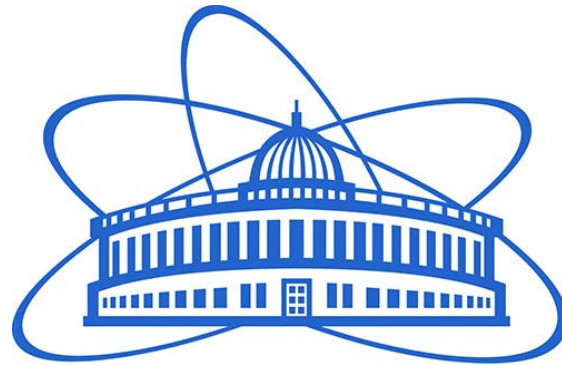


DN-6 diffractometer for studies of materials at ultrahigh pressures

**D.P. Kozlenko, S.E. Kichanov, E.V. Lukin,
A.V. Rutkauskas, B.N. Savenko,**



Joint Institute for Nuclear Research
Frank Laboratory of Neutron Physics

Neutron Diffraction at High Pressure

Synchrotron radiation source

Flux at sample position

$\sim 10^{16}-10^{18}$

$V \sim \mu\text{m}^3$

Exposition time ~ 1 sec

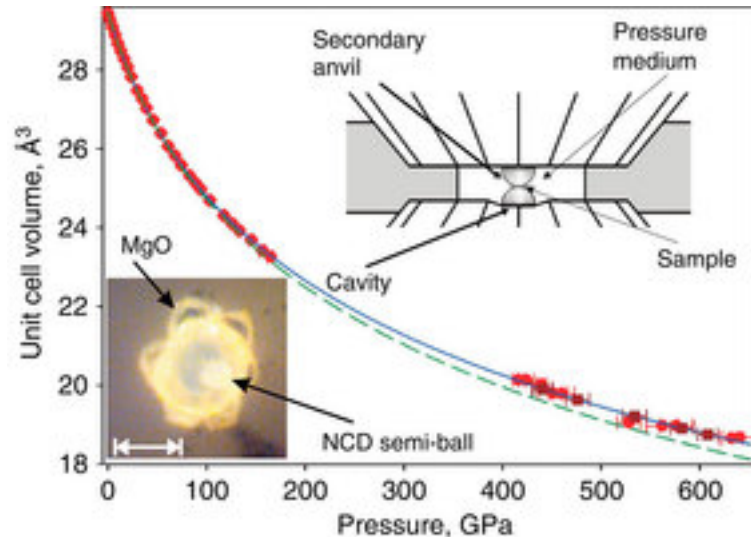
Neutron source

Flux at sample position

$\sim 10^6-10^8$

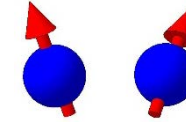
$V \sim 10-100$ mm³

Exposition time $\sim 2-10$ h



Dubrovinsky, L. et al. Implementation of micro-ball nanodiamond anvils for high-pressure studies above 6 Mbar. Nat. Commun. 3:1163 doi: 10.1038/ncomms2160 (2012).

• Magnetic structure



Magnetic phase transitions or spin-reorientation transitions

• Compounds contained light elements: H, D, Li, O ...



Structural phase transition in molecular crystals, in hydrides, hydrate, pressure induced ice forms and others....

• High penetrating effect: pressure cells, ovens, magnets, refrigerators...

High pressure neutron diffractometers throughout the world

Facility	Instrument	Pressure range (GPa)	d-spacing range (Å)	Flux at sample (n/s/cm ²)
ISIS RAL (UK)	Pearl	20 (WC)	0.5-3.2	2*10 ⁷
LLB (France)	G6.1	12 (SA) 43 (DA)*	3-60	2*10 ⁷
ILL (France)	D20	10 (WC)	0.8-60	10 ⁸
LANSCE (USA)	HiPPO	8 (WC)	0.12-22	10 ⁷
J-PARC (Japan)	PLANET	20 (WC) 40 (WC)*	0.2-4.1	10 ⁸
SNS (USA)	SNAP	25 (WC) 94(DA)*	0.5-3.65	10 ⁸
IR-8	DISK	8 (SA)	0.8-60	1.6*10 ⁶

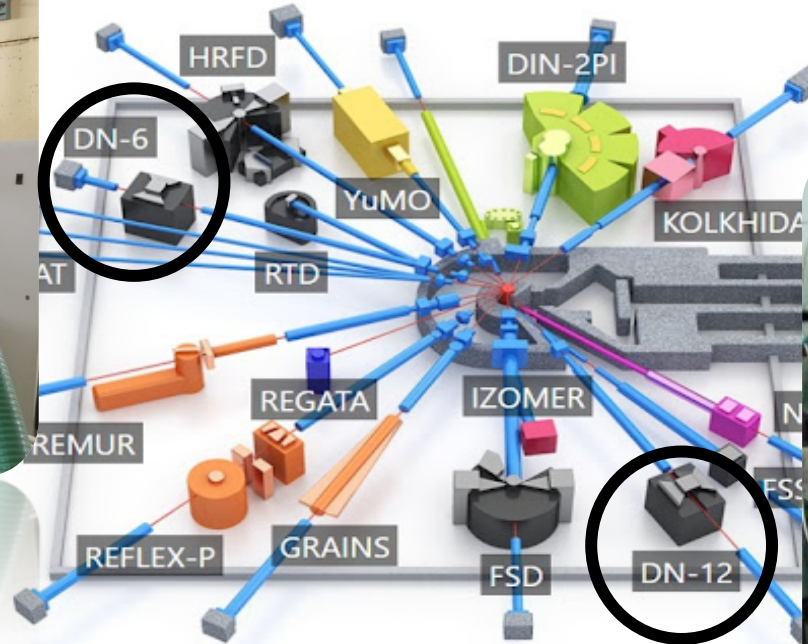
*-not routine

WC (Tungsten carbide anvils),
SA (Sapphire anvils), DA (Diamond anvils)

High-pressure DN-6 and DN-12 diffractometers.



DN-6 diffractometer on 6 beamline



**High-flux pulsed reactor
IBR-2**

**Thermal neutron flux in
pulse $5 \cdot 10^{15} \text{ n/s/cm}^2$**



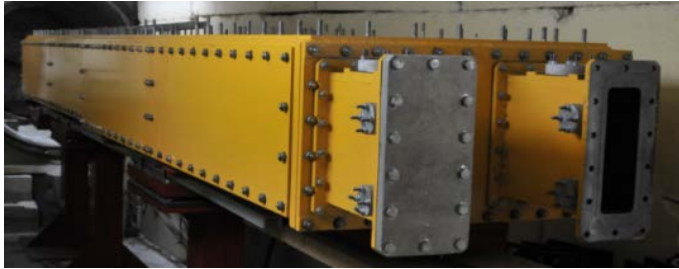
DN-12 diffractometer on 12 beamline

DN-6 and DN-12 diffractometers. Main parameters.

Parameters	DN-6	DN-12
Neutron flux density at sample position	$\sim 3.5 \times 10^7$ n/cm ² /s	$\sim 1.5 \times 10^6$ n/cm ² /s
TOF distance:	30.5 m	26.0 m
Ranges:		
wavelengths		0.8 – 10 Å
scattering angles	32° – 93°	45° – 135°
d-spacing	0.5 – 11.2 Å	0.6 – 12 Å
Resolution $\Delta d/d$ (d=2 Å)		
at $2\theta=45^\circ$	~0.04	0.030
at $2\theta=90^\circ$	0.025	0.022
at $2\theta=135^\circ$		0.012
Average sample volume	0.01 mm ³	1 mm ³
Temperature range	4-320K	10-320K
Pressure range	↑ 50 GPa with diamond anvils ↑ 12 GPa with sapphire anvils	↑ 8 GPa with sapphire anvils
Exposition time per pressure point	2-20 h	12-36 h

DN-6: general information

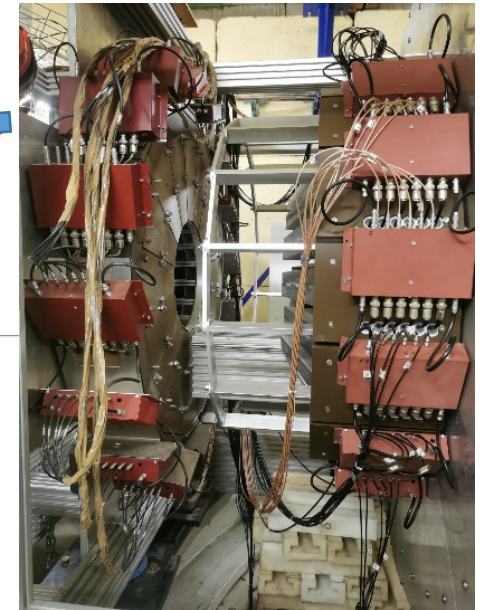
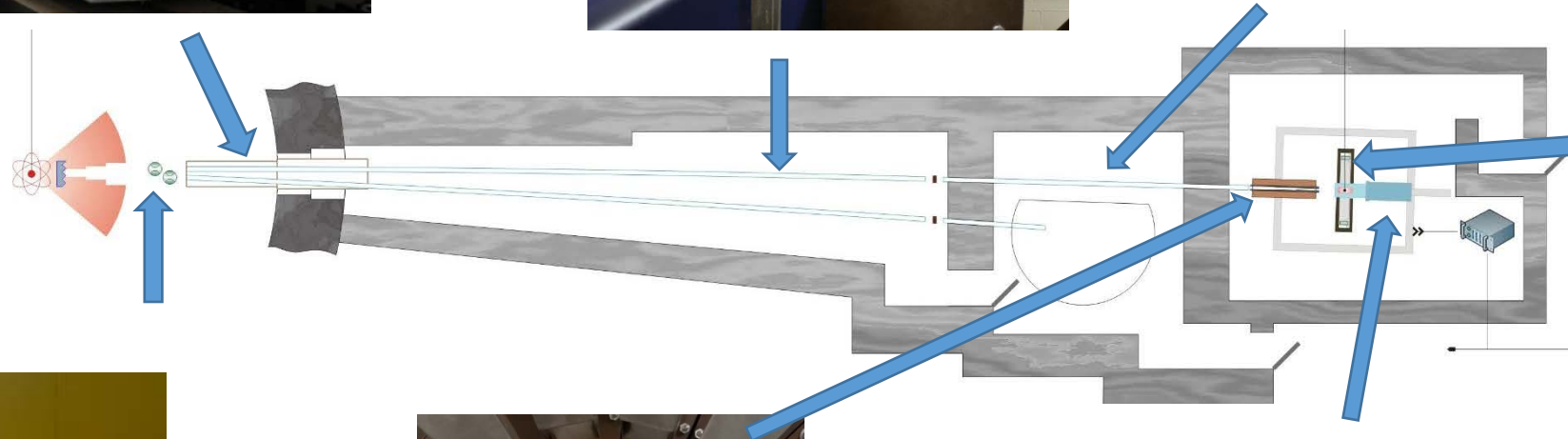
Neutron flux splitter



Curved part of neutron guide



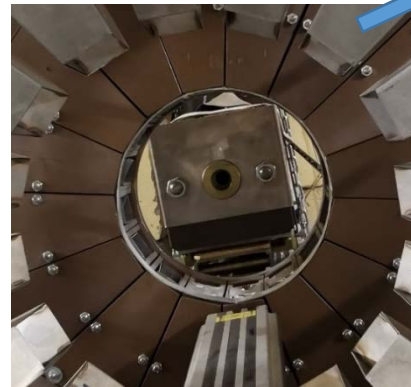
Focusing part of neutron guide



Detector system

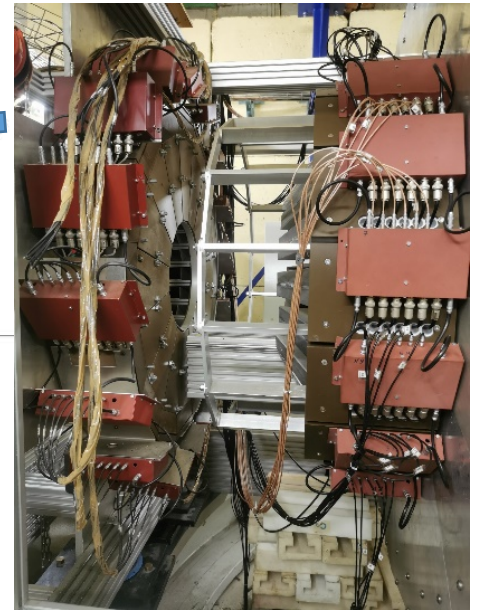


Background chopper

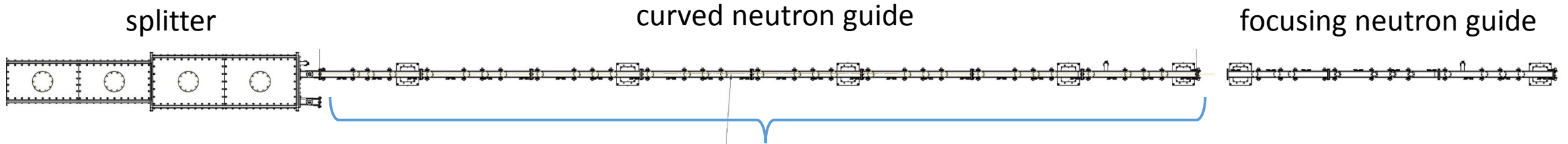


Collimator

Close circle refrigerator



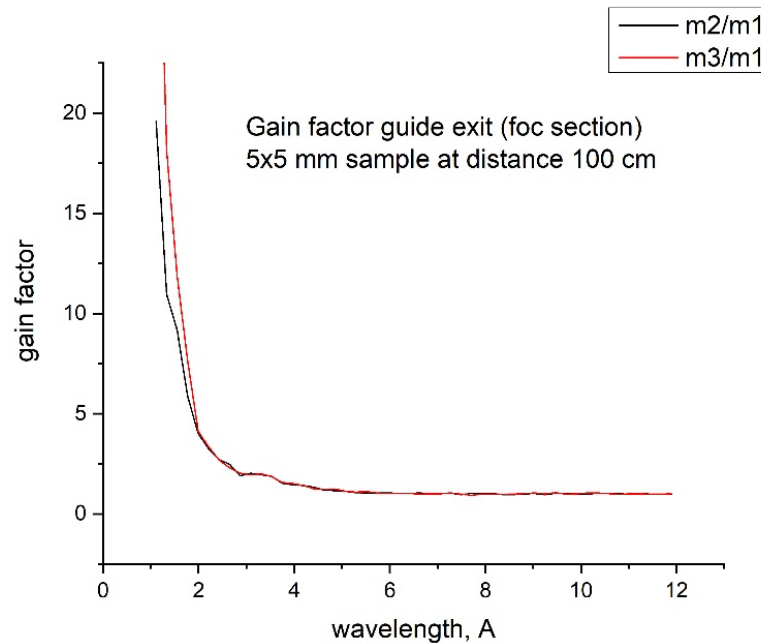
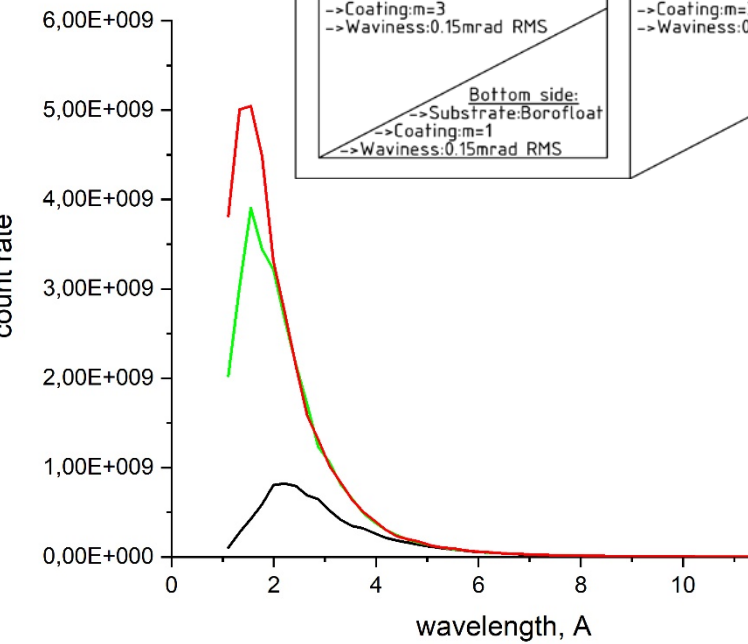
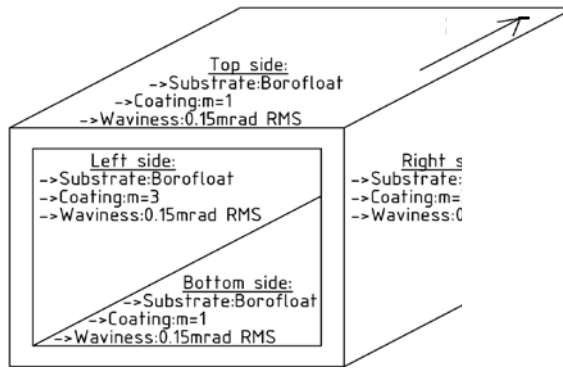
DN-6: neutron guide/curved part



Curved neutron guide

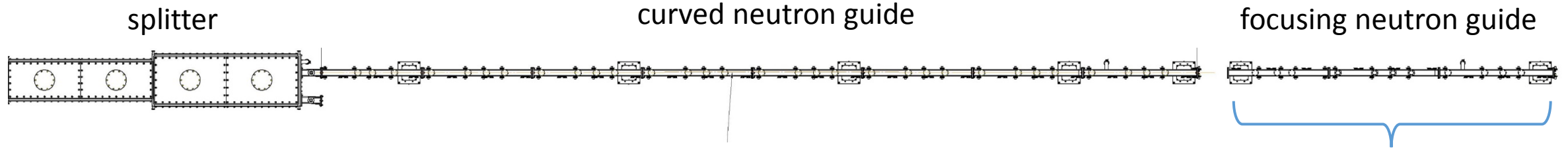
Cross section: 180.00 mm(h)x 15.00 mm(w)

- Radius of curvature: 1875 meters



— m2/m1
— m3/m1

DN-6: neutron guide/focusing part



vertical parabolic tapered neutron guide

-Profile (hor./vert.): parallel/parabolic tapered (linar appr.)

-Entrance: 180.00 mm(h)x 15.00 mm(w)

-Exit: ~60 mm (h)x 15.00 mm(w)

Reflectivity:

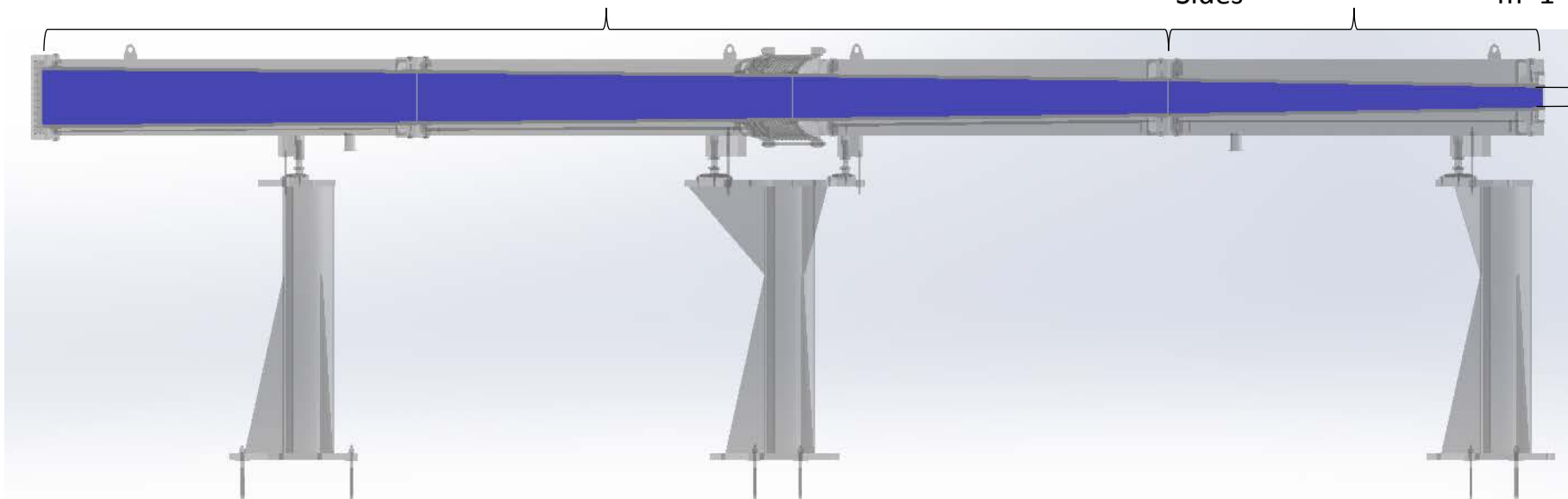
-Top/bottom $R_{ave}=92\%$
-Sides $R_{total}\sim 99,5\%$

First three units:

Coating: Ti/Ni
-Top/bottom $m=3$
-Sides $m=1$

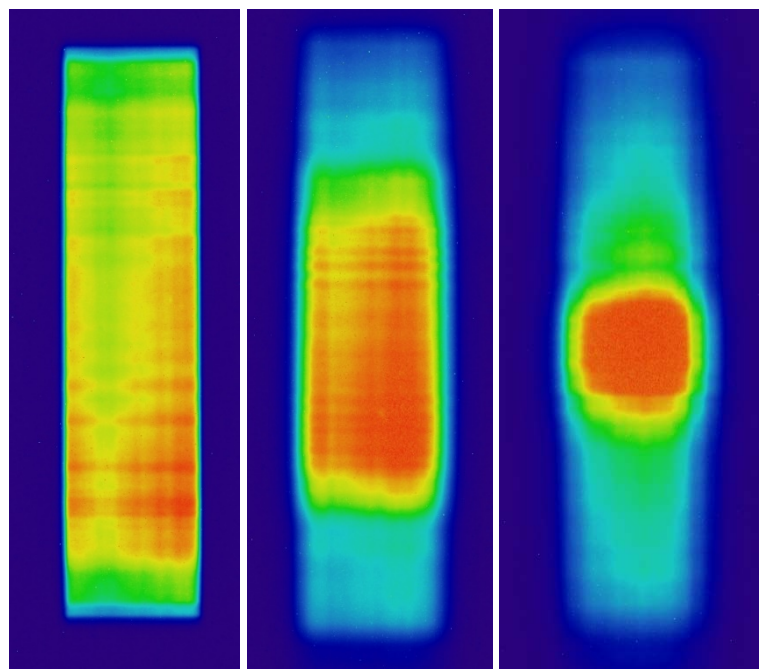
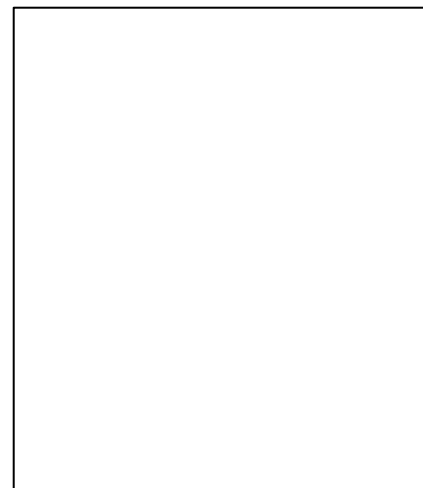
Last unit:

Coating: Ti/Ni
-Top/bottom $m=5$
-Sides $m=1$



Focus at 87 cm
downstream

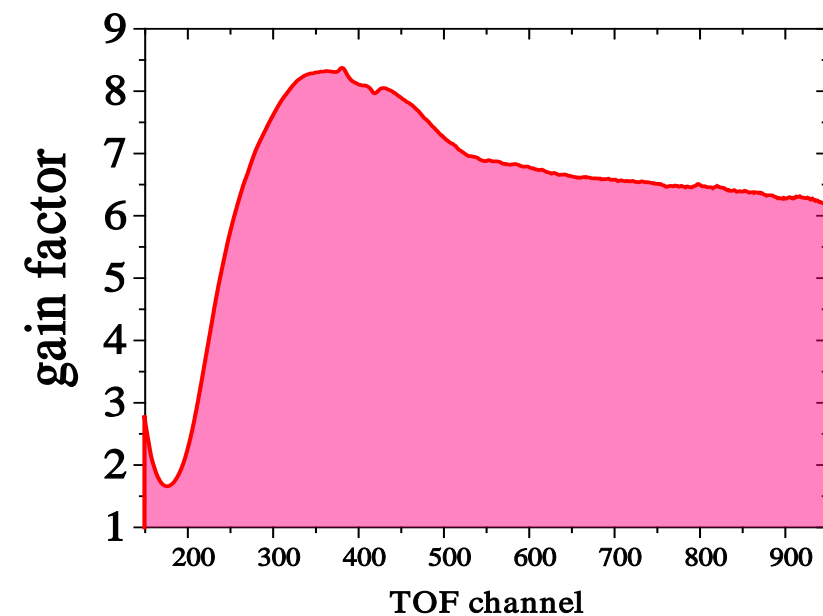
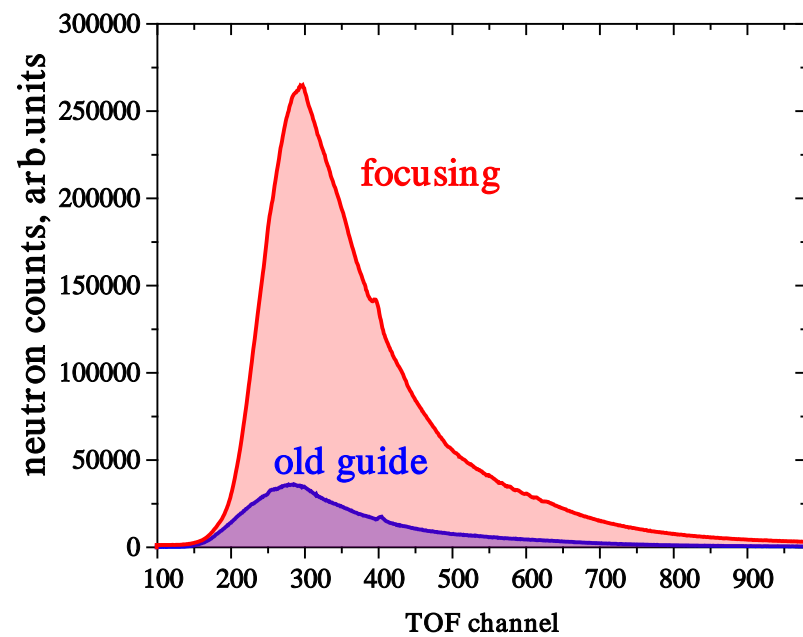
DN-6: neutron guide/focusing part



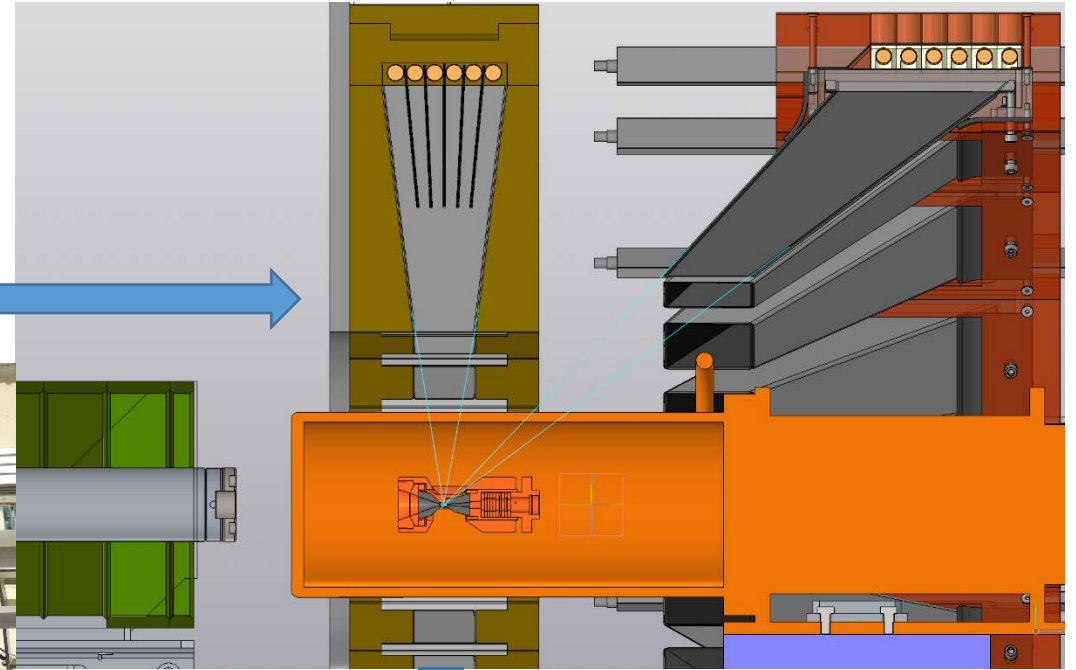
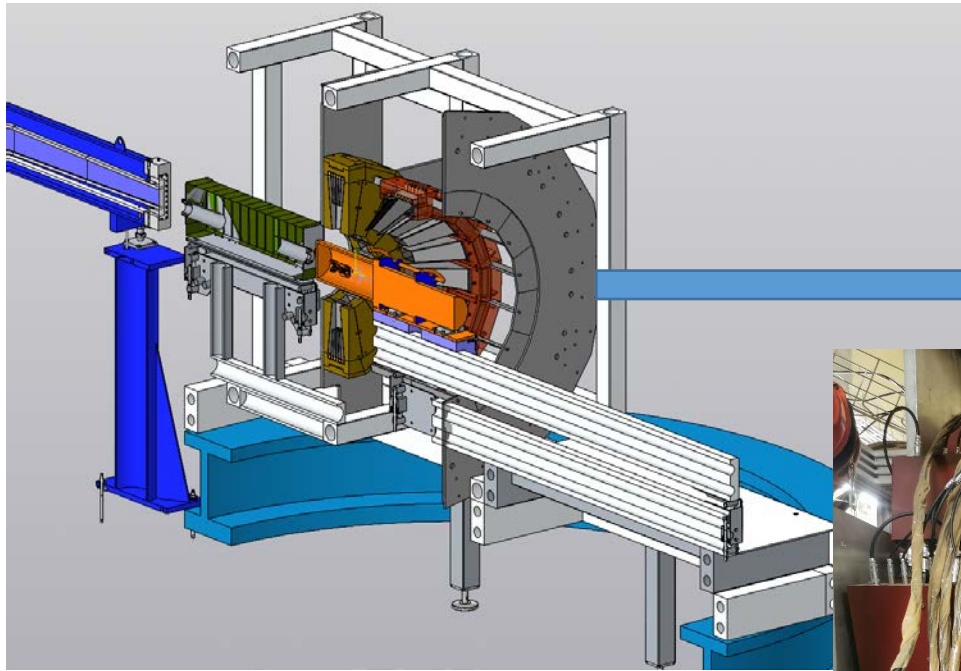
0 cm

45 cm

87 cm



DN-6: detector system/90 degrees detector ring



96 independent neutron counters group in

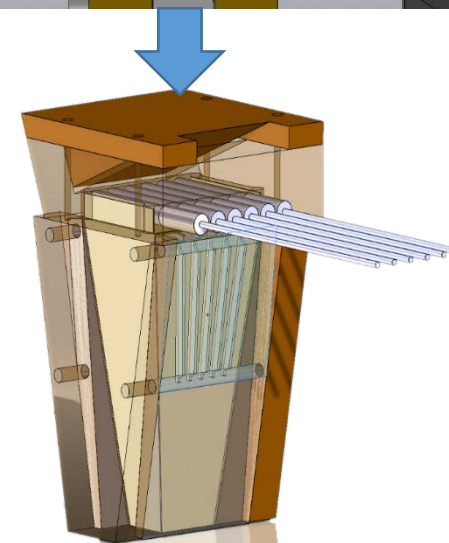
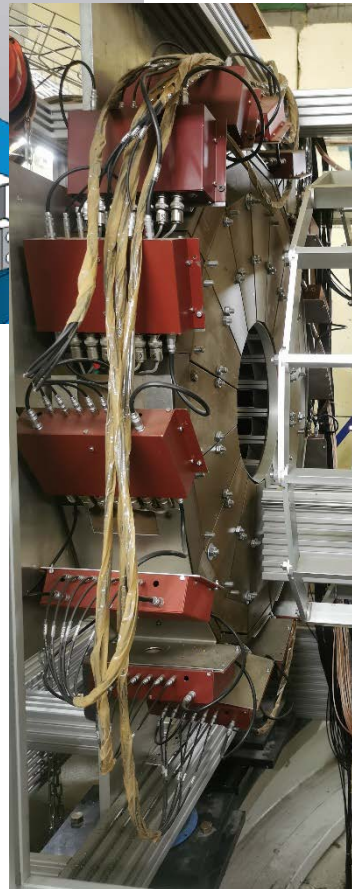
16 independent modules within

6 counters in each module

87-93 2θ deg. covering

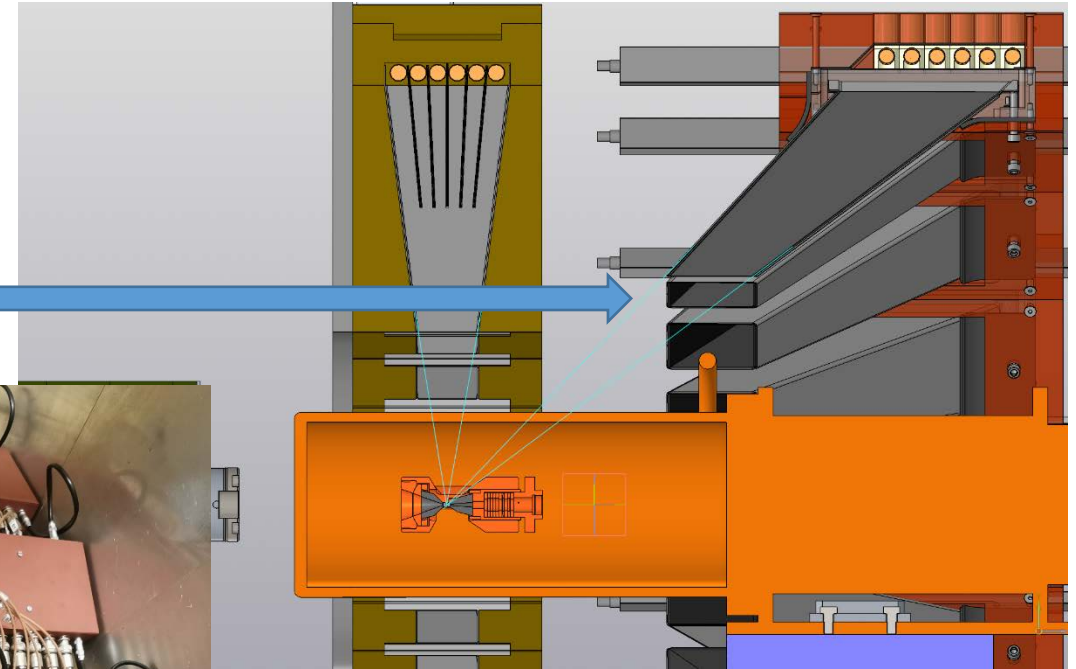
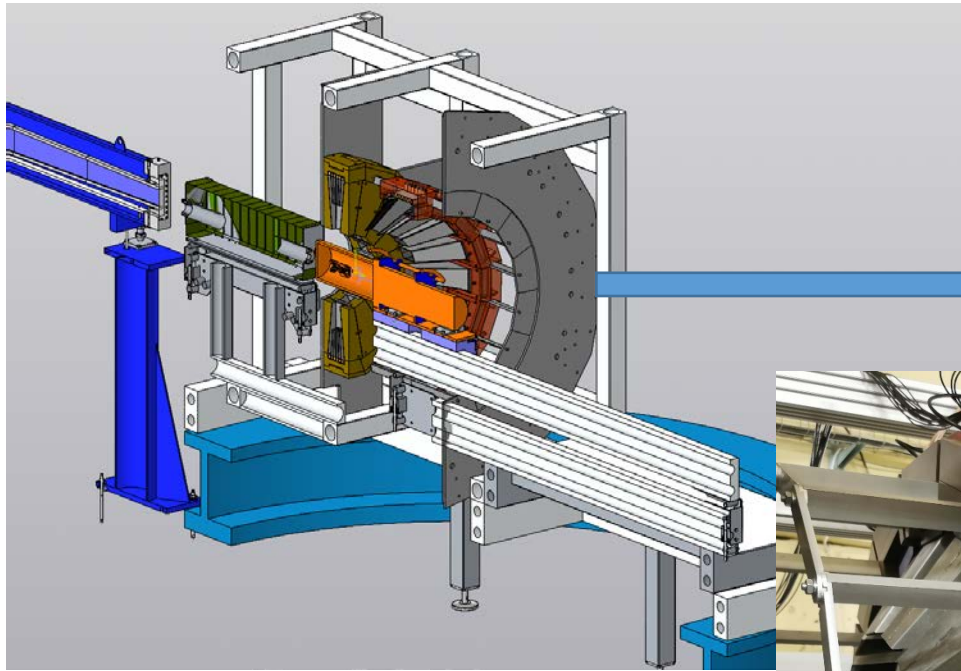


He filled neutron counter (8 bar)



One module

DN-6: detector system/45 degrees detector ring



90 independent neutron counters group in
15 independent modules within
6 counters in each module
32-54 2θ deg. covering

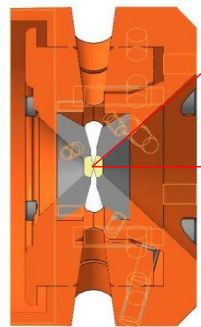
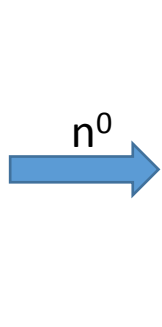
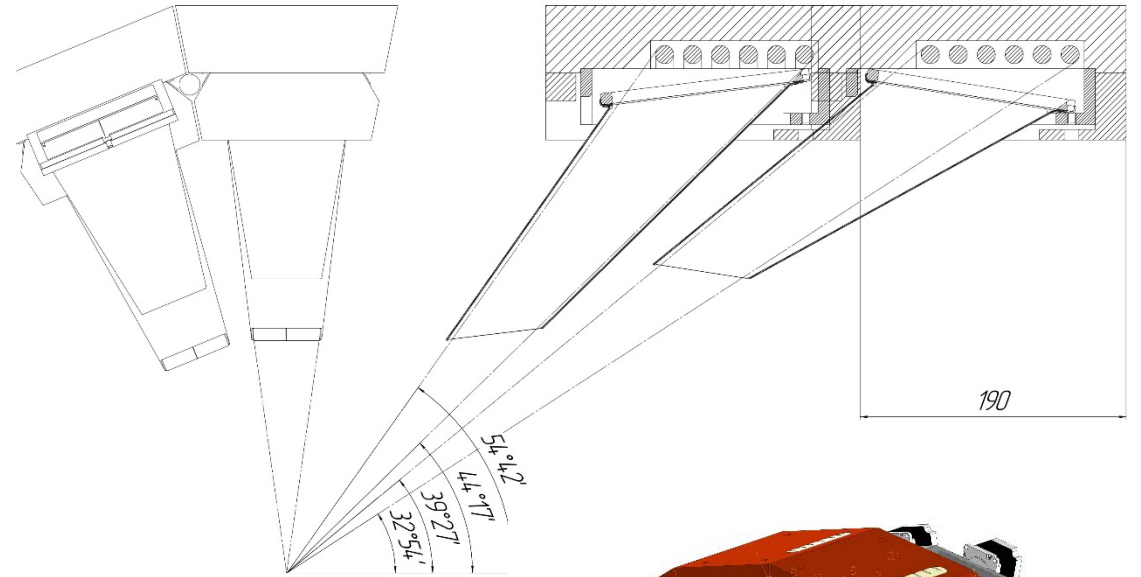
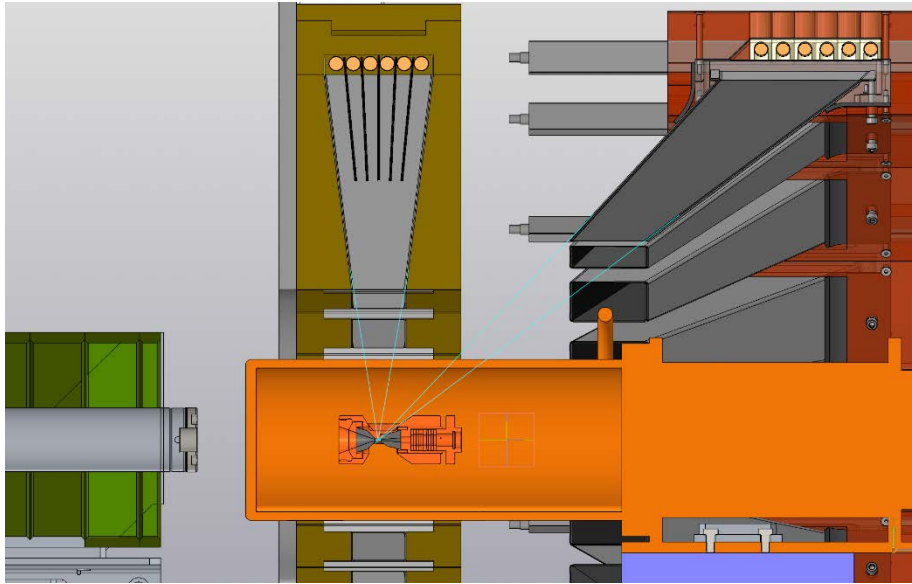


He filled neutron counter (8 bar)

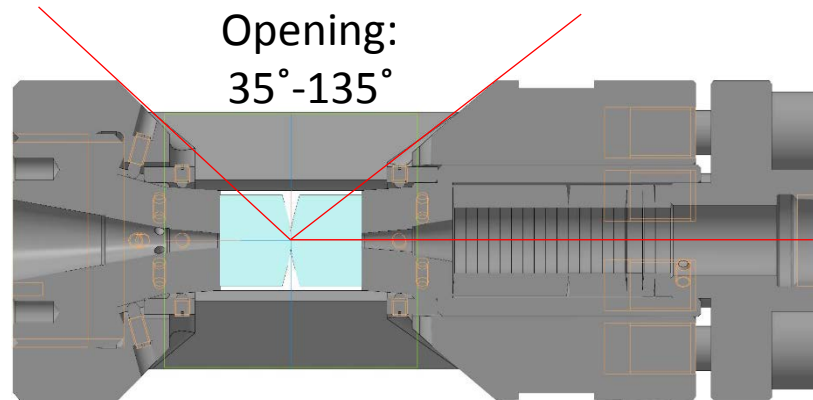


45 deg. detector ring

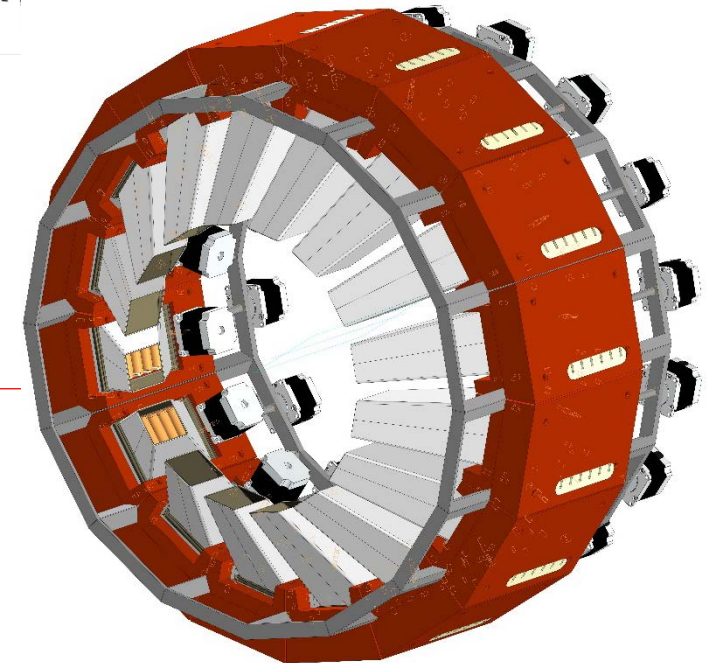
DN-6: detector system/45 degrees detector ring



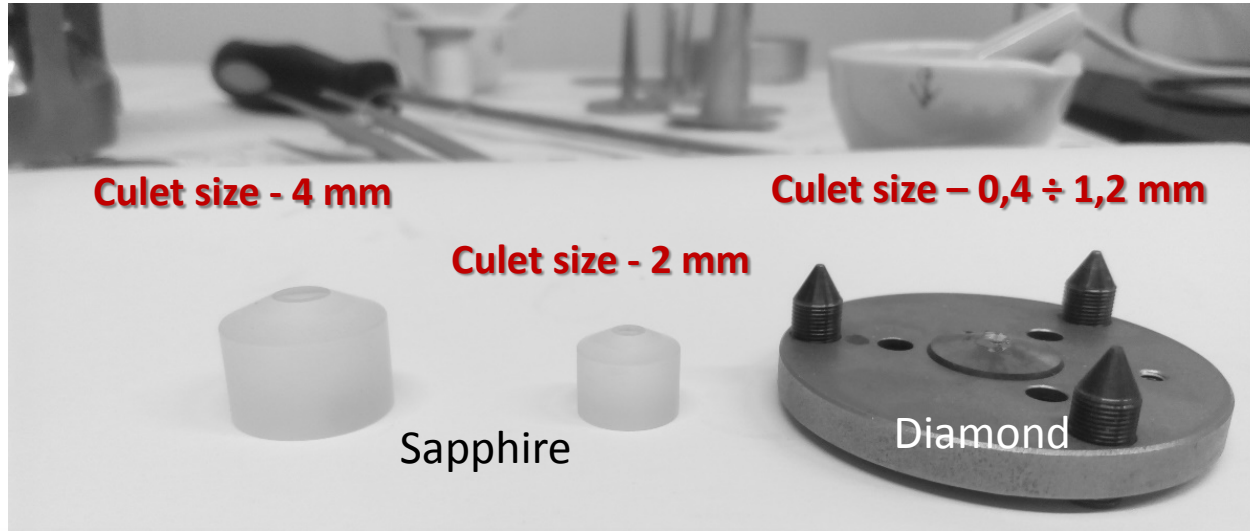
Opening:
 0° - 45°



Opening:
 35° - 135°



DN-6: high-pressure techniques.



Types of anvils used in high pressure experiments

Types of cells used in high pressure experiments



Sapphire anvils cells



Diamond anvils cells (DAC)

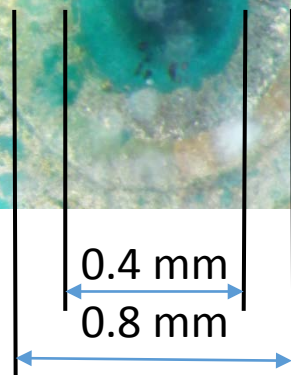
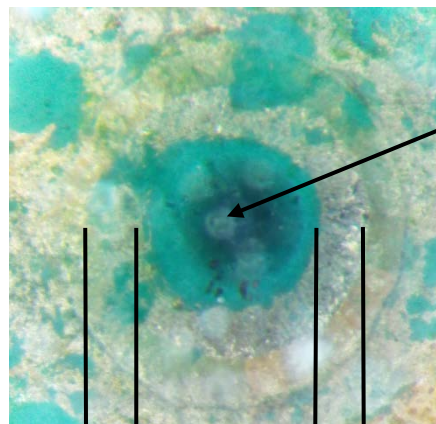


Böhler Almax Plate DAC

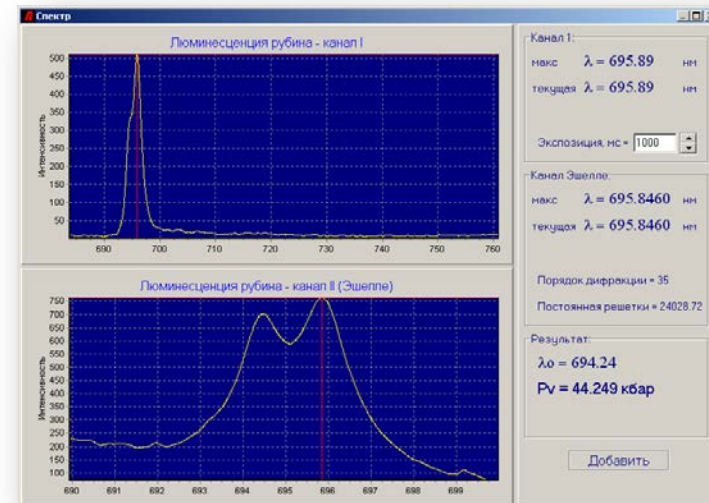
DN-6: Diamond anvil cell.



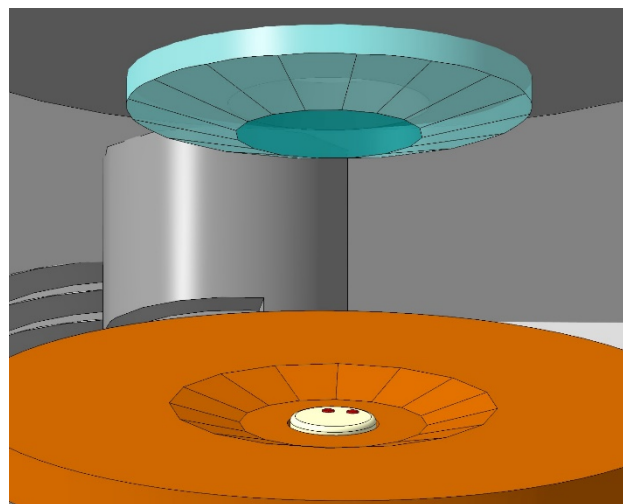
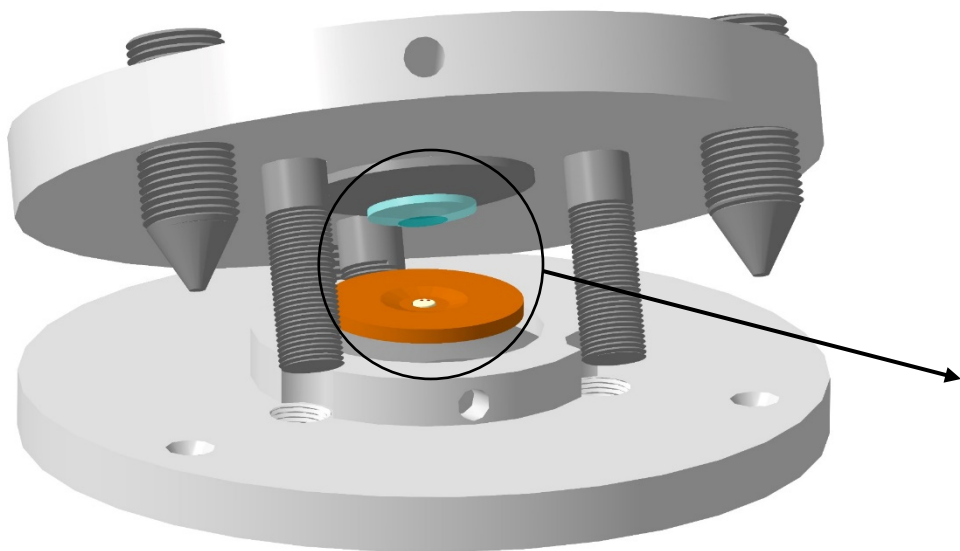
Thickness = 0.1 mm
Diameter = 0.4 mm
 $V \sim 0.02 \text{ mm}^3$
 $P_{\text{max}} \sim 40 \text{ GPa}$



Ruby ball

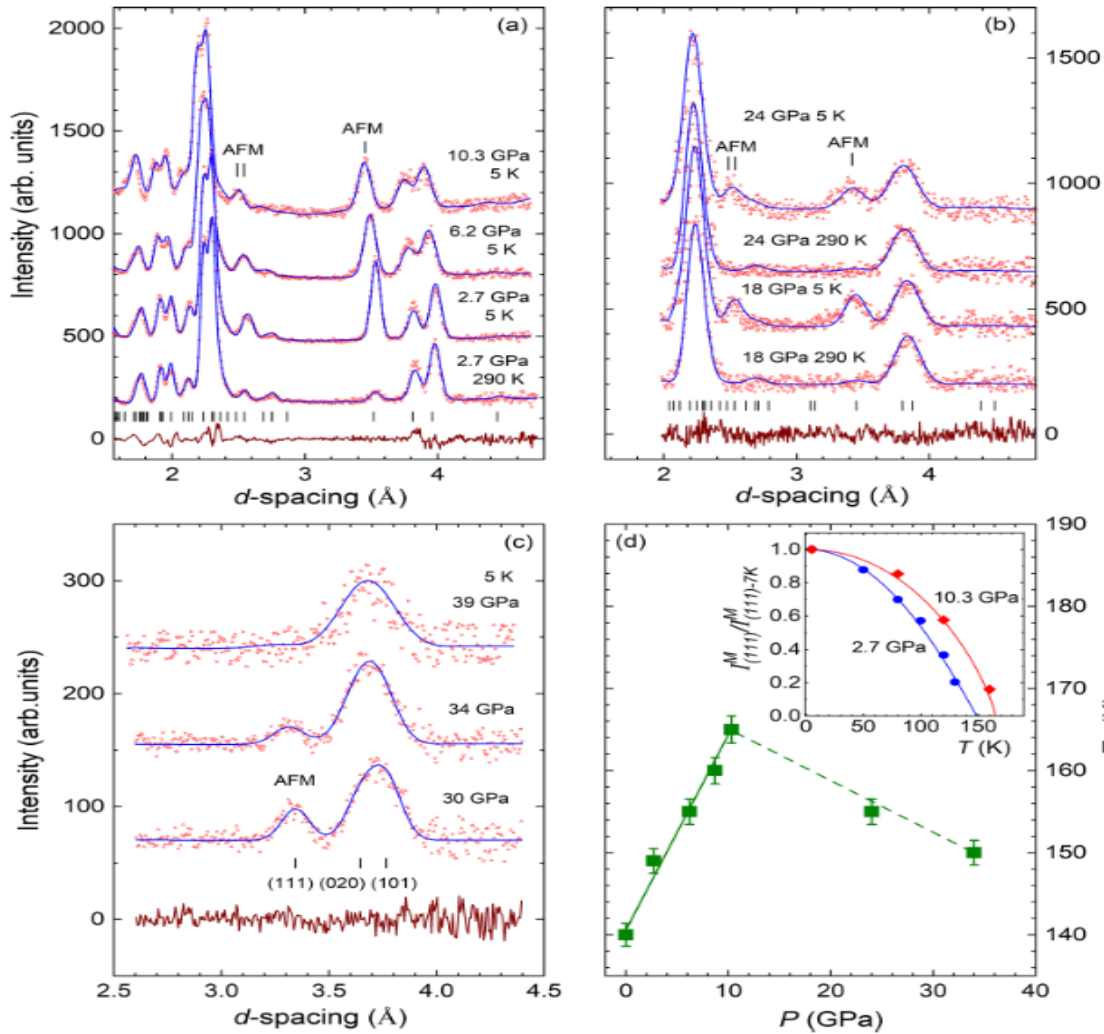


Boehler-Almax Plate DAC

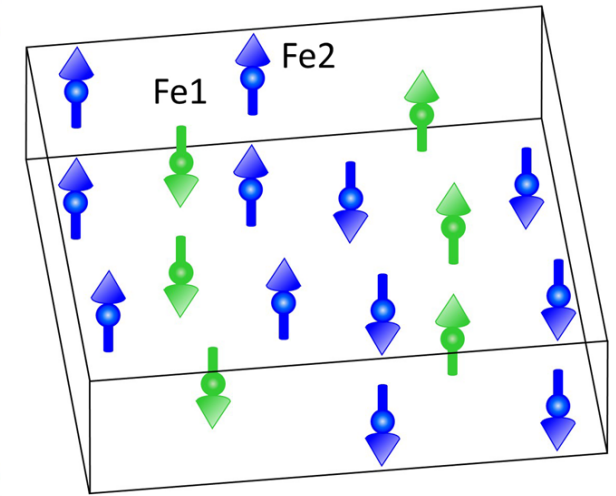
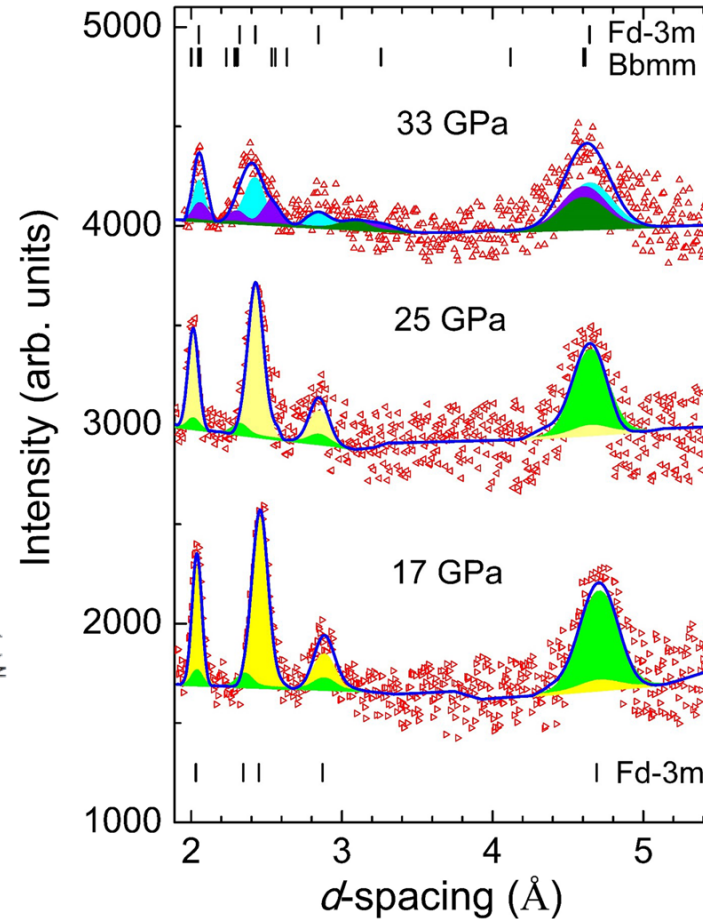


Culet size from 0.4 to 1.2 mm

DN-6: some scientific results.



*D. P. Kozlenko, E. V. Lukin, S. E. Kichanov, Z. Jiráček, N. O. Golosova, and B. N. Savenko “High-pressure evolution of the magnetic order in LaMnO_3 ”, Phys. Rev. B **107**, 144426 (2024)*



*Kozlenko, D.P., Dubrovinsky, L.S., Kichanov, S.E. et al. Magnetic and electronic properties of magnetite across the high pressure anomaly. Sci Rep **9**, 4464 (2019).*

Summary

Neutron diffractometer DN-6 at the beamline 6b of the high-flux pulsed reactor IBR-2 in the Frank Laboratory of Neutron Physics is used to study the effect of high pressure on the crystal and magnetic structures of materials. The instrument is equipped with high-pressure cells with sapphire and diamond anvils that provides pressures up to 50 GPa and a closed-cycle helium refrigerator that produced temperatures on the order of 4K.

Thank you for your attention!