Diamond anvil and clamp cells

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2019-2022

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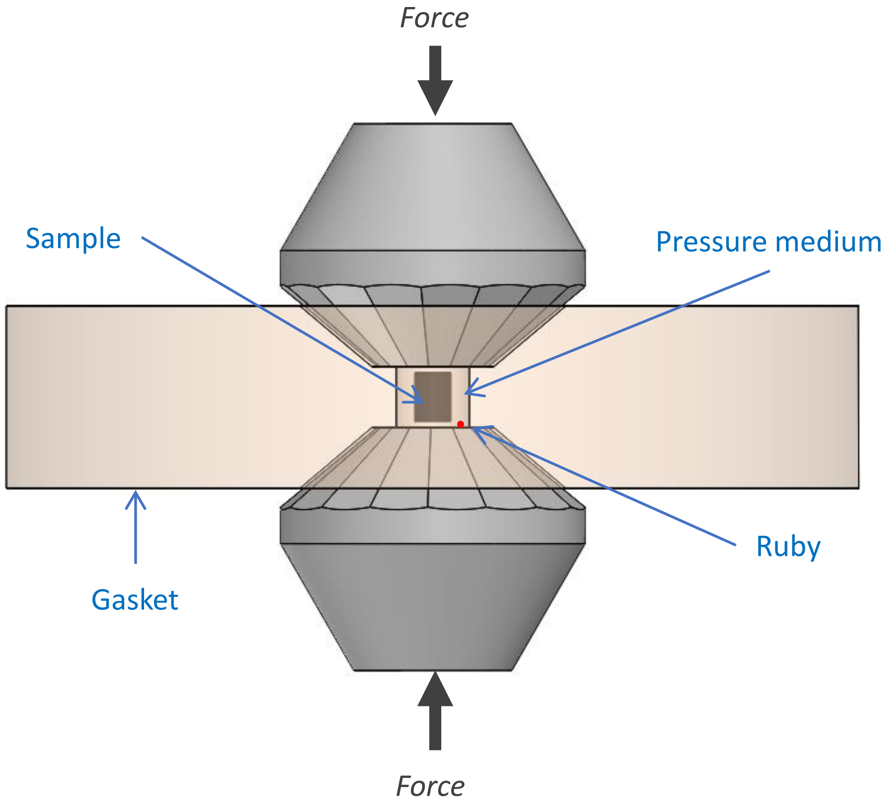
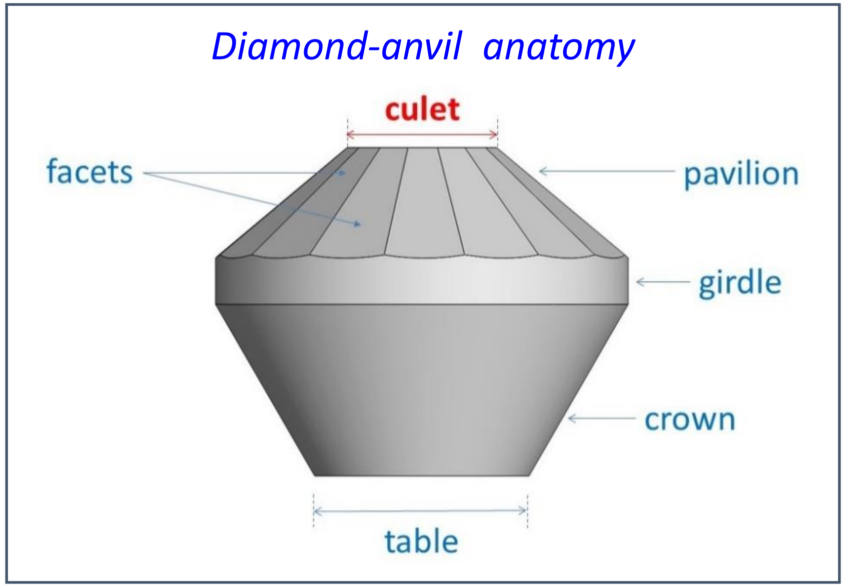
GE971/5-2

Diamond anvil cell (DAC) – the most versatile tool in the high-pressure technology

- X-ray diffraction and absorption
- Inelastic X-ray scattering
- Raman and infrared spectroscopy
- Optical spectroscopy
- Transport measurements
- Magnetism

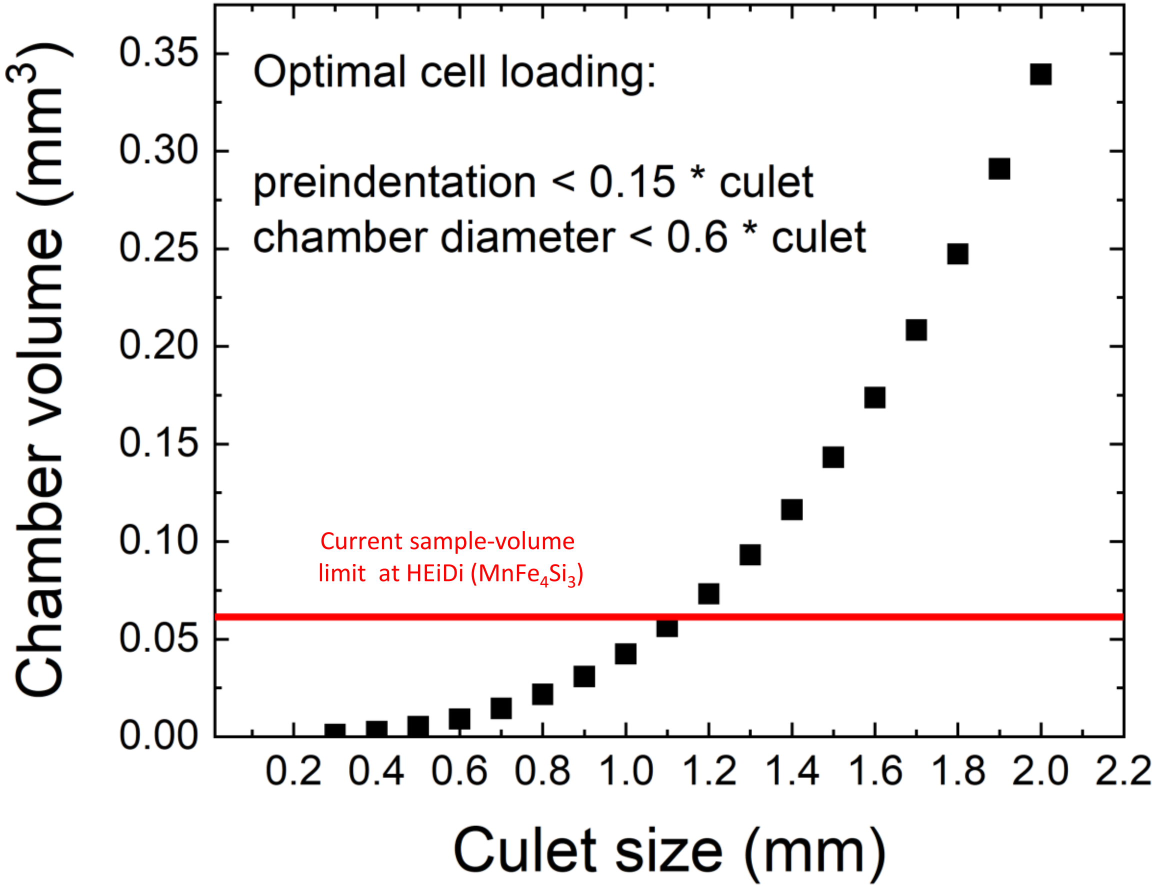


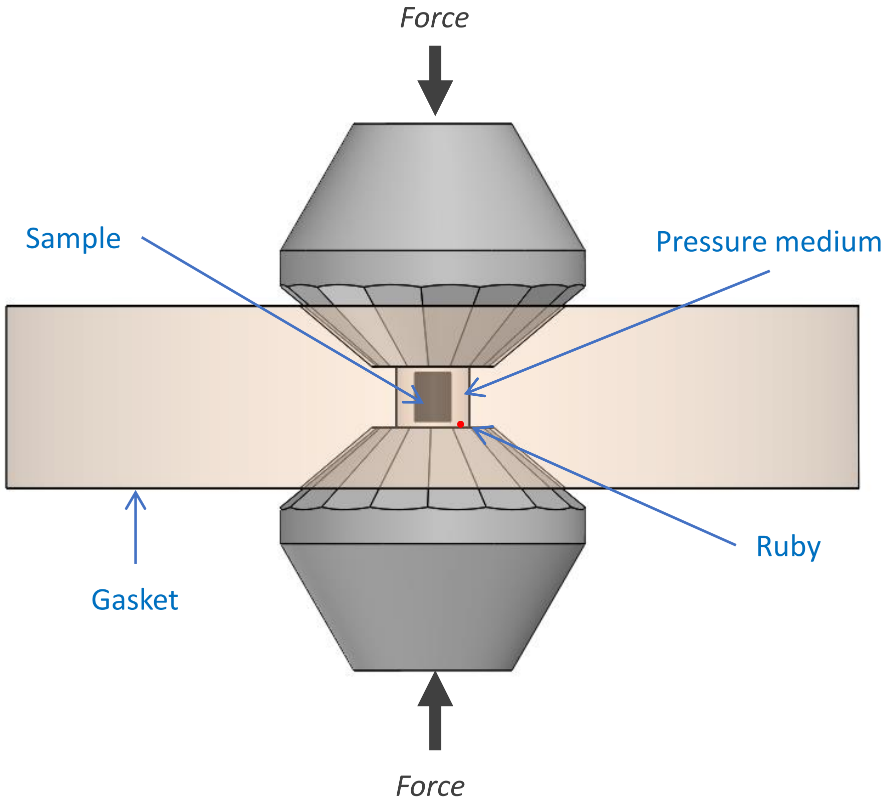


Some relevant properties of diamond:

- a good electrical insulator & heat conductor
- „transparent“ to hard X-rays
- very weak attenuation of hot neutrons

Diamond anvil cell (DAC) – the most versatile tool in the high-pressure technology





Diamonds: 1/8 to 1/3 carat (25 to 70 mg)

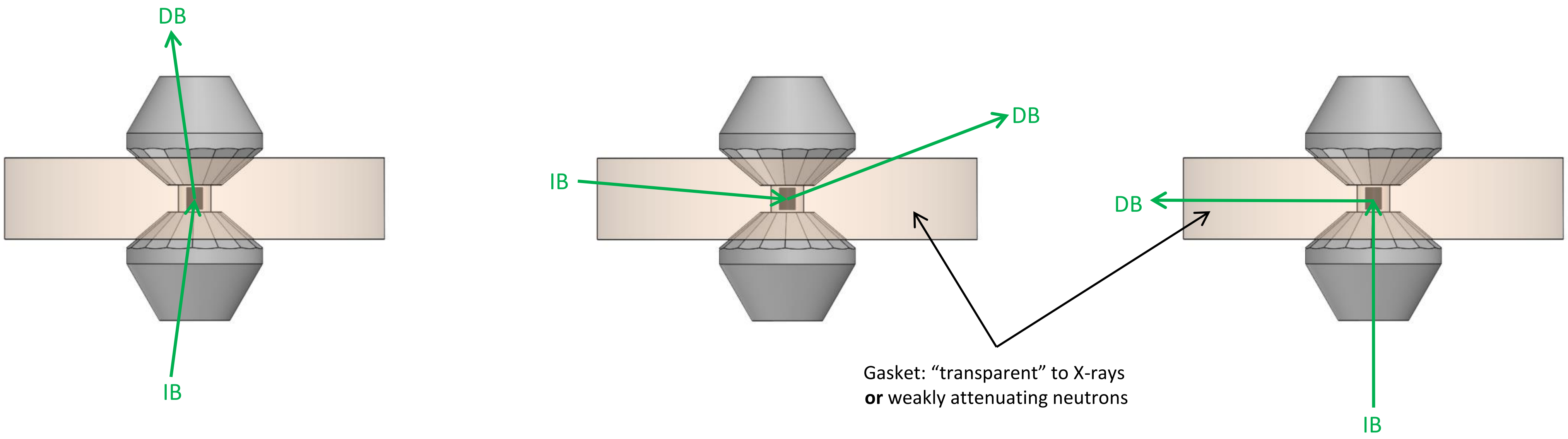
Diameter of the diamond culets:

1 mm → ~ 5-10 GPa

500 μm → ~ 30 GPa

250 - 300 μm → ~ 100 GPa

Experimental geometries for X-ray and neutron diffraction



"*transmission*"
X-rays **and** neutrons

"*panoramic*"
X-rays **or** neutrons

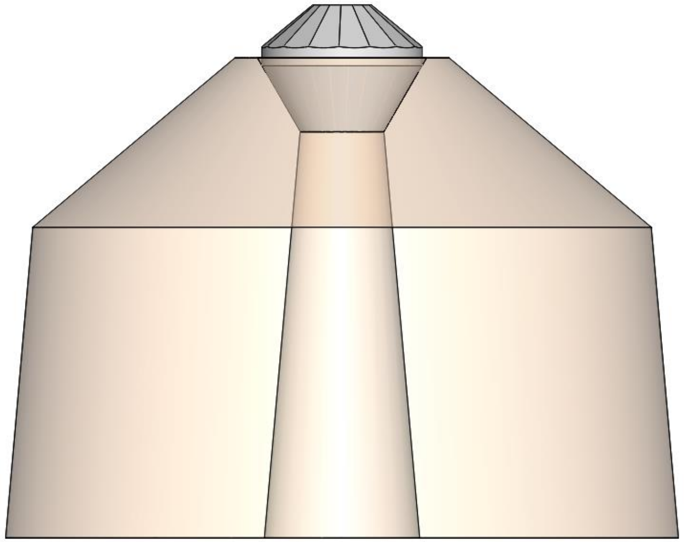
"*90°*"
X-rays **or** neutrons

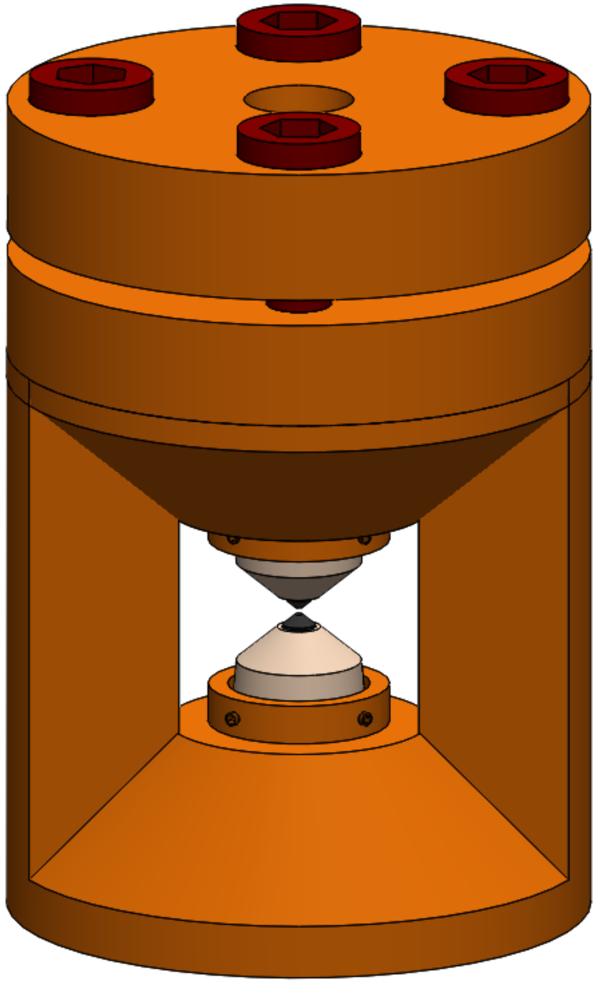
Monochromatic hot neutrons at HEiDi & POLI

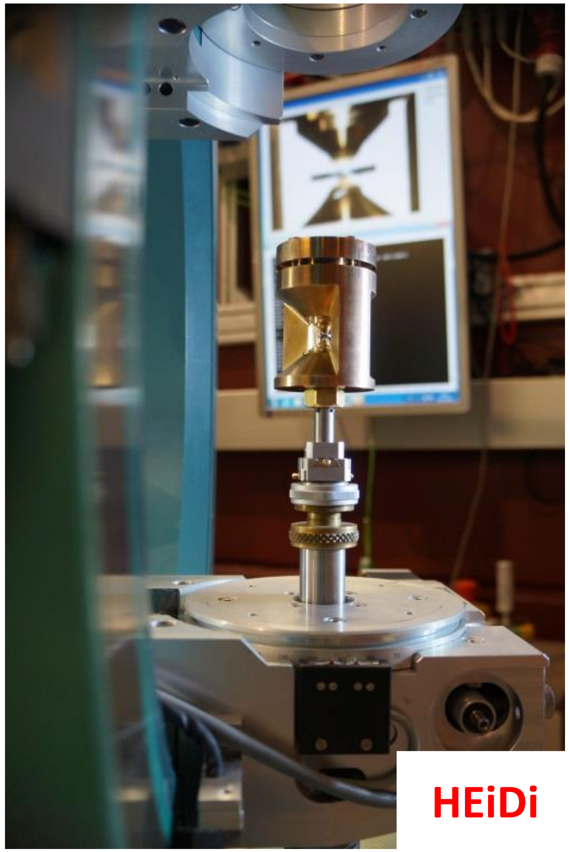
Spallation facilities

DB – diffracted beam
IB – incident beam

Panoramic diamond anvil cell





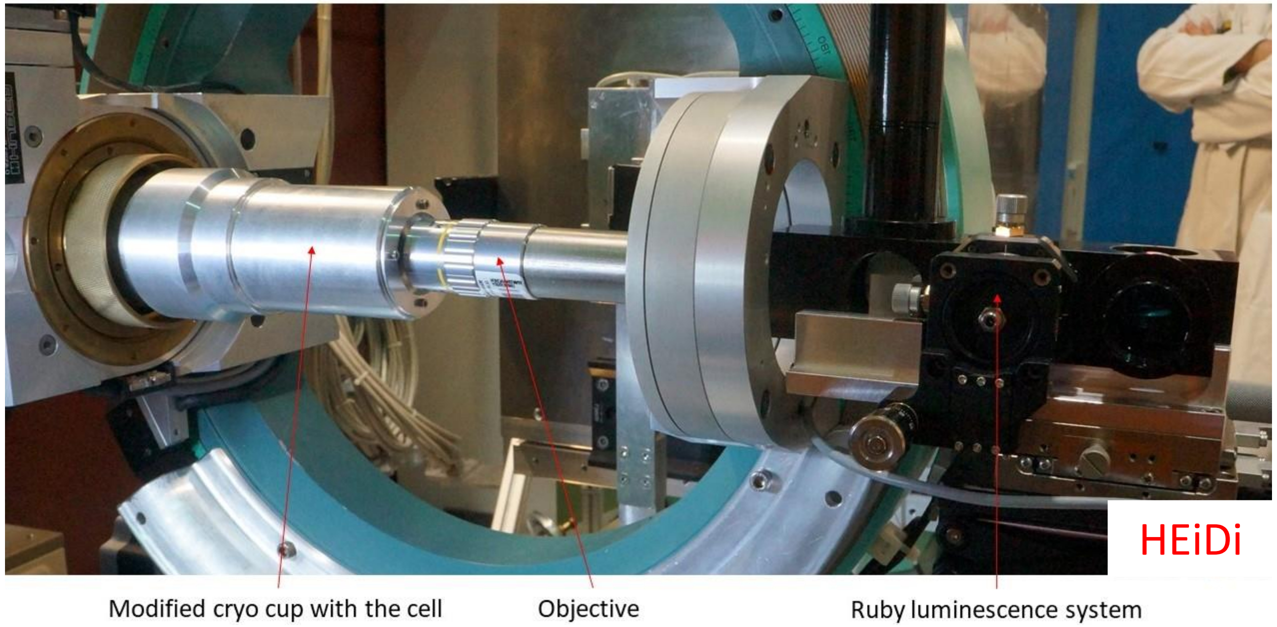


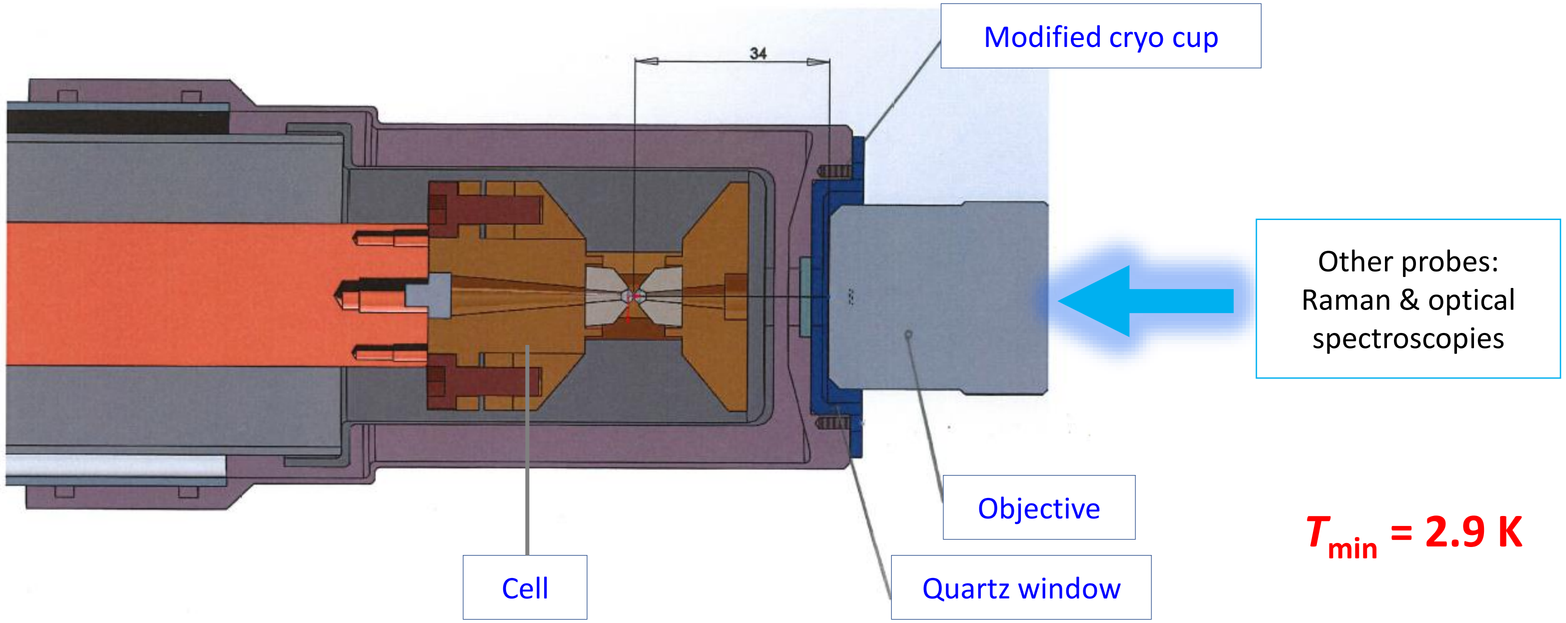
- Two horizontal openings 135°
- Vertical openings 80°
- Berylco-25 (CuBe)
- Conical diamonds (type Ia, 30° aperture)
- 40-50 mm in diameter
- 60-75 mm in height
- Fitted to closed cycle cryostats
- Optionally gas-membrane driven
- Optical access to the sample
- Optimized software for the data collection strategy (shadowing, etc.) by Martin Meven

A. Grzechnik, M. Meven, K. Friese
J. Appl. Cryst. **51**, 351 (2018)

Panoramic cell in the cryostats at HEiDi and POLI

A. Eich *et al.*
Mater. Res. Expr. **6**, 096118 (2019)

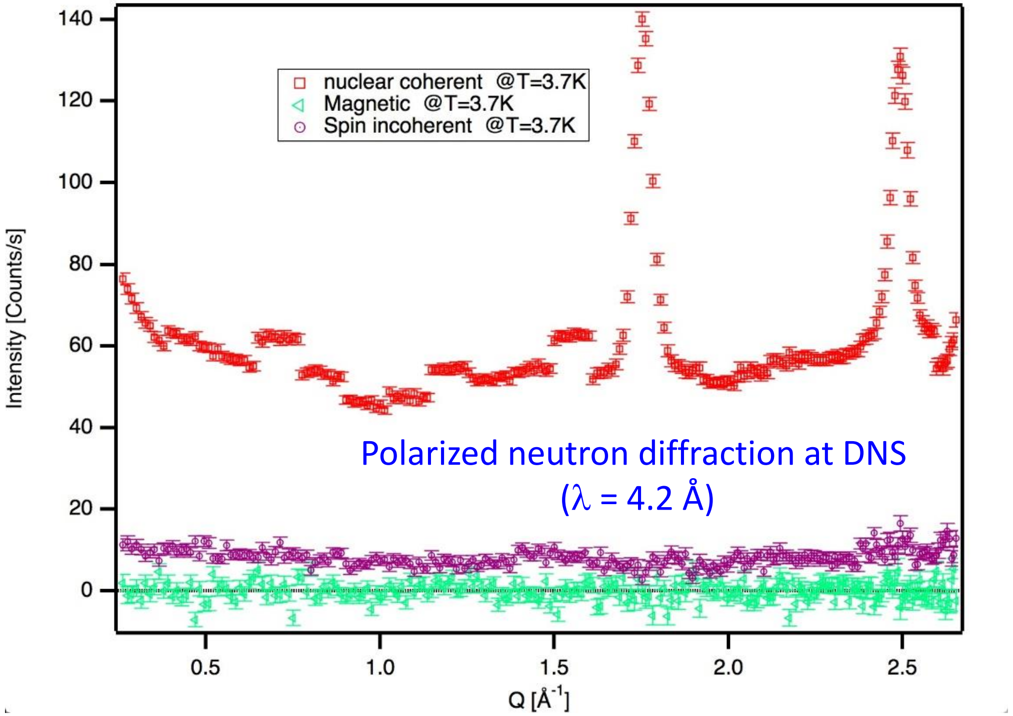


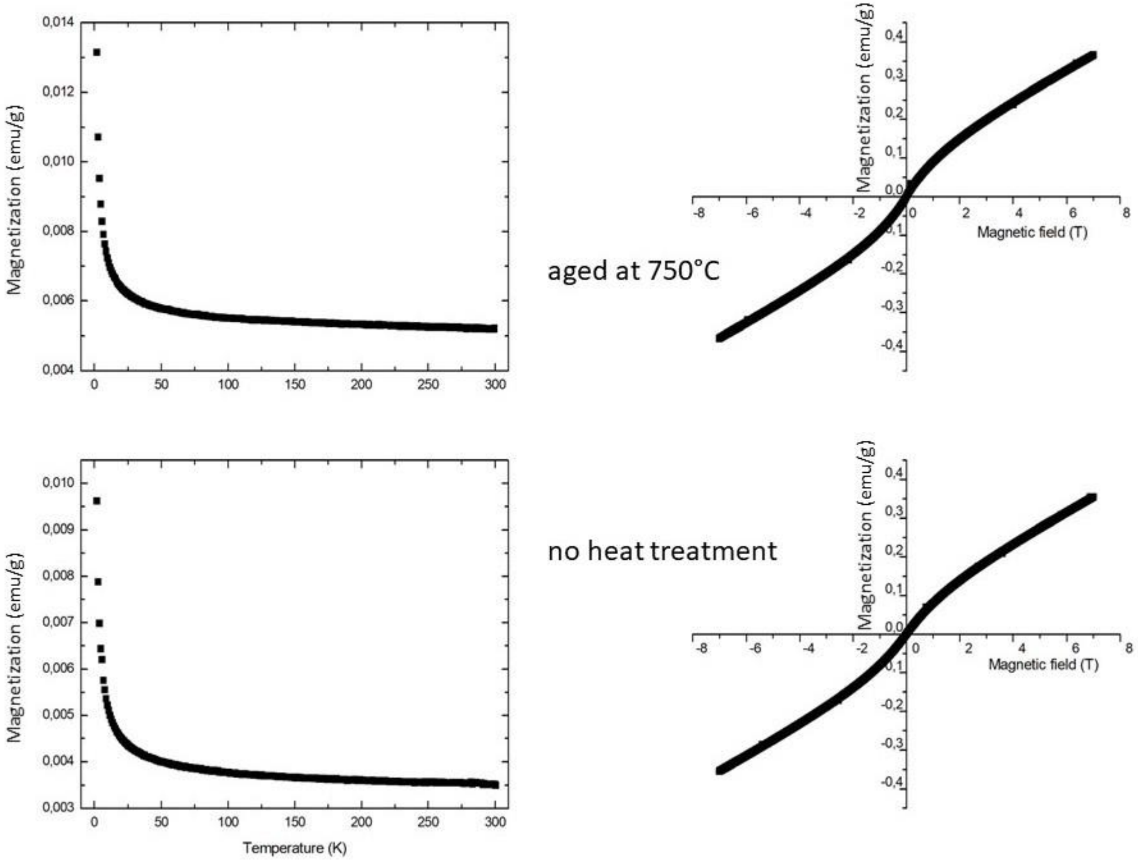


T_{min} = 2.9 K

NiCrAl alloy for neutron scattering at high pressures (σ_y < 2.2 GPa)

57.0 wt.% Ni, 40.0 wt.% Cr, 3.0 wt.% Al





Yao Cheng, M.Sc. Thesis (2018)
Institute of Crystallography, RWTH Aachen University

Cheng *et al.*, *Materials Science and Technology*
DOI: 10.1080/02670836.2019.1578077 (2019)

Overview of the panoramic cells at MLZ

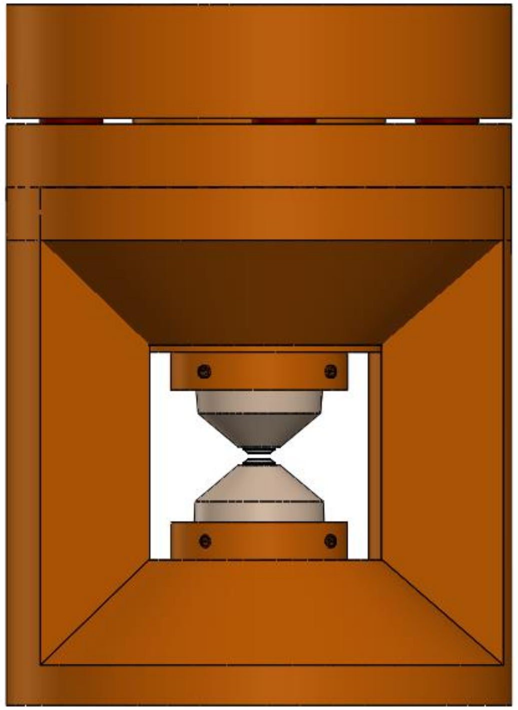
CuBe (σ_y= 1.2 GPa):

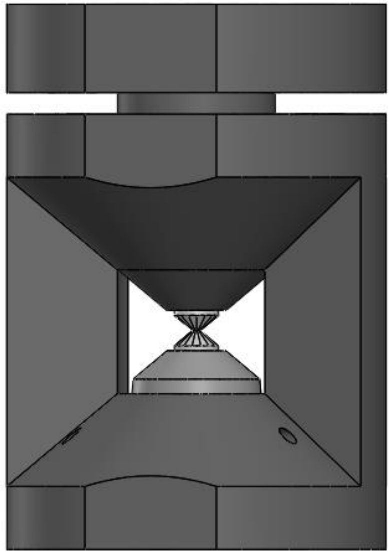
40-50 mm in diameter

Horizontal opening 135°

Vertical opening 80°

Culets up to 2 mm





NiCrAl (σ_y < 2.2 GPa):

30 mm in diameter

Horizontal opening 150°

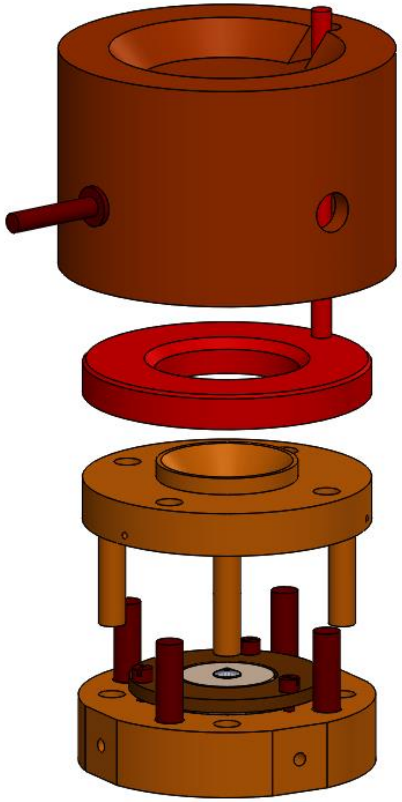
Vertical opening 80°

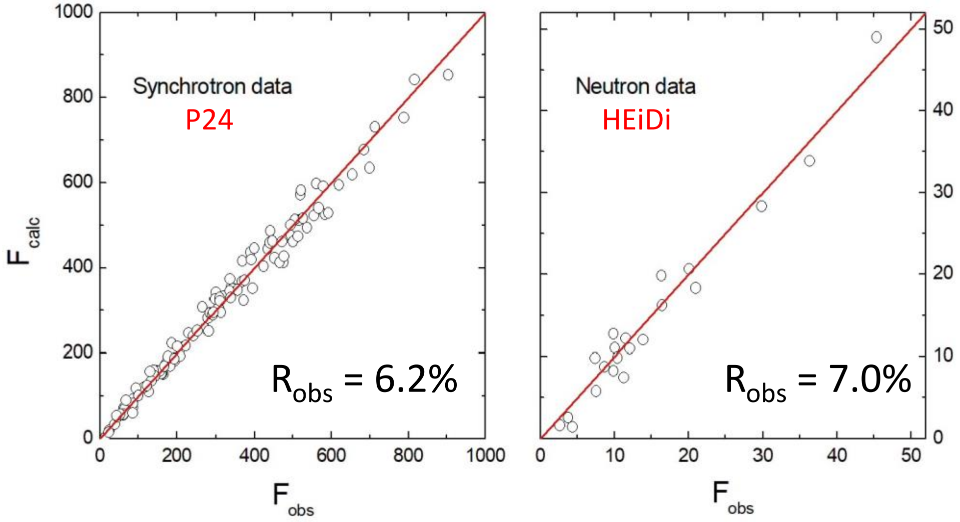
Culets up to 2 mm

Membrane diamond anvil cell
*mem*DAC: 4θ = 80°

The transmission geometry

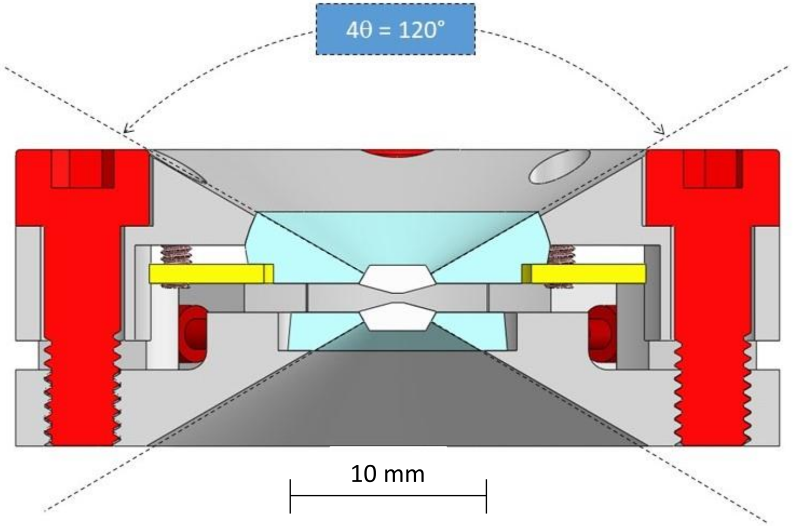
- Cell dimensions:
  44 mm in diameter, 25 mm in height
- Membrane cup:
  49 mm in diameter, 33 mm in height
- Remote pressure change using a He gas membrane
- Standard routines to search for reflections in the reciprocal space with the point detector at HEiDi
- Optimized software for the data collection strategy (shadowing, etc.) by Martin Meven

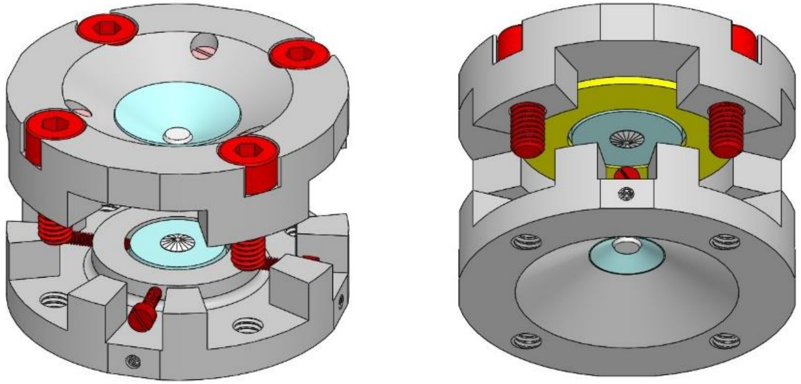




A. Grzechnik, M. Meven, C. Paulmann & K. Friese
J. Appl. Cryst. **53**, 9 (2020)

Yao-DAC for X-ray and neutron single-crystal diffraction





Yao Cheng, M.Sc. Thesis (2018)
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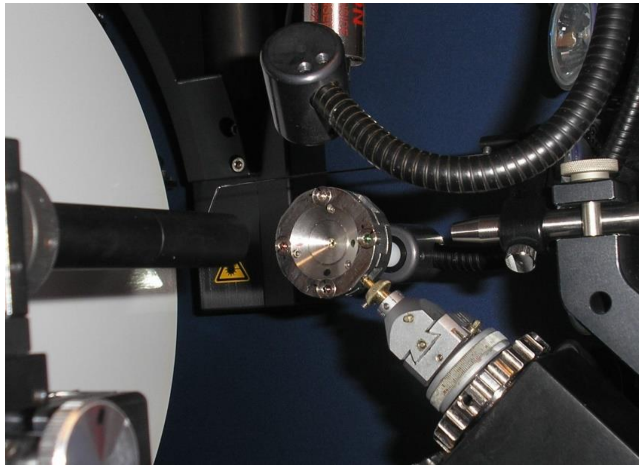
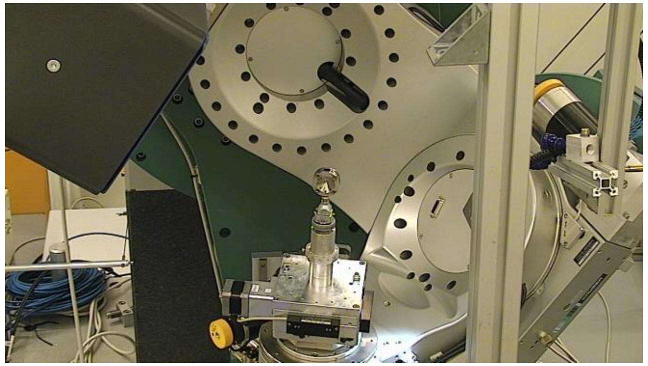


IPDS-II in Aachen (Mo-Kα)



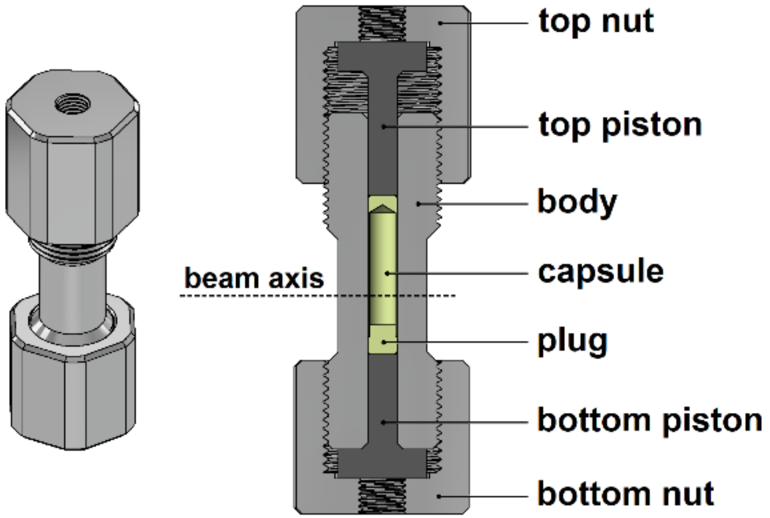
P24 (EH1) at Petra-III (Hamburg)
κ diffractometer & PILATUS detector (λ = 0.4508 Å)

Measurements:

- IPDS-II (Aachen), P24 (Petra-III), SNBL (ESRF)
- HEiDi?

Clamp Cells

Andreas Eich - doctoral thesis (2022)
Muni Kishore Babu Poli – technical support



A. Eich *et al*., *High Press. Res*. **41**, 88-96 (2020)

The cells are adapted to fit into the cryostats and high-field magnets on the beamlines :

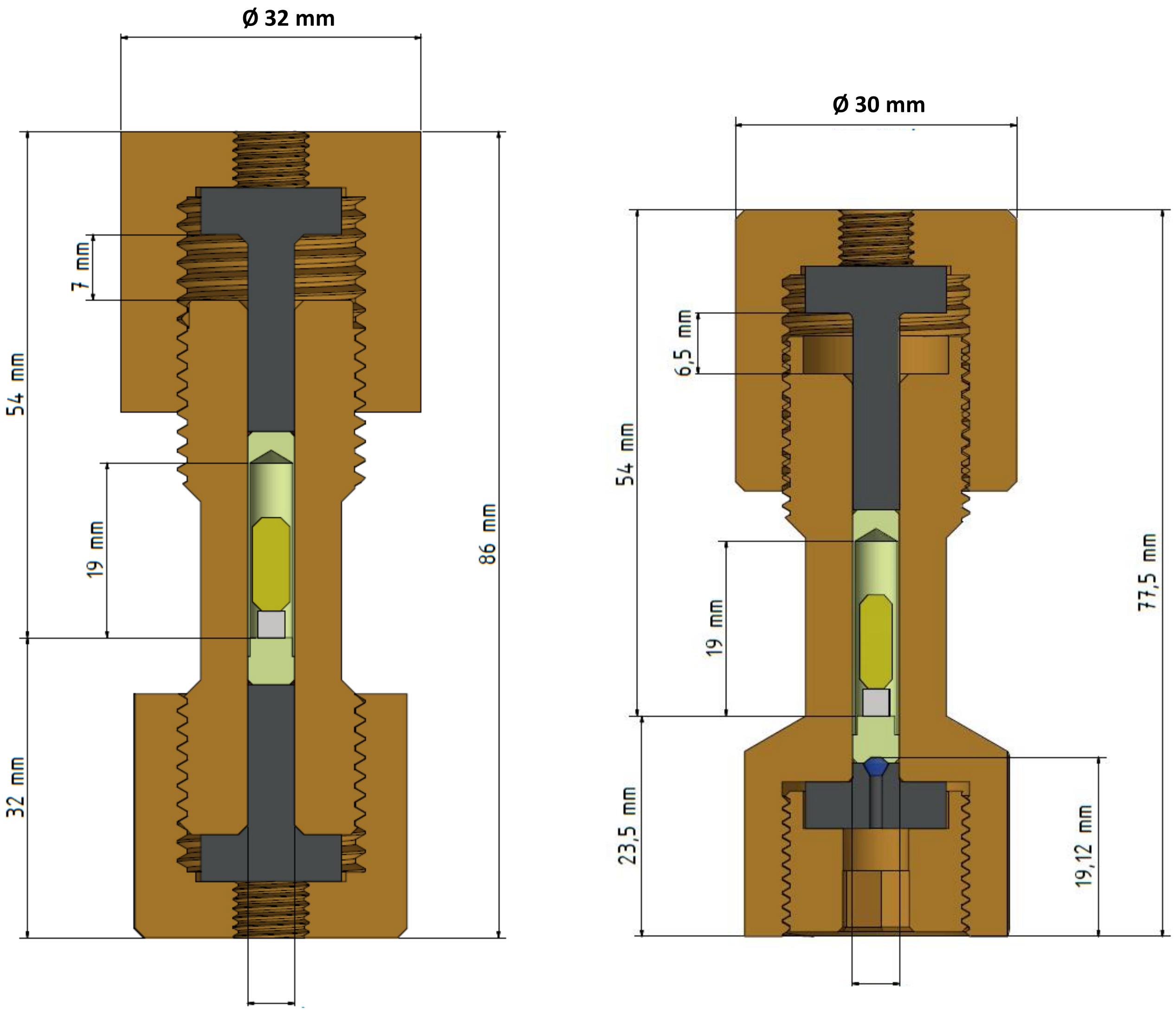
DNS: a diffuse scattering neutron *time-of-flight* spectrometer

MIRA: a cold three-axes spectrometer with polarization analysis

POLI: a double focusing polarized hot neutron diffractometer

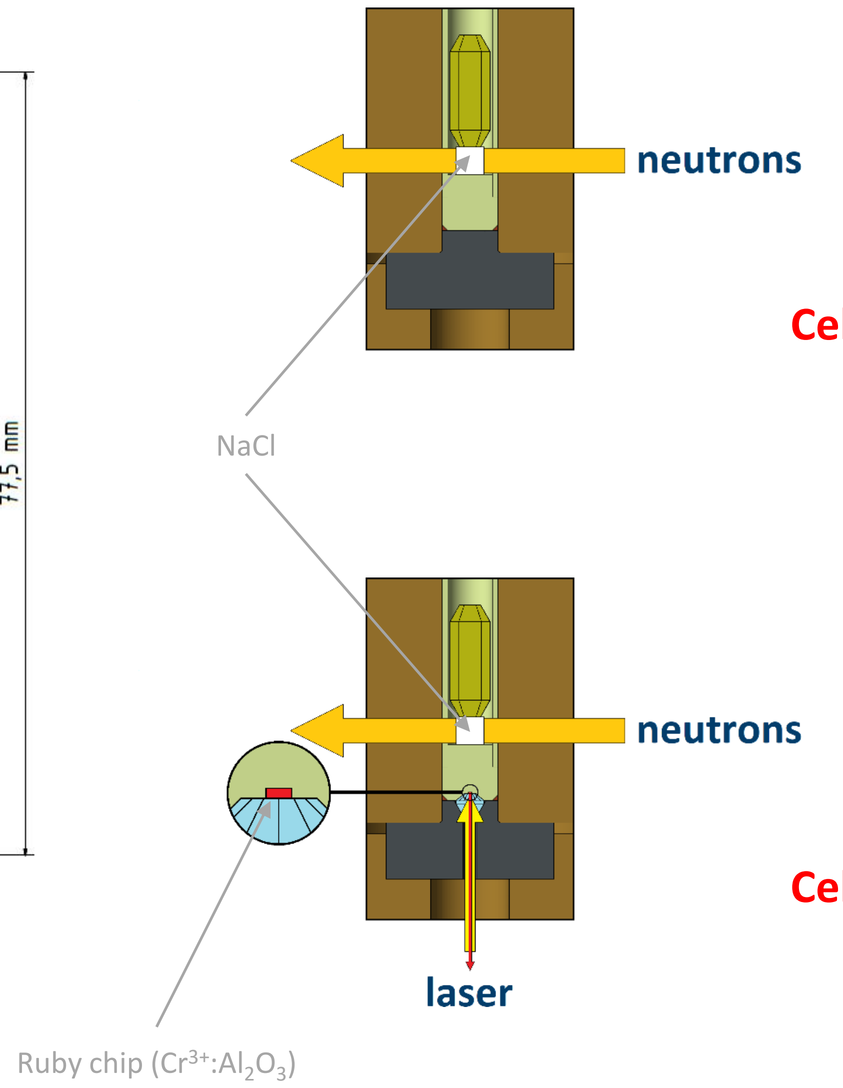
HEiDi: a single-crystal diffractometer on a hot source

Monobloc cells

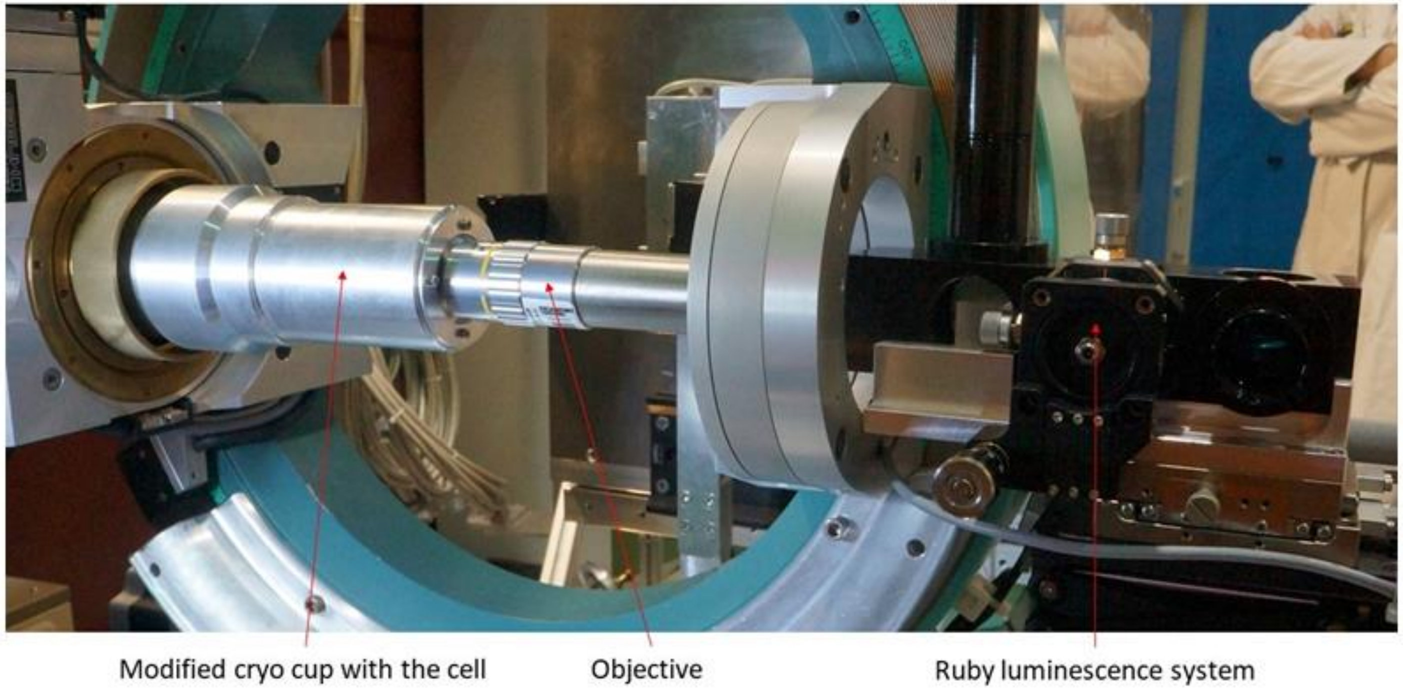


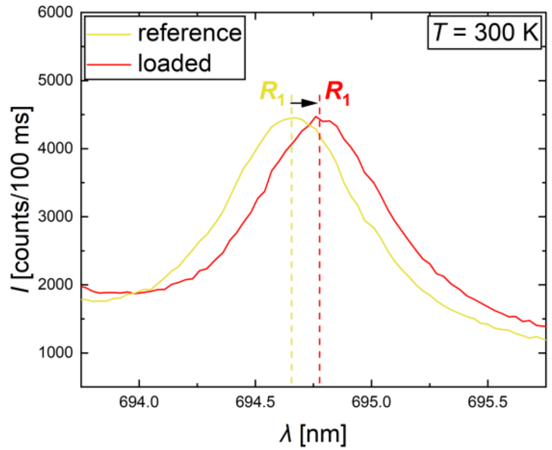
Cell M1

Cell M2

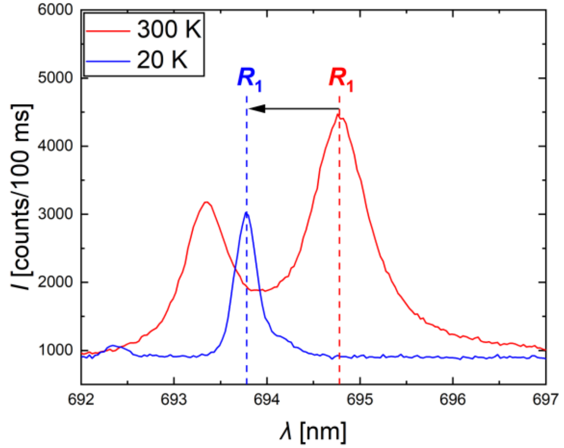


Ruby luminescence for pressure calibration at HEiDi

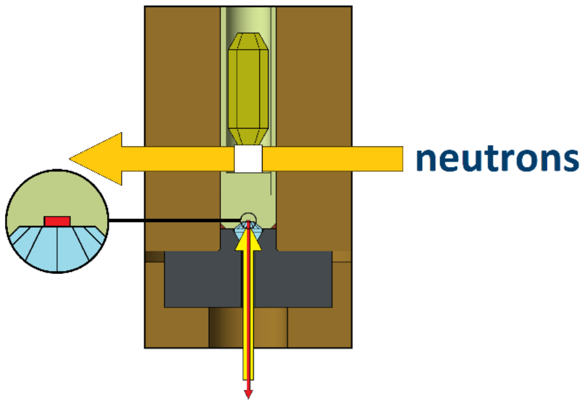




ruby R_1 shift with pressure

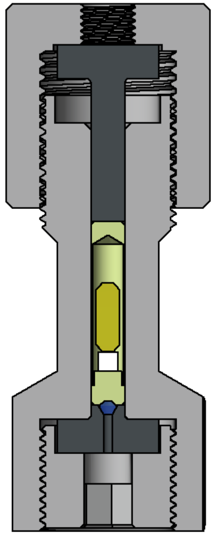


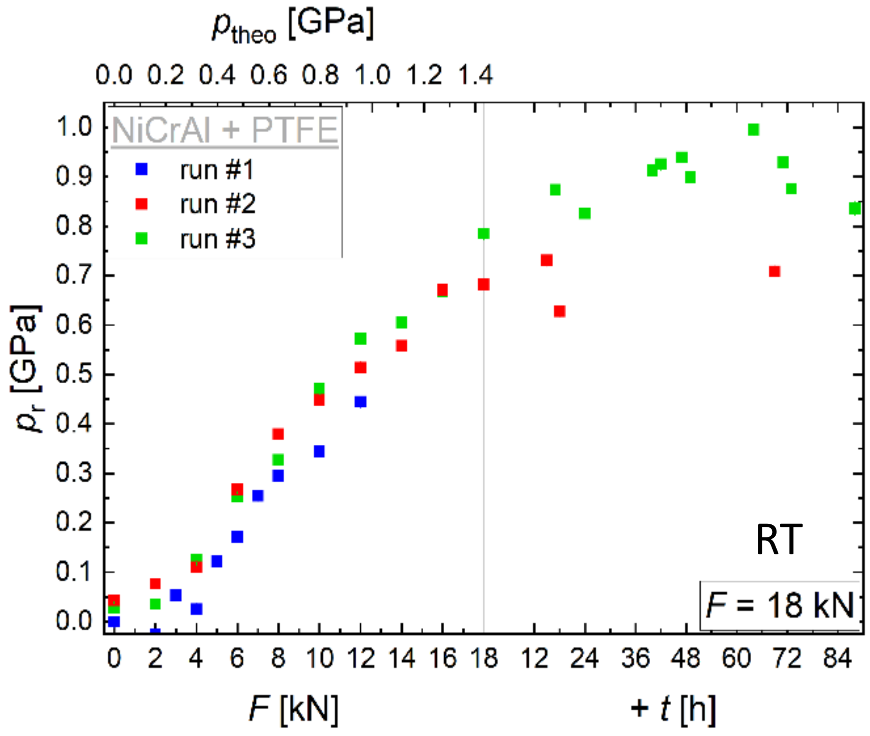
ruby R_1 shift with temperature

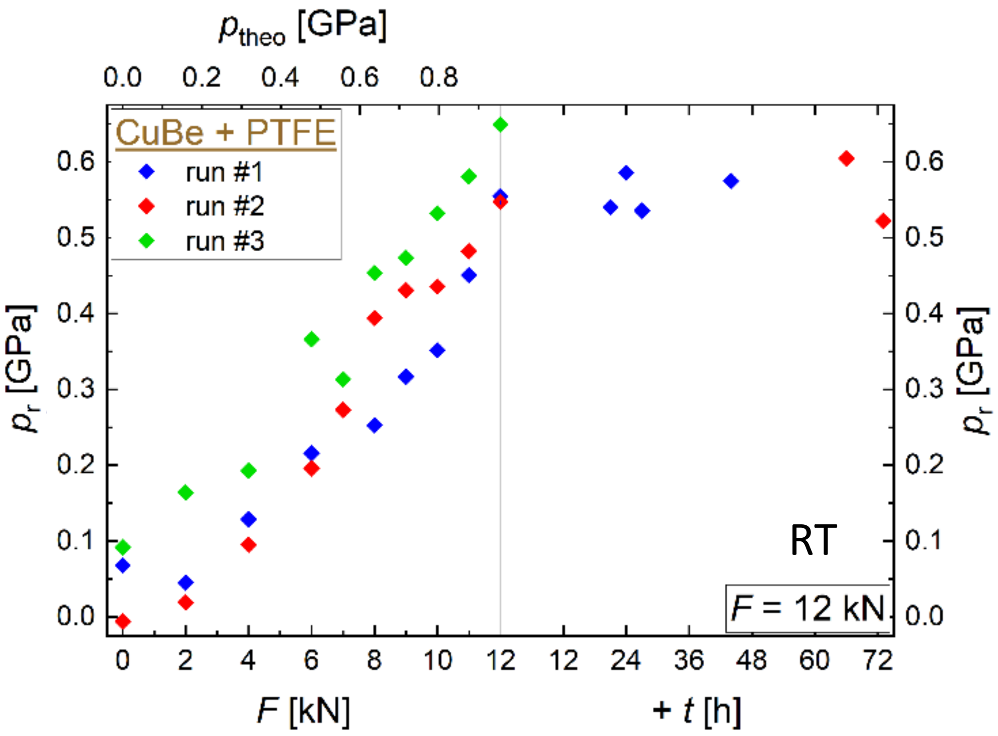


Pressure calibration of the cells M2 with ruby luminescence

Cell M2
NiCrAl and CuBe

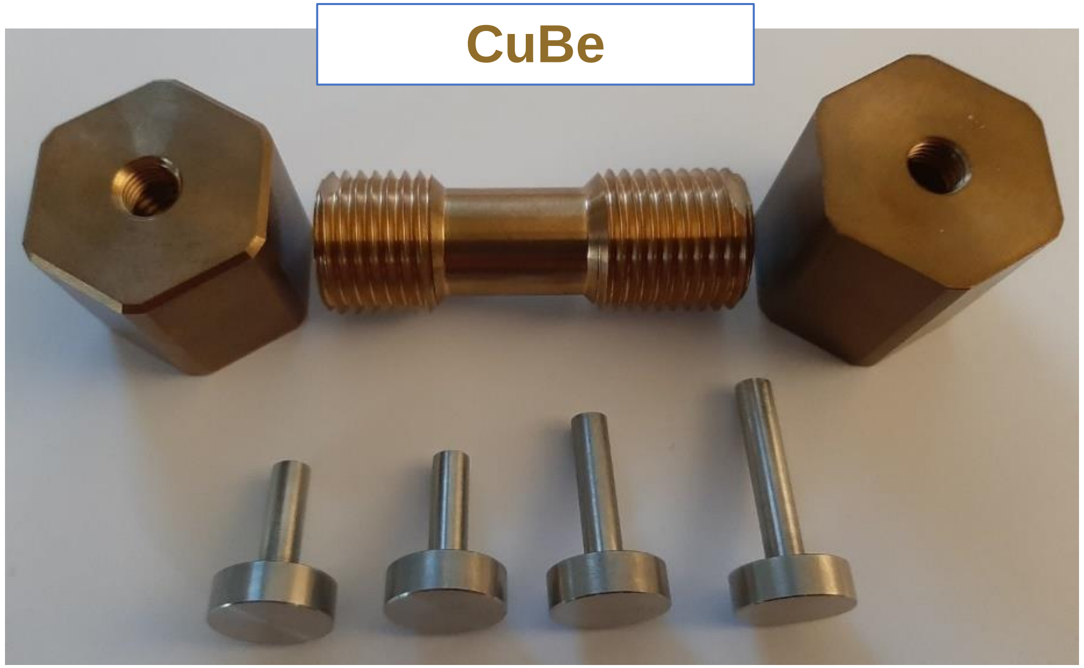


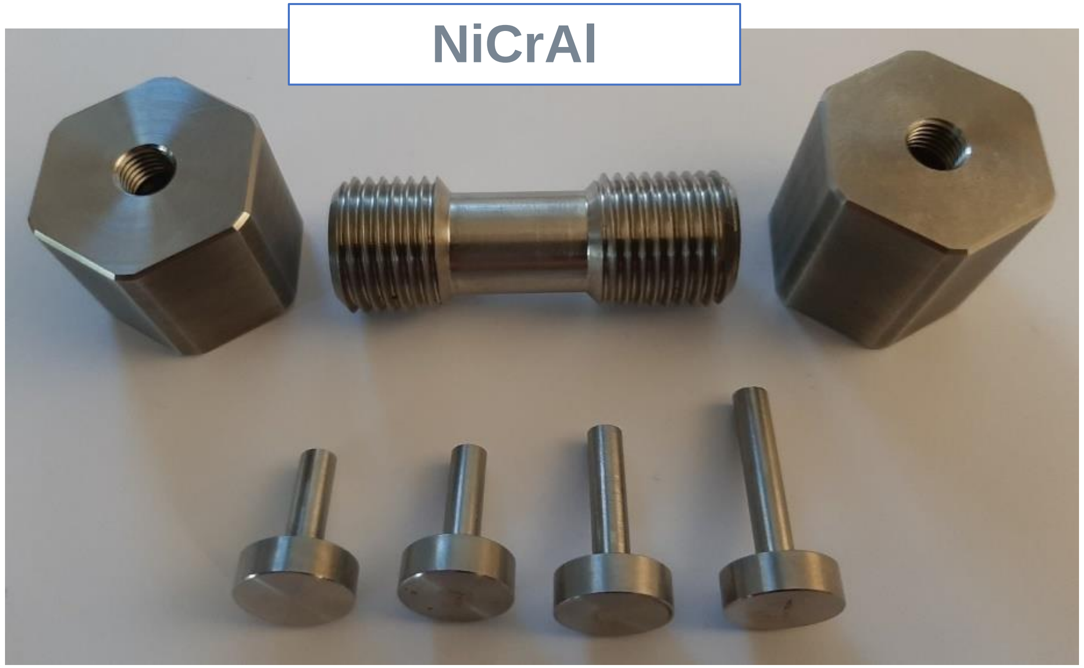




Seats & pistons: NiCrAl or Ni-WC

Monobloc cells





Diamond in a seat

Thrust pistons

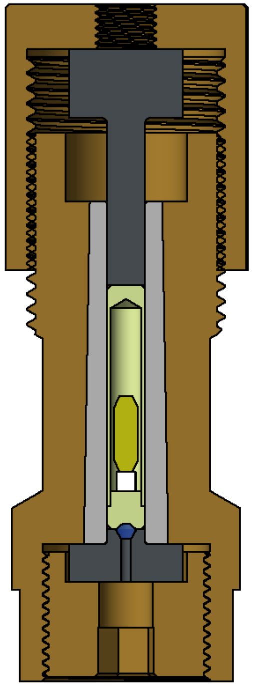
Extrusion rings:
Cu or soft CuBe

Sample capsules:
PTFE, AlMg5, or Pb

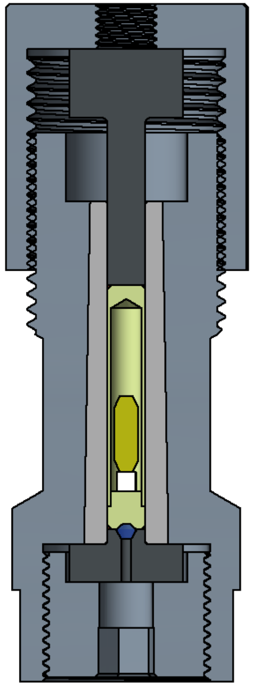
Diamond in a seat

Fretted cells F1 for pressures above 2 GPa

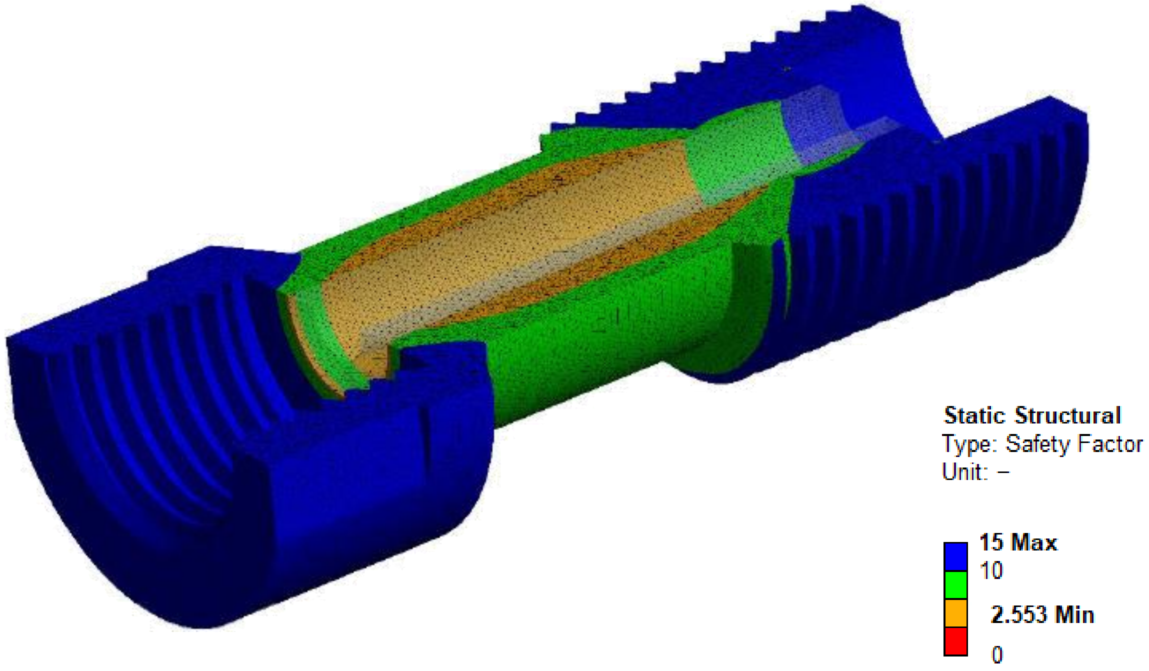
Andreas Eich - design
Muni Kishore Babu Poli – simulation



NiCrAl / CuBe
2.5 GPa

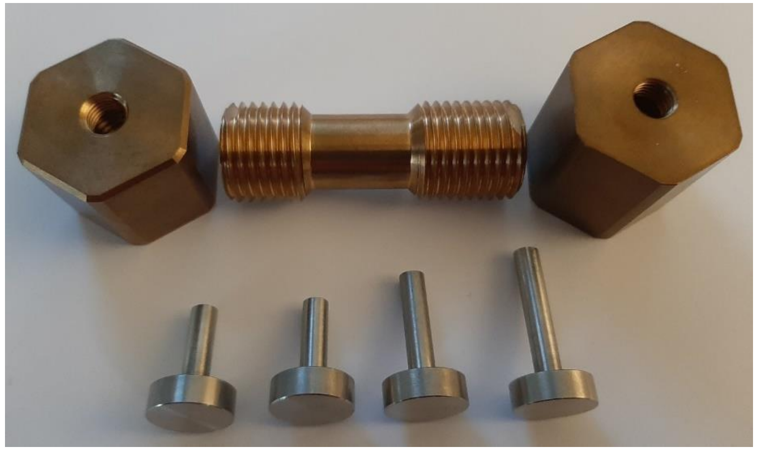


NiCrAl / TiZr
2.0 GPa



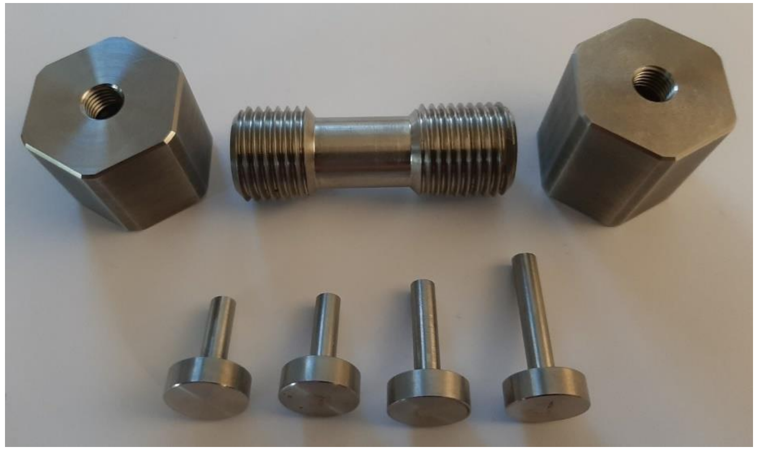
TiZr: σ_y = 0.7 GPa
CuBe: σ_y = 1.2 GPa
NiCrAl: σ_y ≈ 2.0 GPa

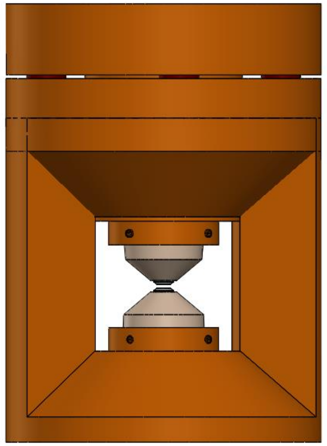
Neutron transmission

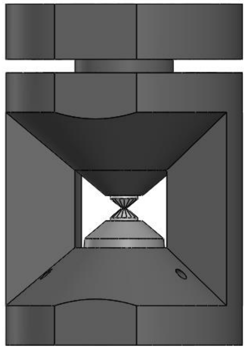


Final remarks

- A range of diamond anvil and clamp cells has been designed and produced
- Some of them have already been used at MLZ
- Tests with neutrons are required to prove the usefulness of them all for neutron scattering

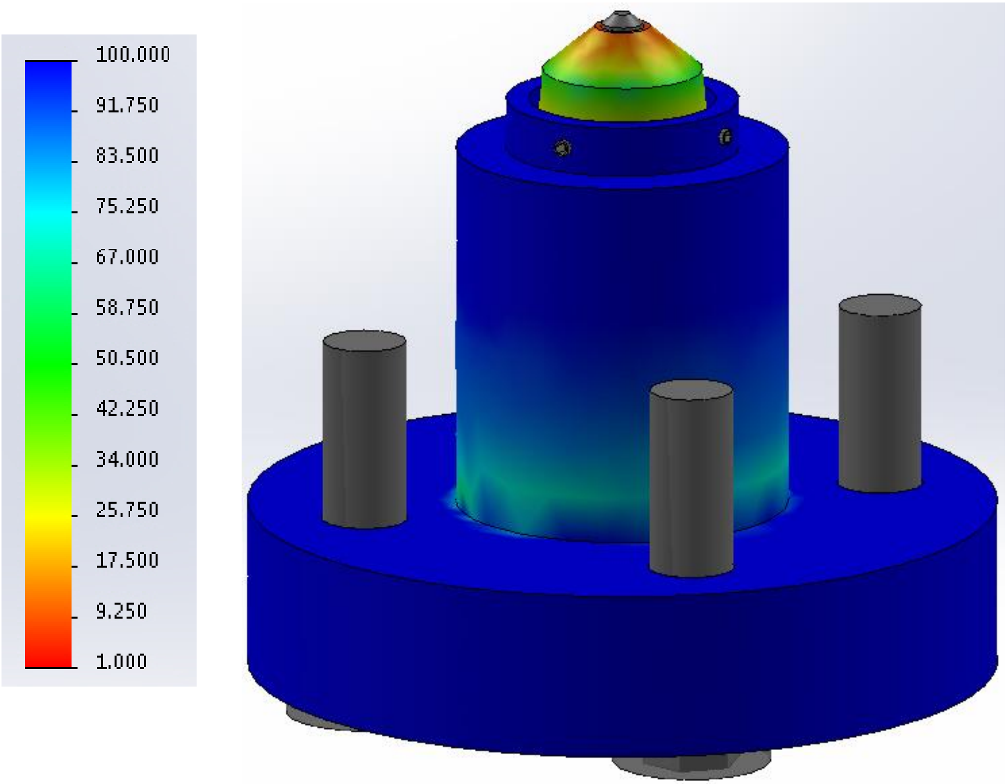




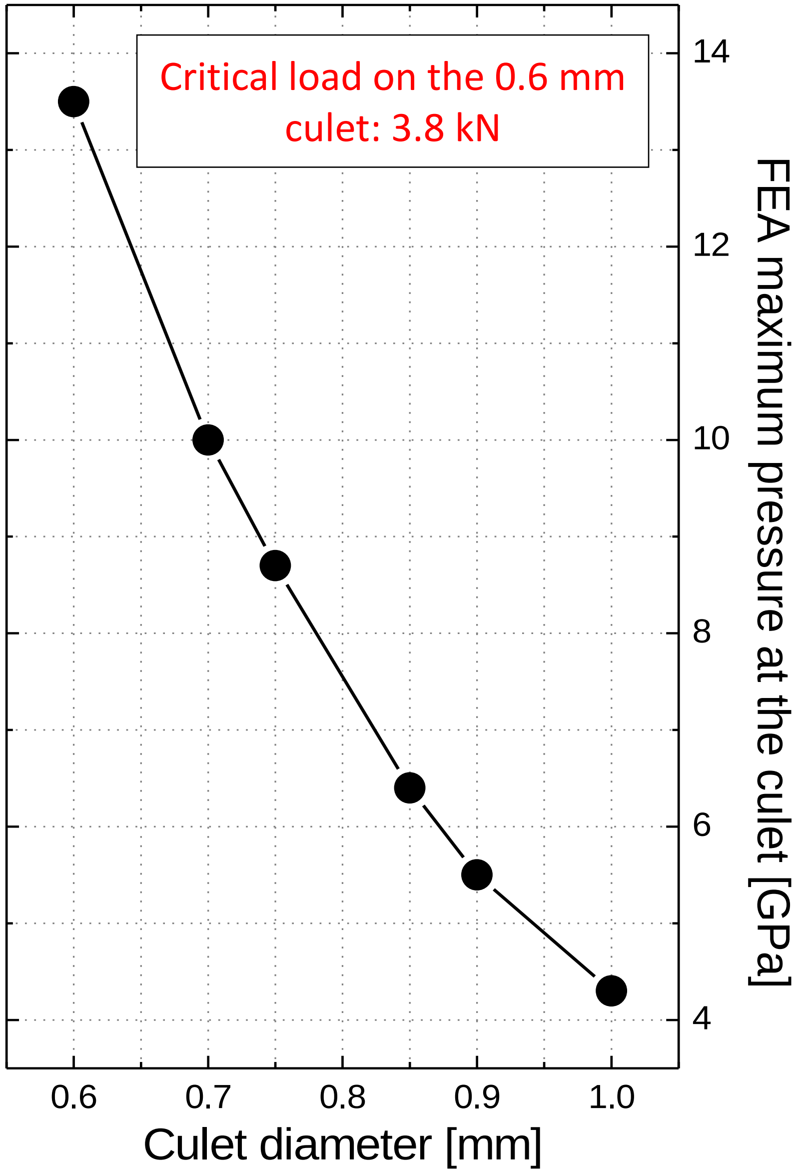


Finite element analysis (FEA): Factor-of-safety distribution (*pan*DAC)

$$\text{Factor of safety} = \frac{\text{yield stress } (\sigma_y)}{\text{working stress}}$$

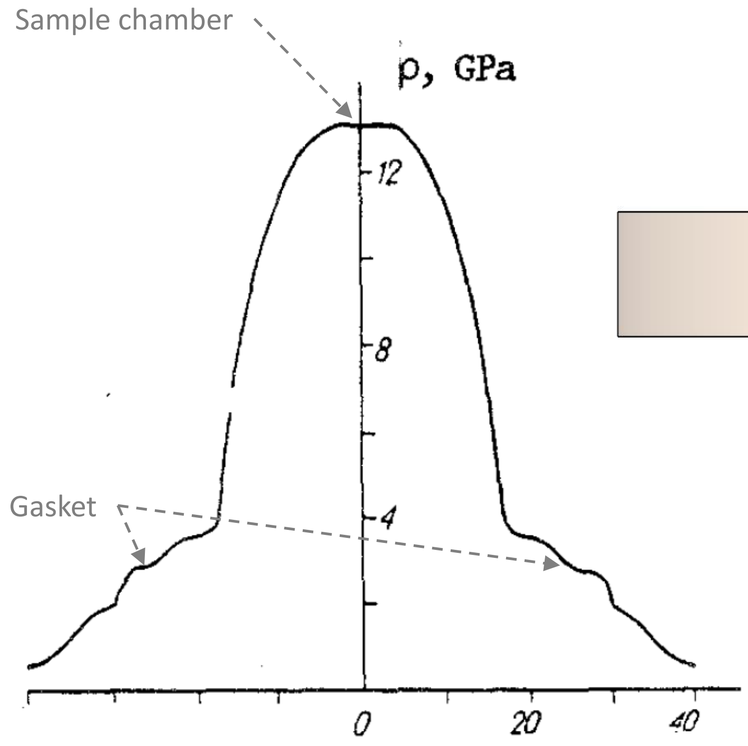


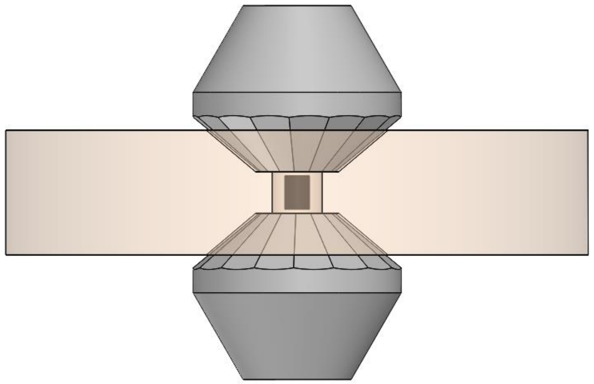
CuBe: σ_y = 1.17 GPa

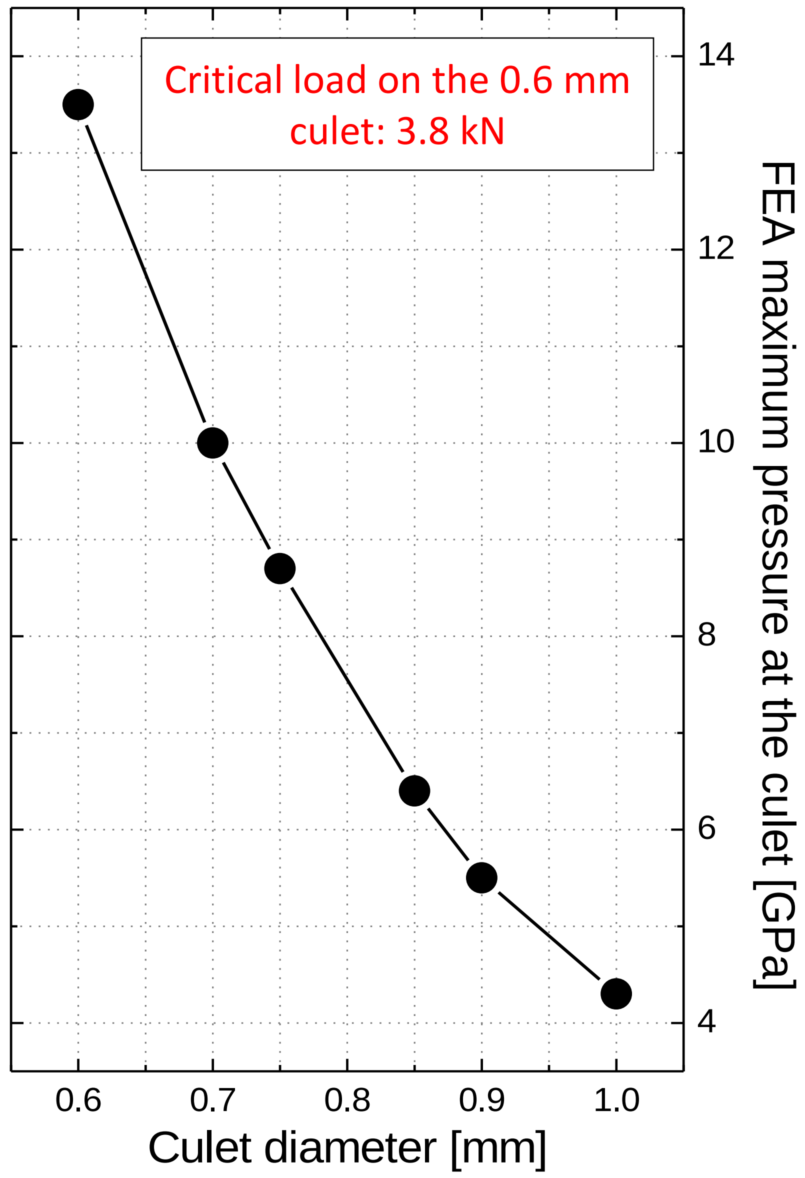


Finite element analysis (FEA): Factor-of-safety distribution (*pan*DAC)

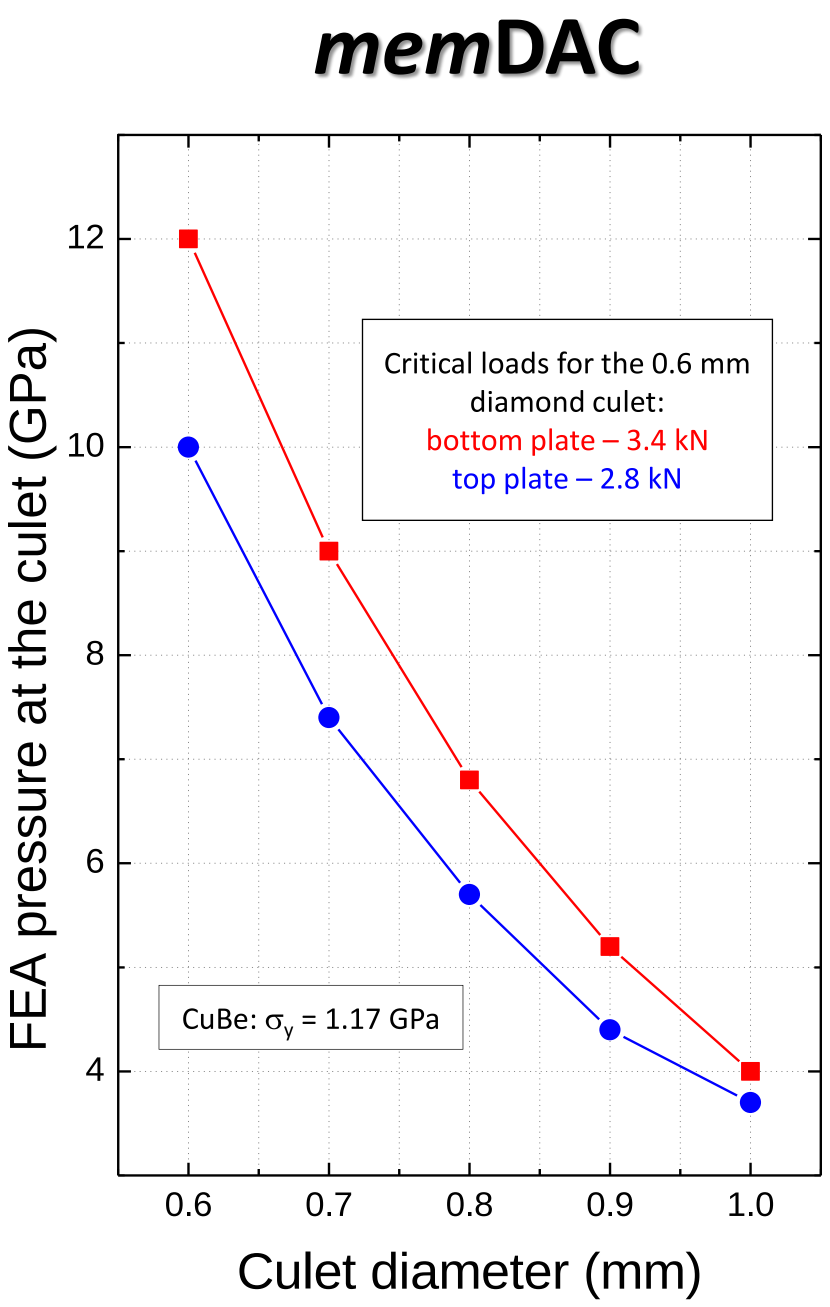
Actual pressure distribution across the culet



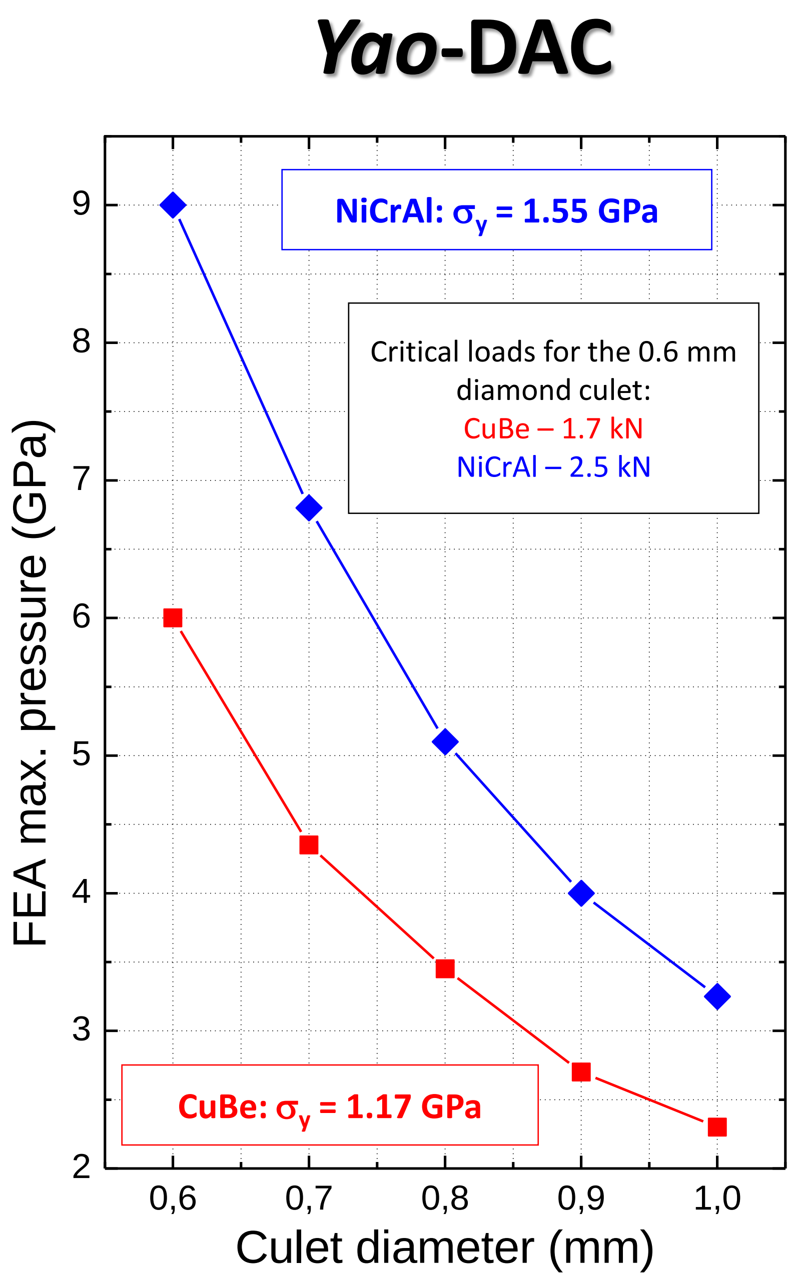




Y.A. Sadkov, L.B. Solodukhina, *J. Appl. Mech. Tech. Phys*. **33**, 903 (1992).



memDAC
FEA pressure at the culet (GPa)
Culet diameter (mm)
Critical loads for the 0.6 mm diamond culet:
bottom plate – 3.4 kN
top plate – 2.8 kN
CuBe: σy = 1.17 GPa



Yao-DAC
FEA max. pressure (GPa)
Culet diameter (mm)
NiCrAl: σy = 1.55 GPa
Critical loads for the 0.6 mm diamond culet:
CuBe – 1.7 kN
NiCrAl – 2.5 kN
CuBe: σy = 1.17 GPa