High Pressure Cells

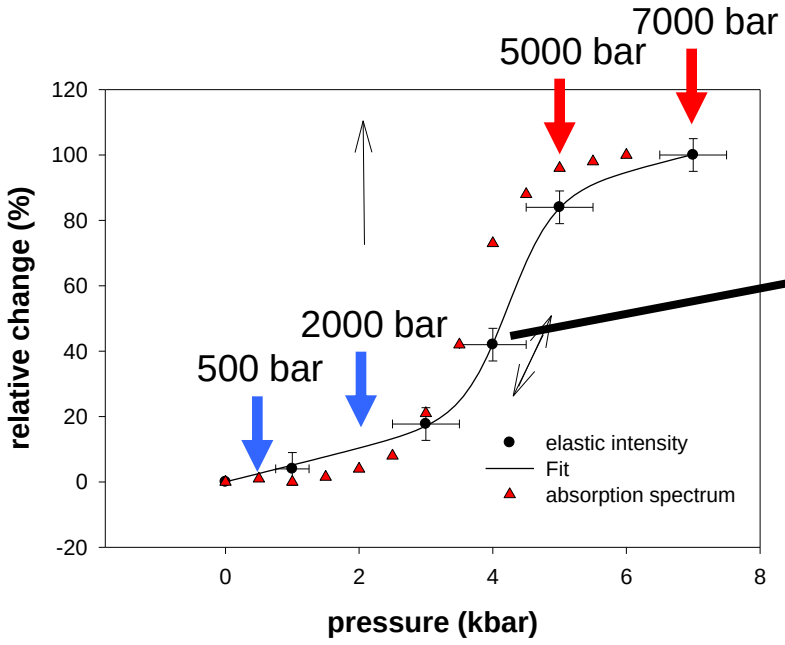
Marie-Sousai Appavou

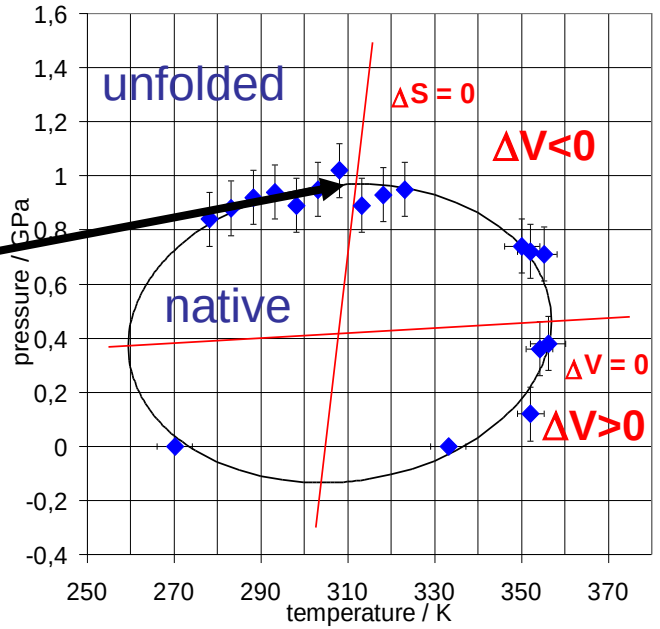
Henrich Frielinghaus

New pressure cells for neutron scattering on KWS, NSE, DNS and SPHERES : one example for protein denaturation

$$d(\Delta G) = -(\Delta S)\,dT + (\Delta V)dp$$

Pressure **$dp/dT = \Delta S / \Delta V$**



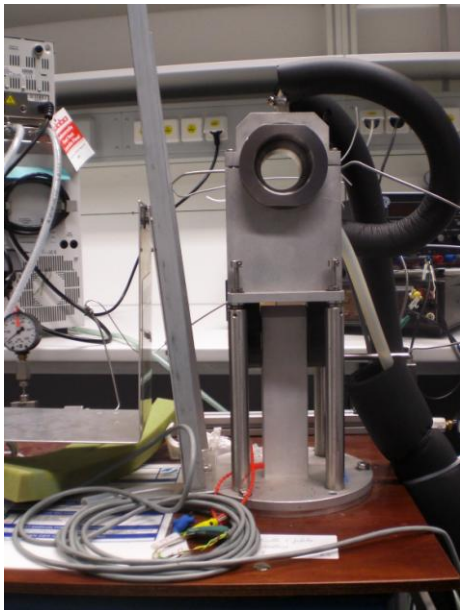


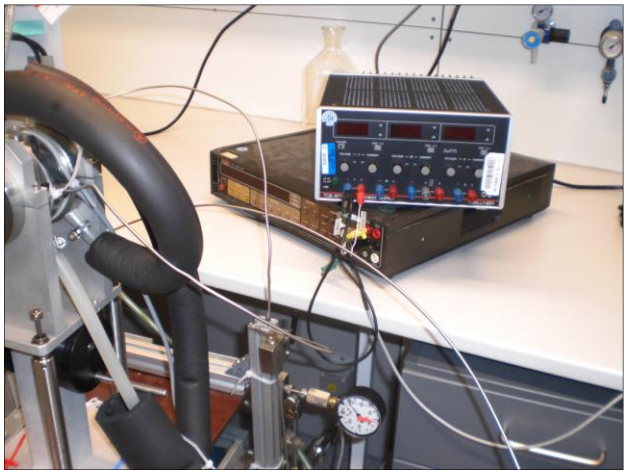
Elastic neutron intensity and Absorbance for Metmyoglobin in solution. **(Doster W, Gebhardt R, 2003)**

(Lesch et al. Biophys.J. 2001)

a pressure cell for spin echo neutron scattering on J-NSE:

What high pressure cells do we have :
At JCNS 500 bar :





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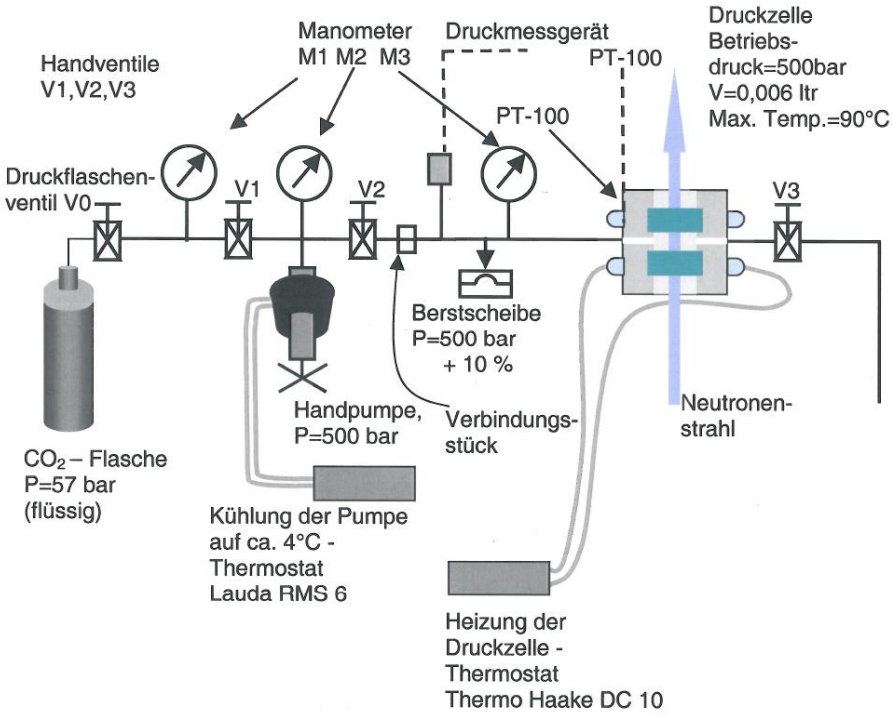
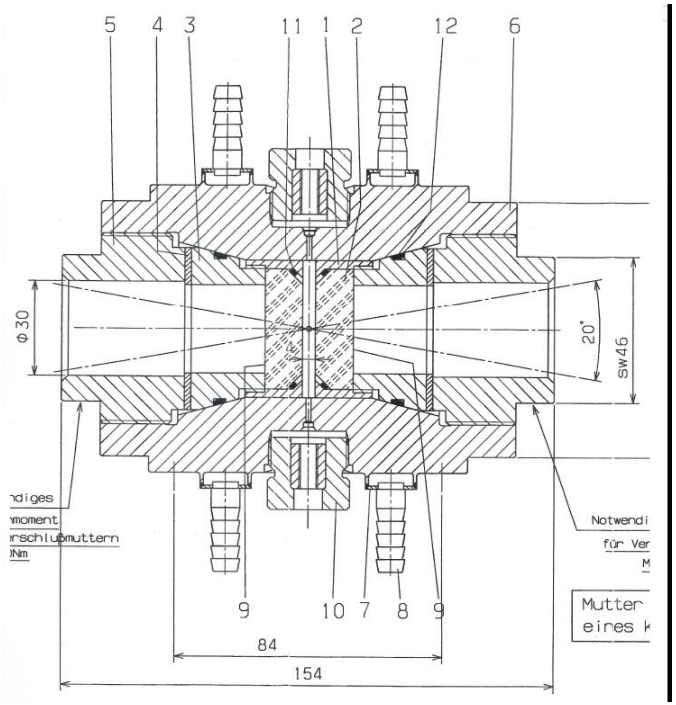
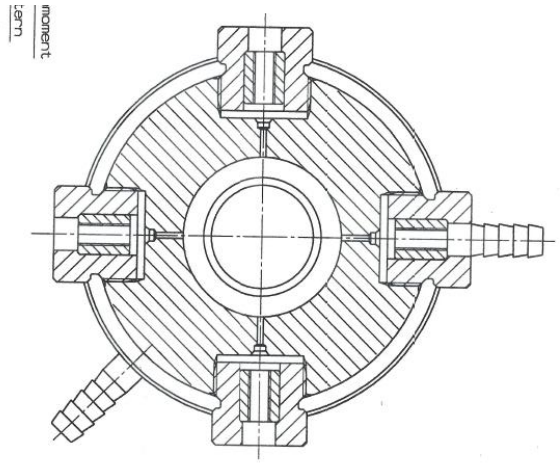
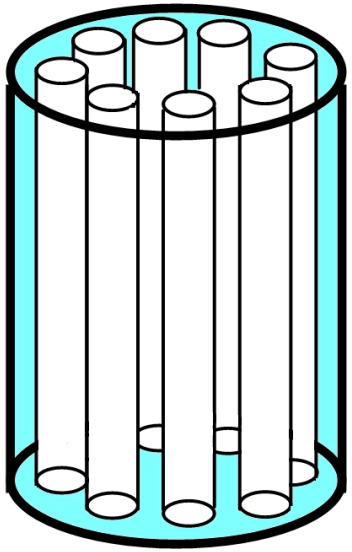






a new pressure cell for spin echo neutron scattering on J-NSE:



•Building a high pressure cell using sapphire block able to sustain 7000 bar : dynamics of pressure-unfolded protein investigated in the nanosecond timescale with Neutron Spin Echo spectrometer (Olaf Holderer)

a new pressure cell for neutron scattering on KWS :

What high pressure cells exist in other facilities :
At PSI 5000 bar:

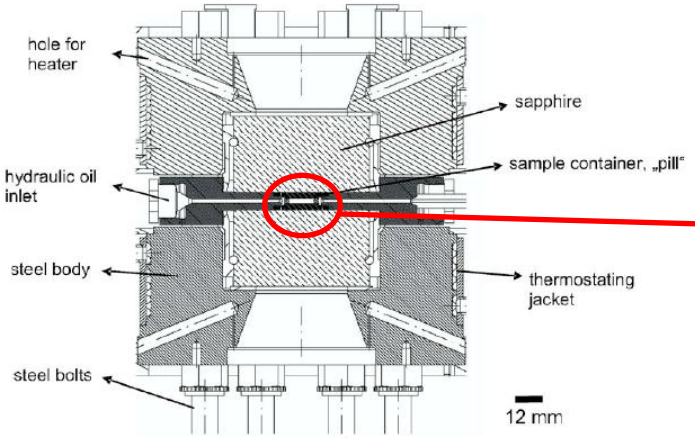


FIG. 1. (Color online) Middle cut through the high pressure cell.

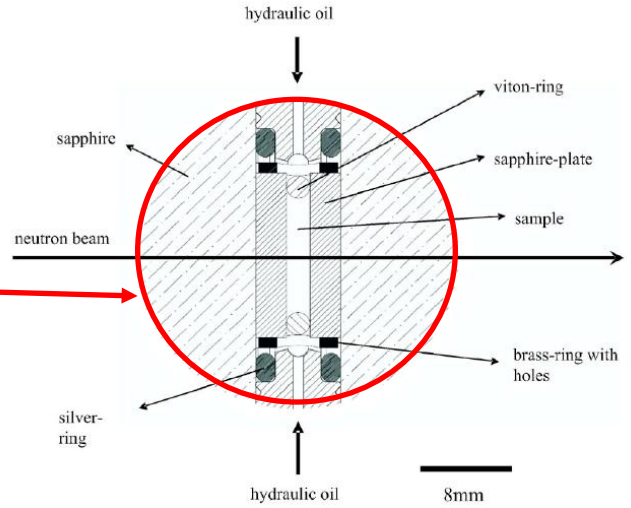


FIG. 3. (Color online) Shown is an inner circular section of the cell as in Fig. 1 with details of the sample container (pill). The cut is along the holes of the brass ring to illustrate where the hydraulic oil can transmit the pressure via the Viton O-ring.

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A high pressure cell for small angle neutron scattering up to 500 MPa in combination with light scattering to investigate liquid samples

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G. Meier
IFF, weiche Materie, FZ-Jülich, Postfach 1913, 52428 Jülich, Germany

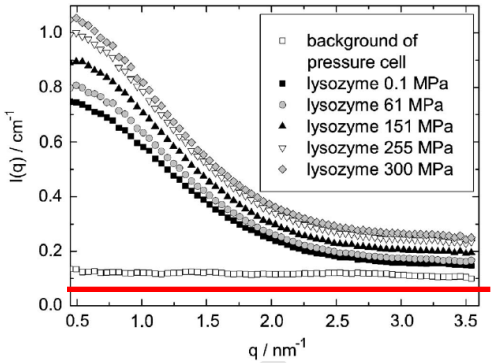


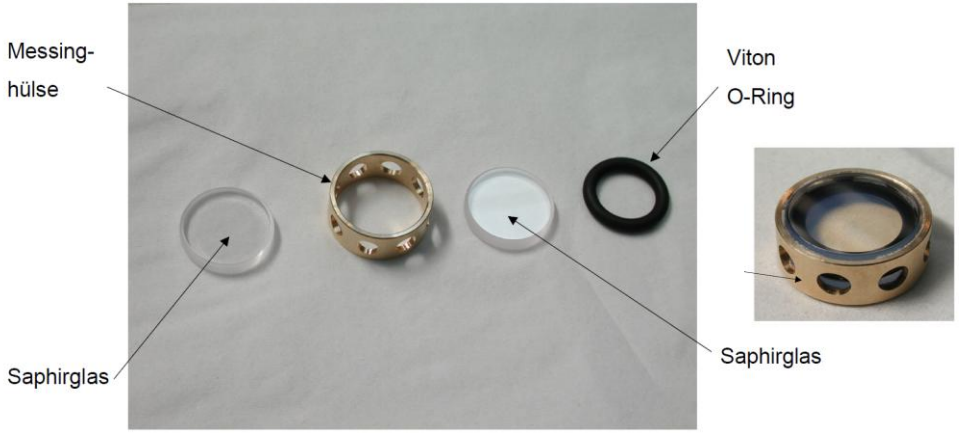
FIG. 7. SANS results for lysozyme in D_2O as a function of pressure as indicated. Also shown for comparison is the background of the high pressure cell. It shows no q dependence and amounts to about 0.12 cm^{-1}.

Level for D2O : 0,03 cm^{-1}
Level for deuterated phosphate buffer : 0,05 cm^{-1}

a new pressure cell for neutron scattering on KWS :

Befüllung der Probenkapseln für die 500 MPa Zelle

Einzelteile der Probenkapsel

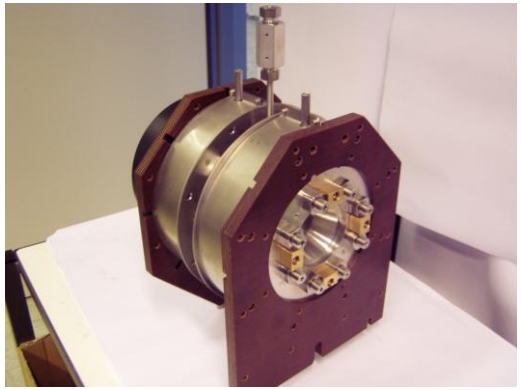
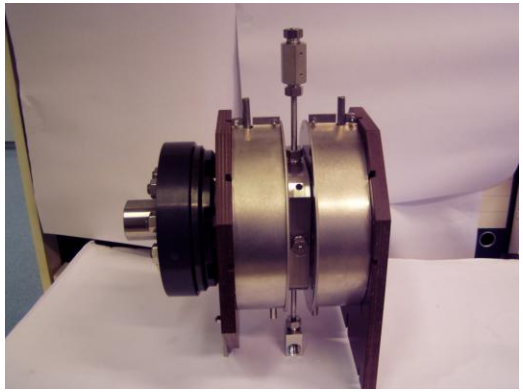
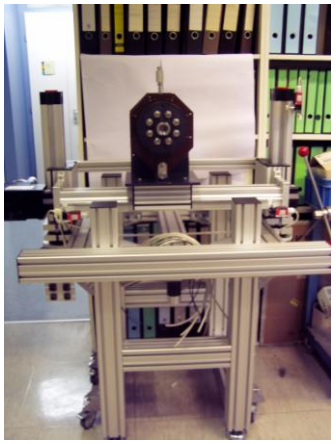
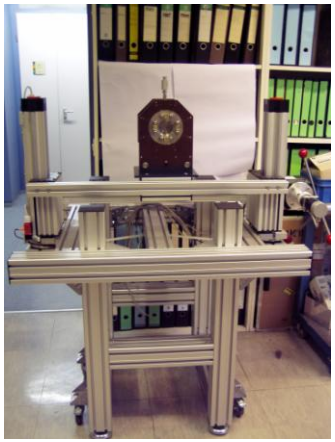


Die zerlegte Hochdruckzelle

From J. Kohlbrecher

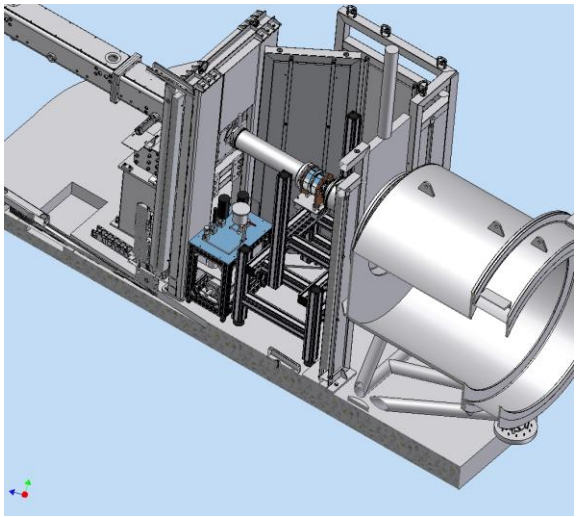
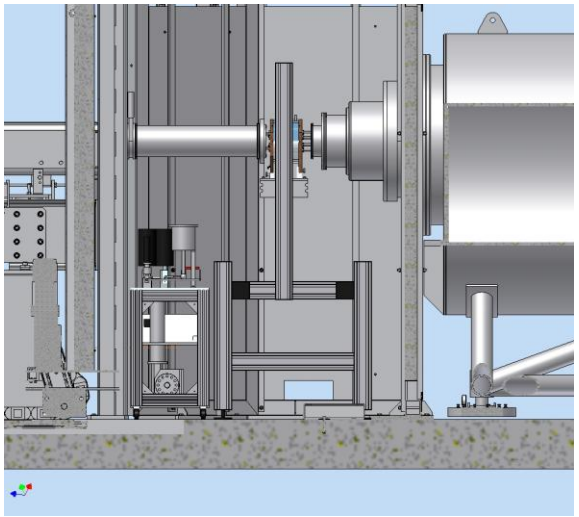
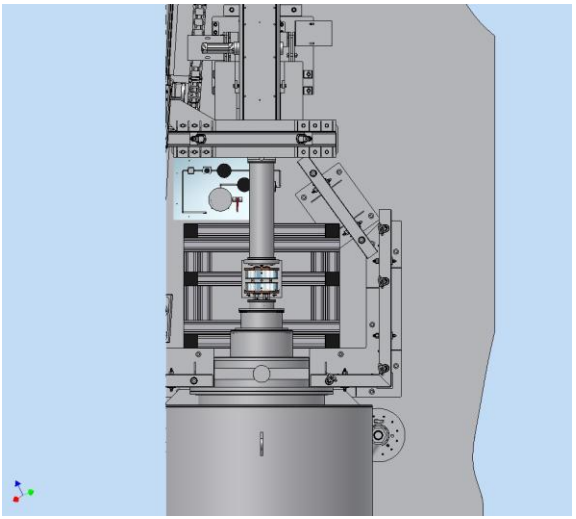
a new pressure cell for neutron scattering on KWS :

What we have at the moment

From H. Feilbach

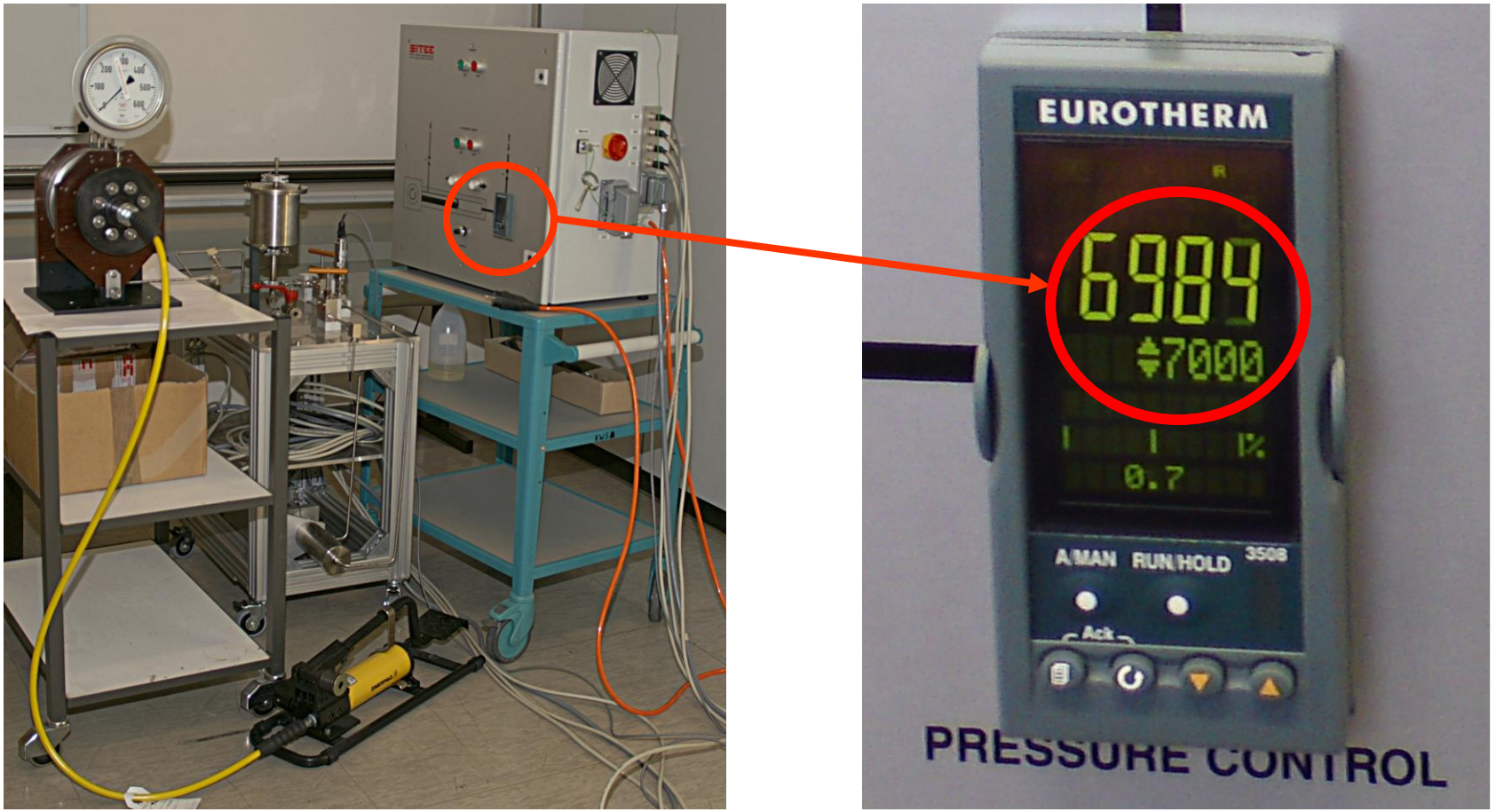
We are using Inconel : a non magnetic material (for polarization analysis and NSE)

From M. Heiderich

Second lab-tests : 21rst of January 2010

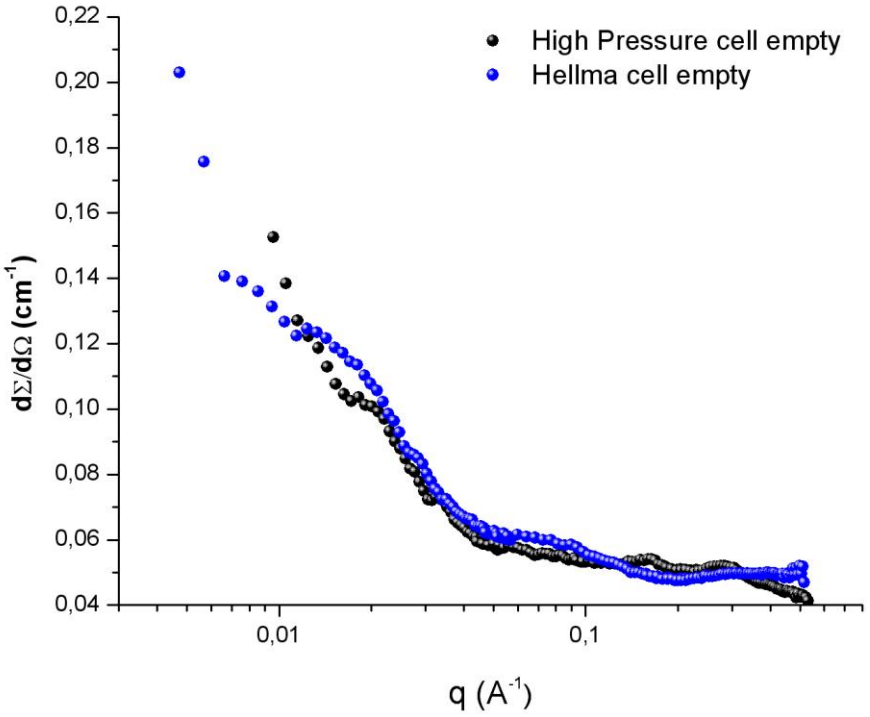
push till the limit



Successful TÜV-test at 7000 bar pressure within 1 % error, 5000 bar allowed by TÜV

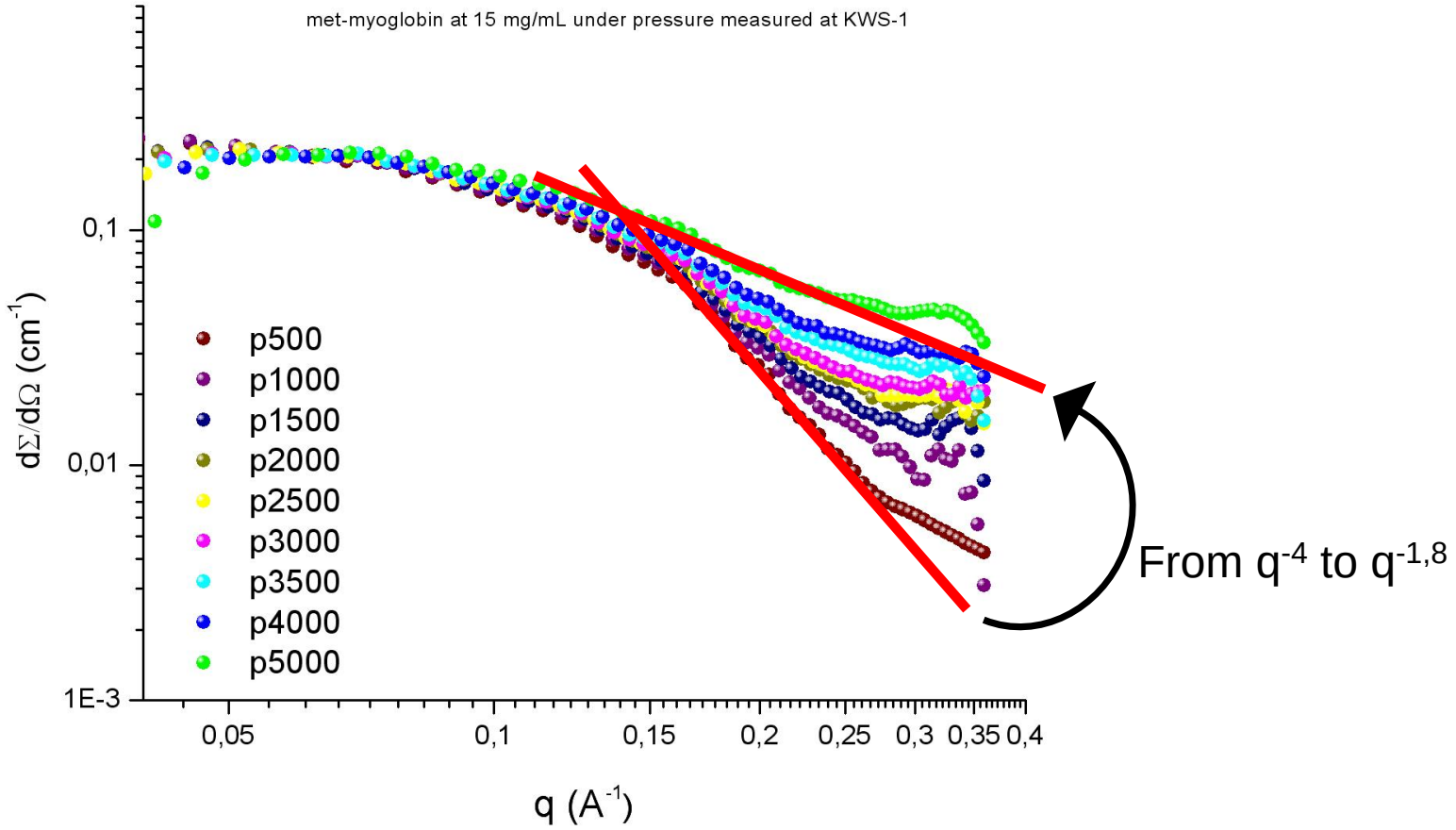
From H. Feilbach, M. Heiderich

In the instruments : KWS-2

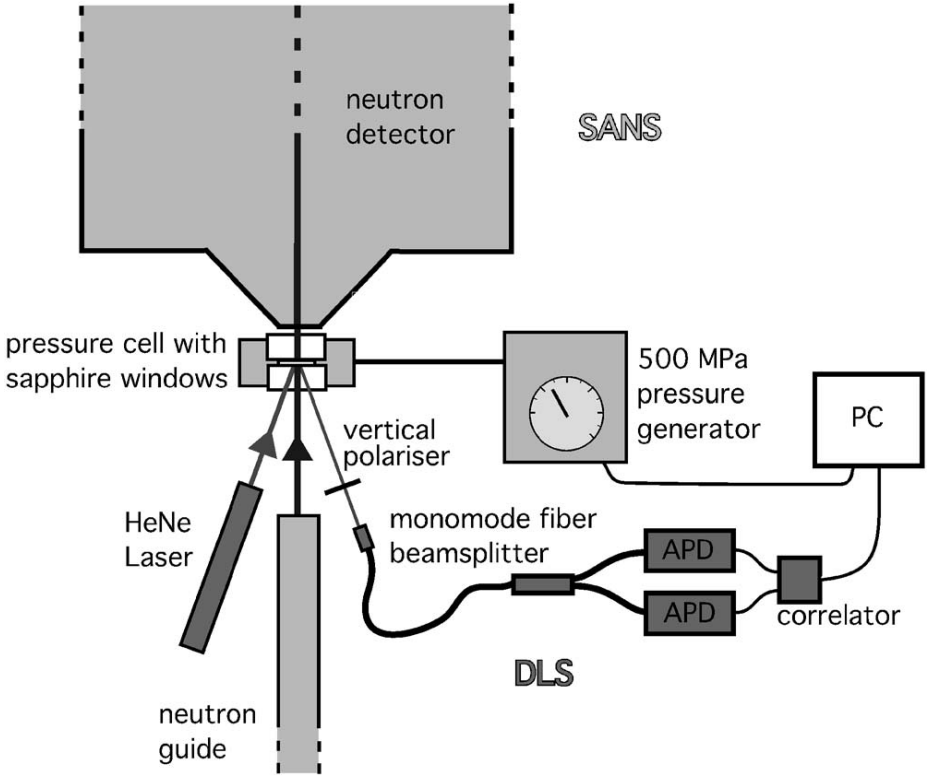


Transmission Empty cell (λ = 4,5 Å)= 0,588

In the instruments : KWS-1



Optical spectroscopy under pressure



Possibility to perform static and dynamic light scattering using the high pressure set-up

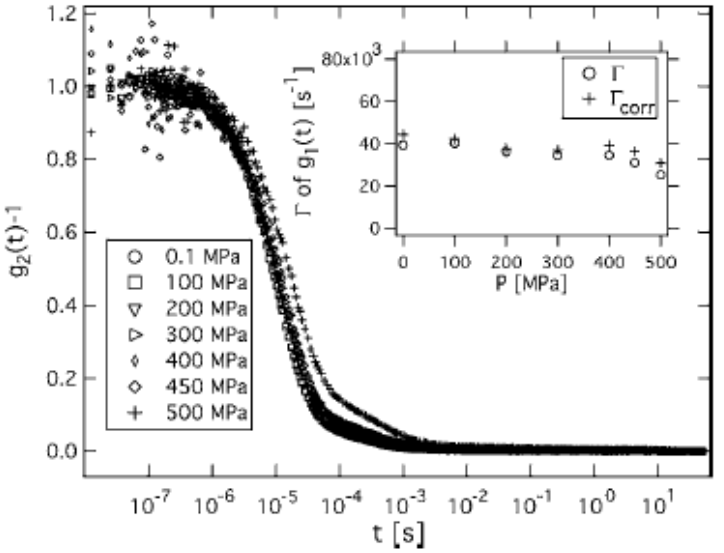


FIG. 6. Normalized intensity autocorrelation functions for lysozyme (50 mg/ml) in 50 mM acetate buffer as a function of pressure as indicated. In the inset, the values for the pressure dependence of the bare relaxation rates Γ and Γ_{corr} are shown (the latter is corrected for the pressure dependencies of η and n).

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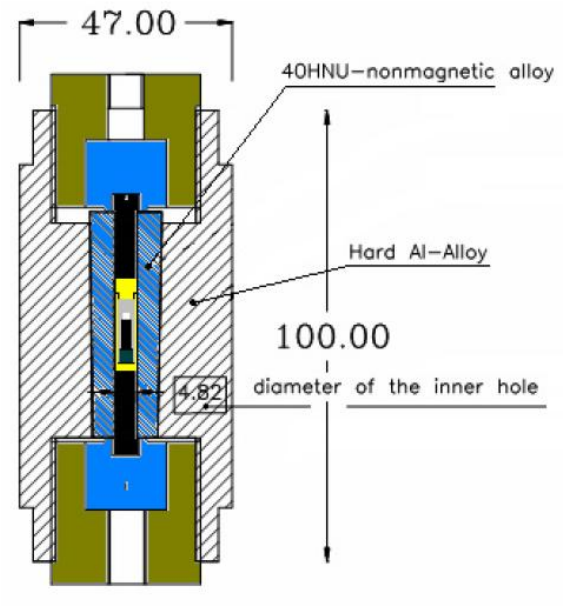
A high pressure cell for small angle neutron scattering up to 500 MPa in combination with light scattering to investigate liquid samples

J. Kohlbrecher, A. Bollhalder, and R. Vavrin[a)]
Laboratory for Neutron Scattering, ETH Zurich and Paul Scherrer Institut, 5232 Villigen PSI, Switzerland

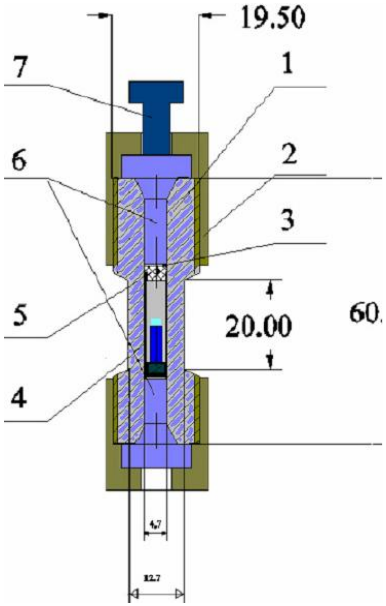
G. Meier
IFF, weiche Materie, FZ-Jülich, Postfach 1913, 52428 Jülich, Germany

a new pressure cell for neutron scattering on other instrument :

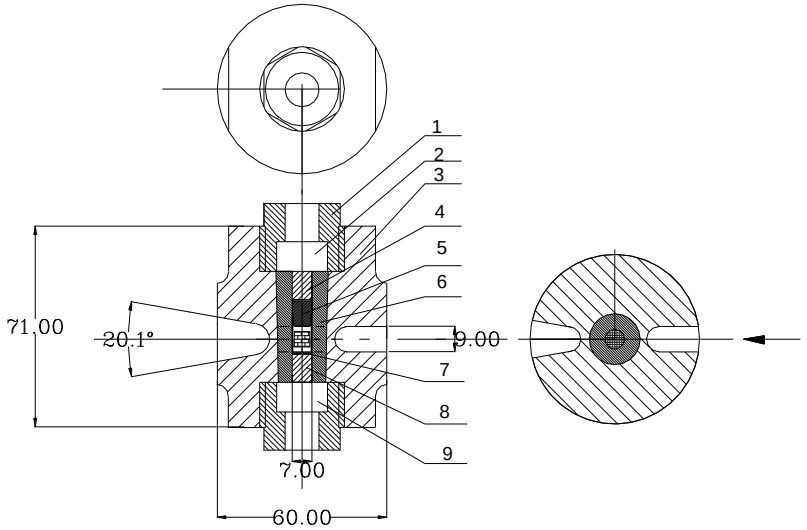
use of NiCrAl alloy, another non magnetic material



P_{RT} (T=300K)=30.5kbar
P_{LT}(T=1,5K) =32.9kbar



Nonmagnetic High Pressure Cell
up to 20kbar
(HMI-2003, ILL-2004-2005)



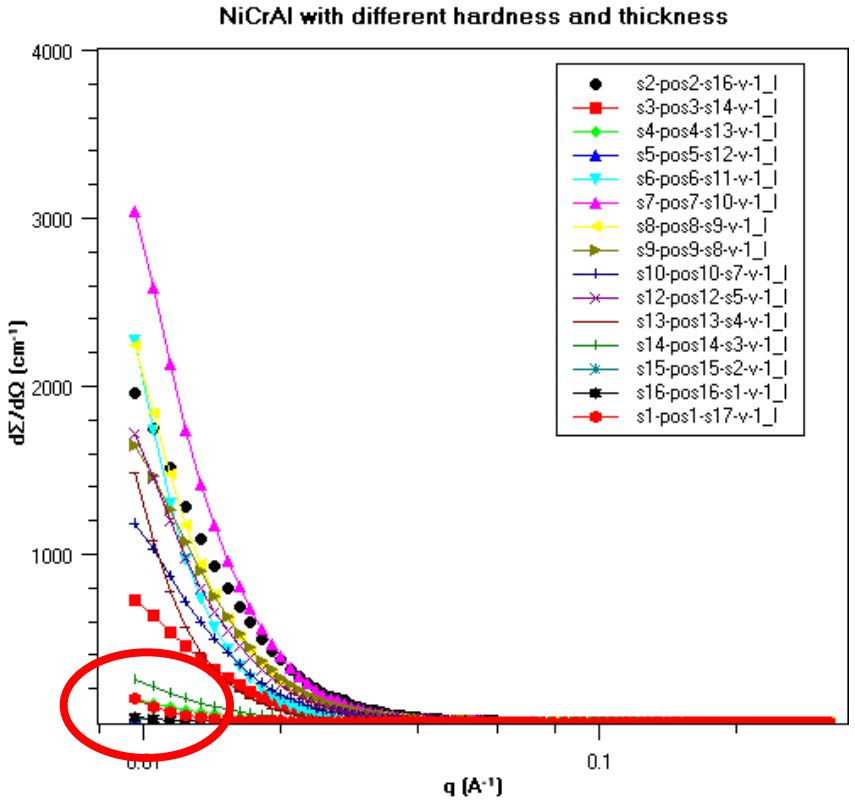
Pressure cells for SANS
GKSS-Dec.2007

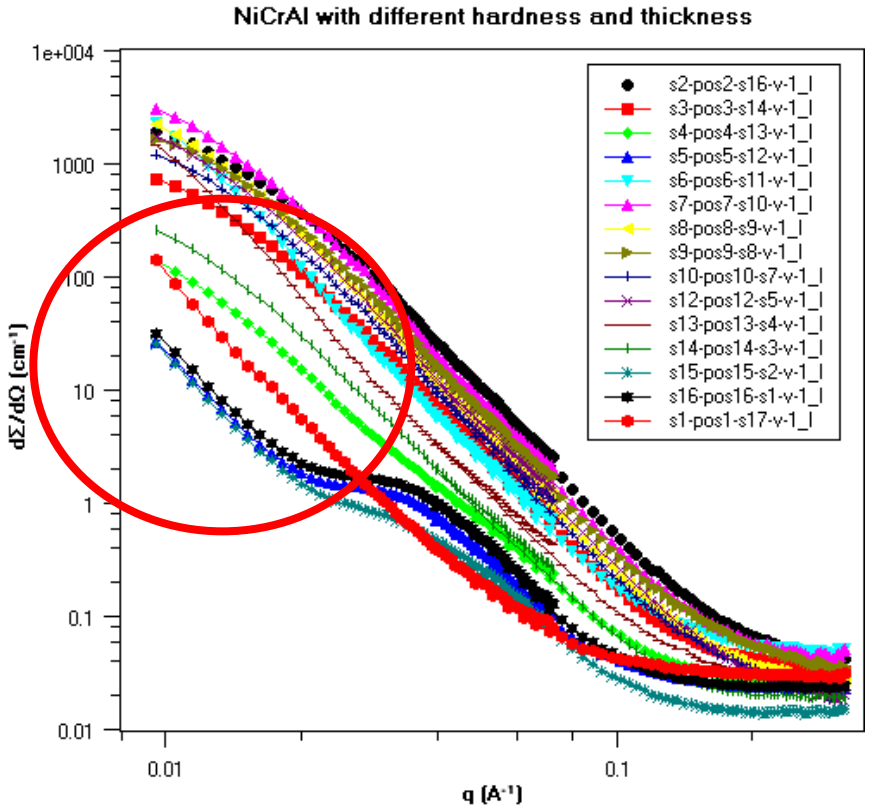
- P=15 kbar
- Still high background from the NiCrAl insert

From R. Sadykov

a new pressure cell for neutron scattering on other instrument :

use of NiCrAl alloy, another non magnetic material





Test measurement on KWS-2 at 4.5 Å
Still to much scattering in the low q-range

From R. Sadykov, V. Litvin and M.S. Appavou

a new pressure cell for neutron scattering on other instrument :

use of NiCrAl alloy, another non magnetic material

sample	thickness, cm	hardness HRc	transmission
s1	0.2	-11.0	0.4725
s2	0.2	16.0	0.516
s3	0.2	27.0	0.5037
s4	0.2	45.0	0.3006
s5	0.2	58.0	0.3501
s7	0.2	48.0	0.407
s8	0.3	48.0	0.347
s9	0.2	54.0	0.33
s10	0.3	54.0	0.2731
s11	0.5	45.0	0.1403
s12	0.5	18.0	0.3167
s13	0.5	24.5	0.3007
s14	0.5	39.0	0.2492
s16	0.5	58.8	0.1575
s17	0.5	23.0	0.2624

Test measurement on KWS-2 at 4.5 Å
Still to low transmission

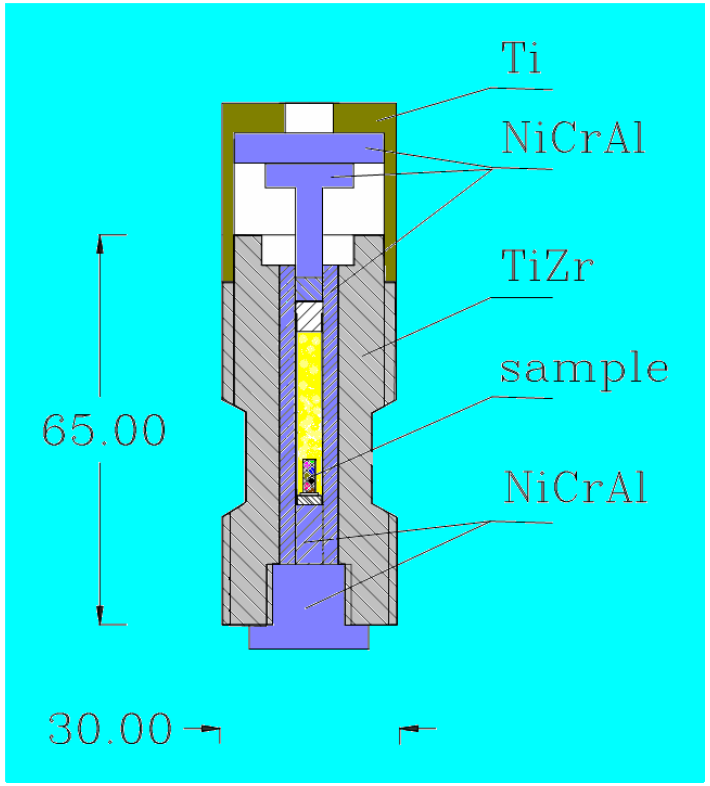
For a specific thickness, hardness is related to pressure sustain

We should extend the investigation to higher q-range on other instrument

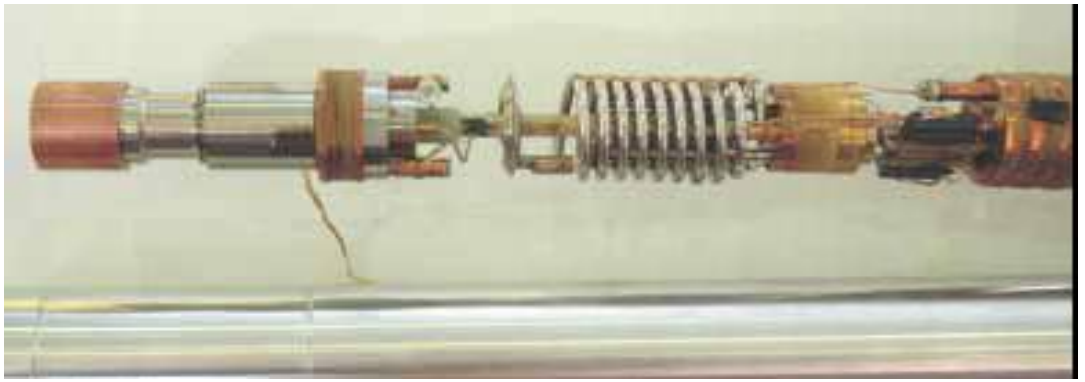
From R. Sadykov, V. Litvin and M.S. Appavou

a new pressure cell for neutron scattering on other instrument :

use of TiZr alloy with NiCrAl alloy



Nonmagnetic High Pressure Cell with TiZr support: up to 25kbar (ISIS 2001)

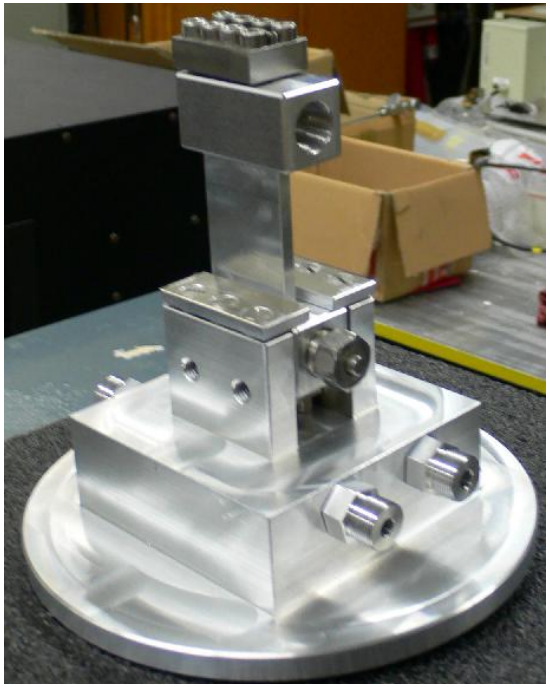


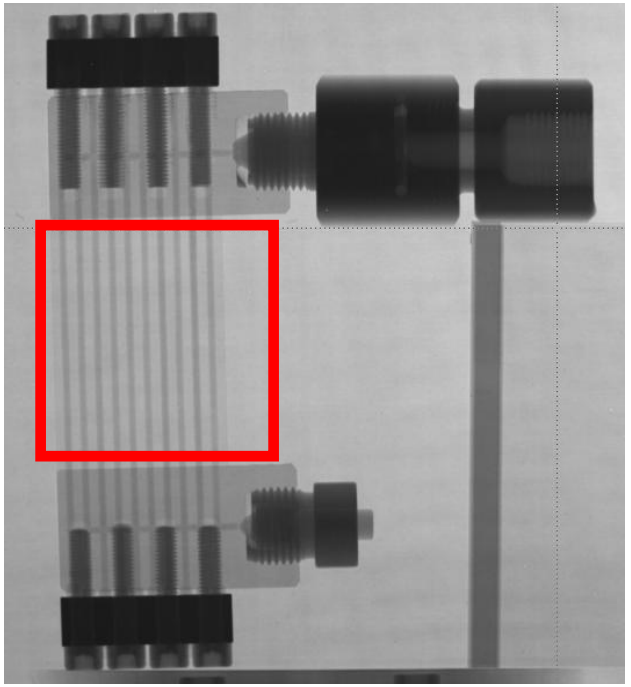
The large bore TiZr + NiCrAl alloys piston cell mounted on the dilution fridge insert.
The cell can accept a crystal up to 4,7mm in diameter and operates at pressures up to 2.5GPa at low temperature.

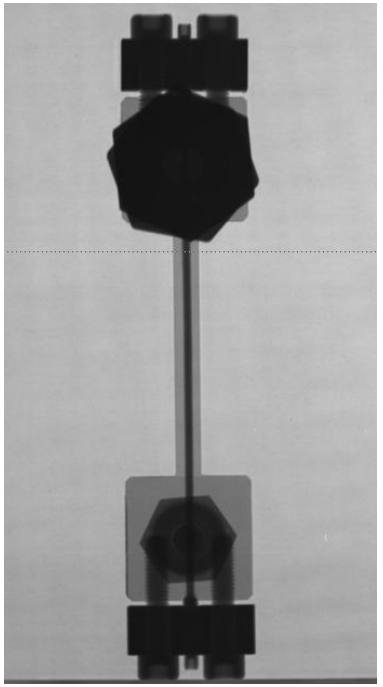
From R. Sadykov

a new pressure cell for neutron scattering on other instrument :

use of TiZr alloy, another geometry







Here is a high pressure cell in AL7075 aluminium alloy for 2000 bar

The calculated transmission of the cell at λ = 6 Å between the channels (5mm thickness) is about 91,2 %, whereas the transmission through the cell (3.4mm thickness) is around 93,9%.

TiZr may allow to sustain pressure up to 7000 bar

Open Questions

- 500bar NSE cell ok!
- 7kbar NSE cell (sapphire) to be reconsidered.
- 5 (7) kbar SANS pressure cell → user friendly
- Metal based cells for:
  - Higher pressure (20 kbar)
  - Other instruments

Thanks to

- Hr Herbert Feilbach
- Hr Kurt Hirtz
- Hr Manfred Heiderich

- Dr Joachim Kohlbrecher
- Dr Rony Vavrin

- Dr Ravil Sadykov
- Hr Vasily Litvin

- Joachim Dörbecker
- Reinhold Funer