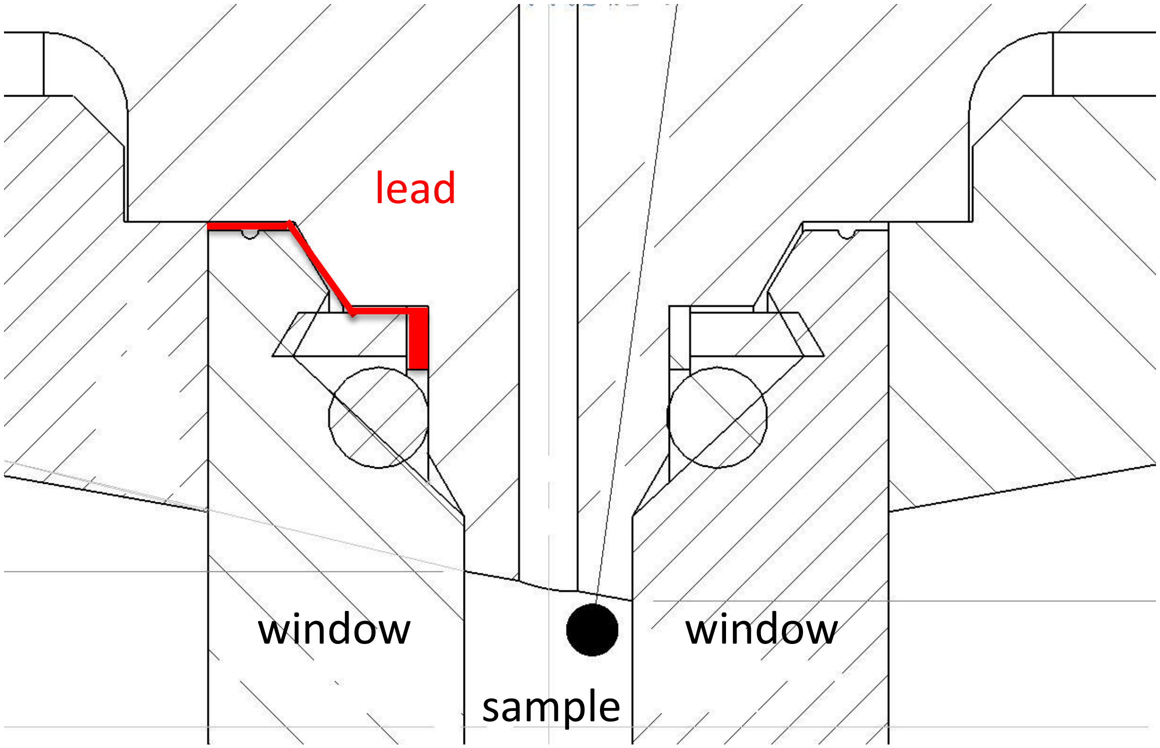
General remarks

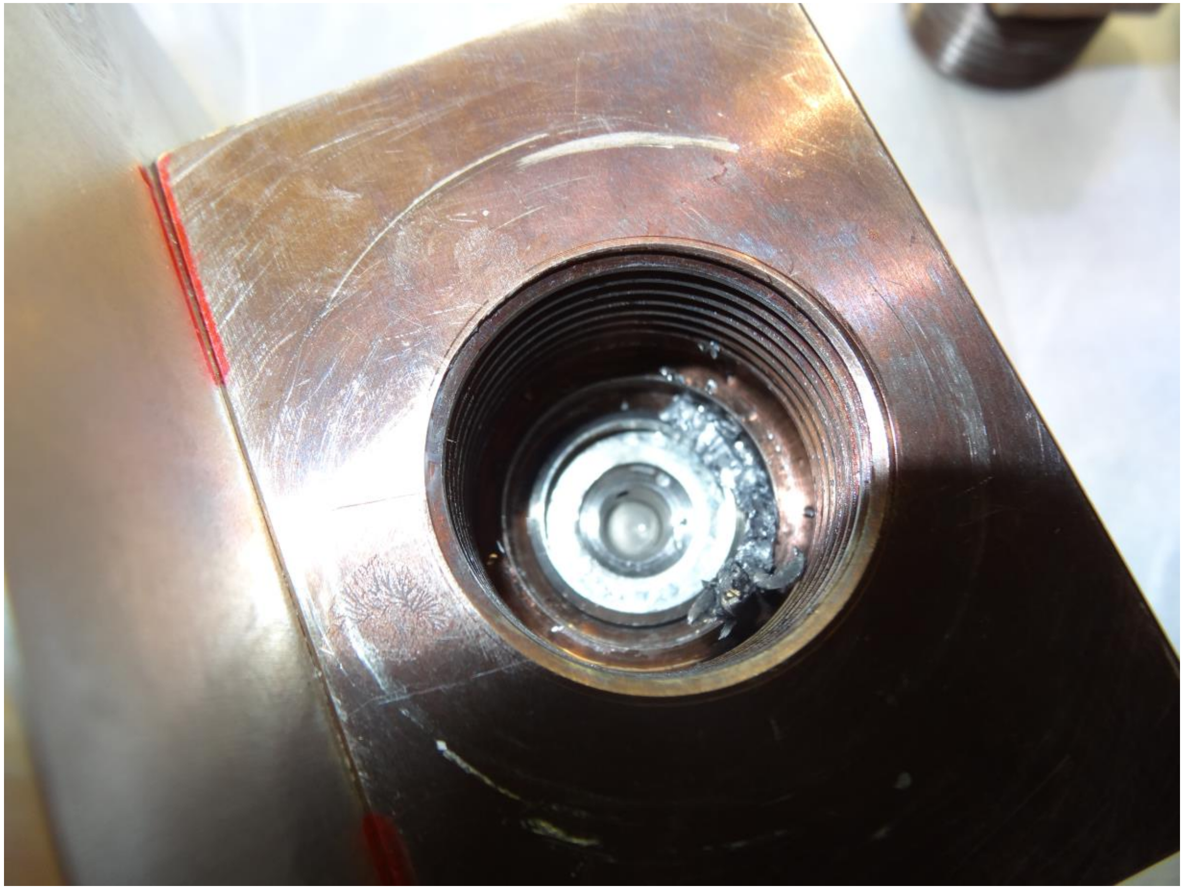
- ✓ The **sample scattering** has to be **above ~10-20% the one of windows**
  - for samples with low scattering -> sapphire should be better

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

- ✓ **Plastification**: necessary for Nb (and maybe TiAl6V4 → to be tested) before the pressure experiment; not necessary for Al7049A

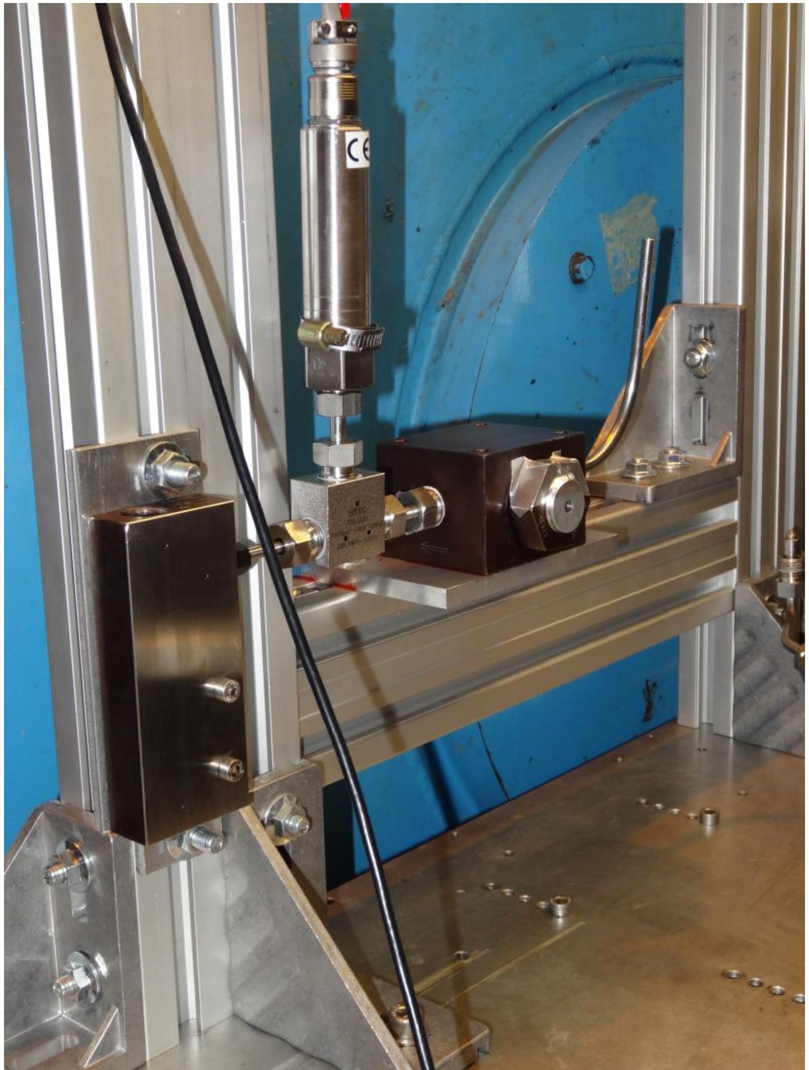
- ✓ **Change of the windows** during the experiment: possible only with tin-platted copper, not with lead


lead
window
window
sample



Results

 To fill and empty the cell: easy, no problem of air bubbles (so, no loss in pressure due to change of volume)



Results

- **Separation piston**: good sealing between the buffer in the sample room → no problem of sample or buffer leakage or mixing of sample/buffer solutions