

- **High pressure cells for neutron scattering.**

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- * sadykov@inr.ru

Общая схема эксперимента по исследованию твердого молекулярного водорода под высоким давлением на импульсном нейтронном источнике ускорителя «Факел»:

- 1 - детектор спектра нейтронов прямого пучка;
- 2 - спектрометр неупругого рассеяния нейтронов (монокристалл + фильтр);
- 3 - образец твердого водорода в камере высокого давления и гелиевом криостате;
- 4 - гидравлический пресс;
- 5 - многодетекторный нейтронный дифрактометр по времени пролета;
- 6 - водяной замедлитель с регулируемой длительностью импульса тепловых нейтронов;
- 7 - урановая мишень ускорителя

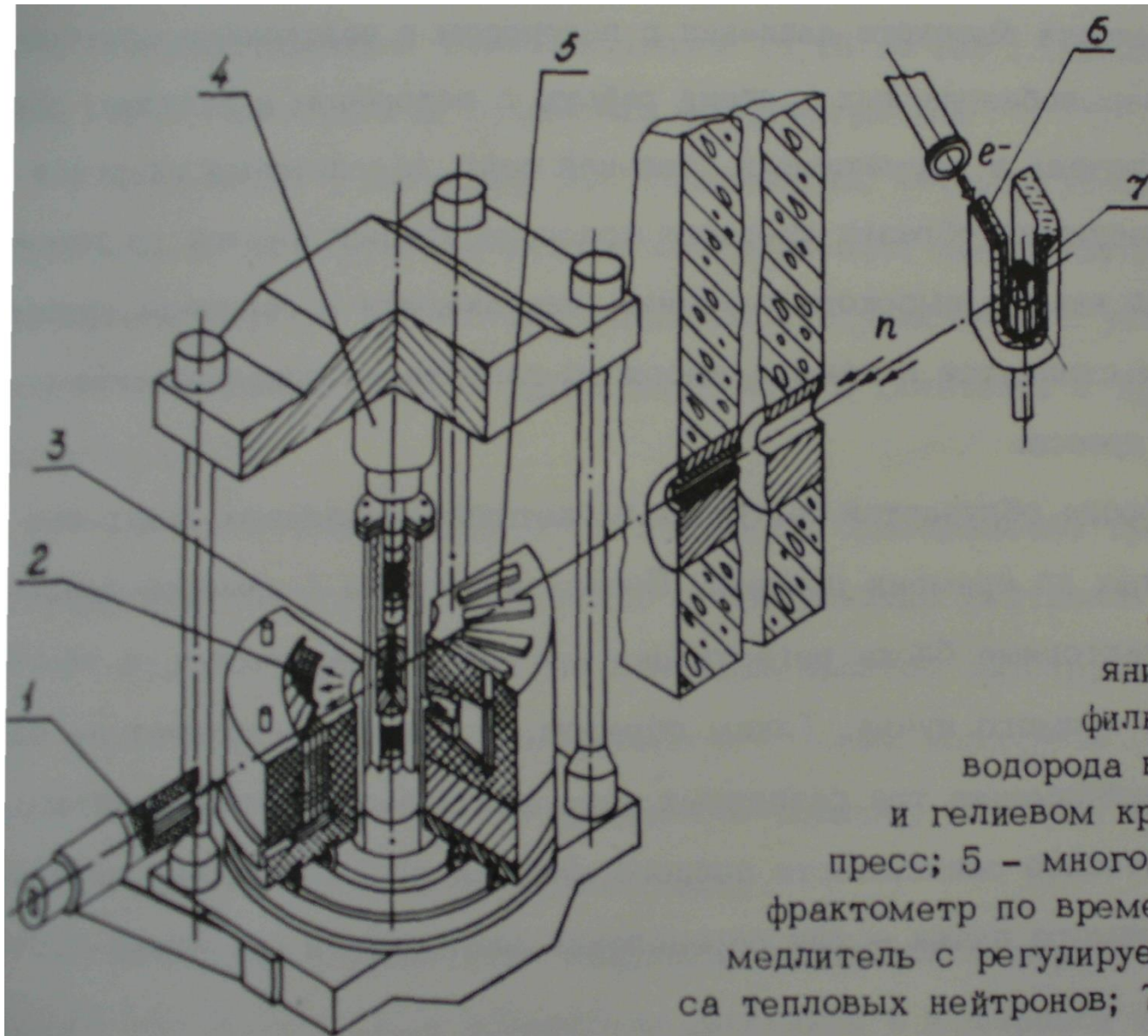
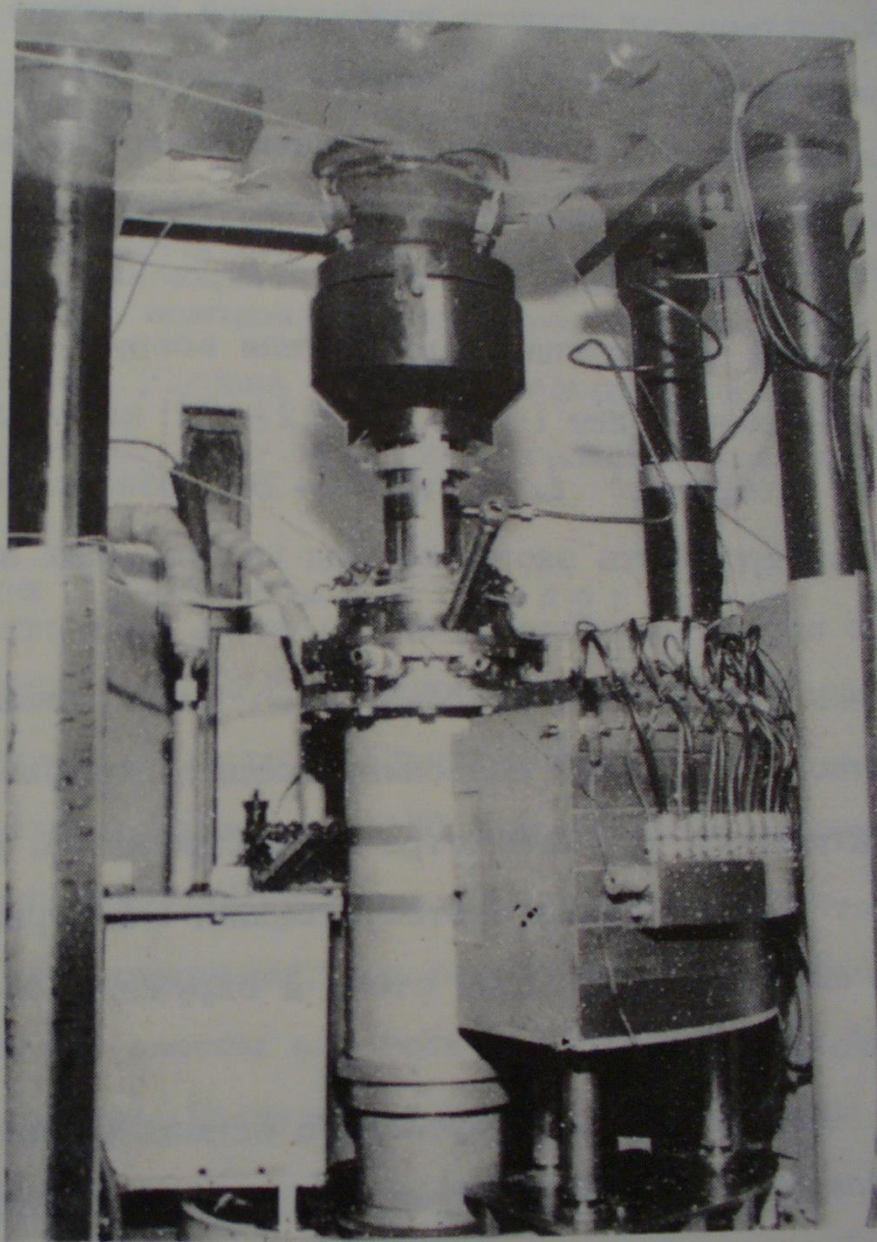


Рис. 2. Гидравлический пресс, гелиевый криостат с камерой высокого давления, блоки дифрактометра и спектрометра на нейтронном пучке. Дверь защитного сейфа установки открыта



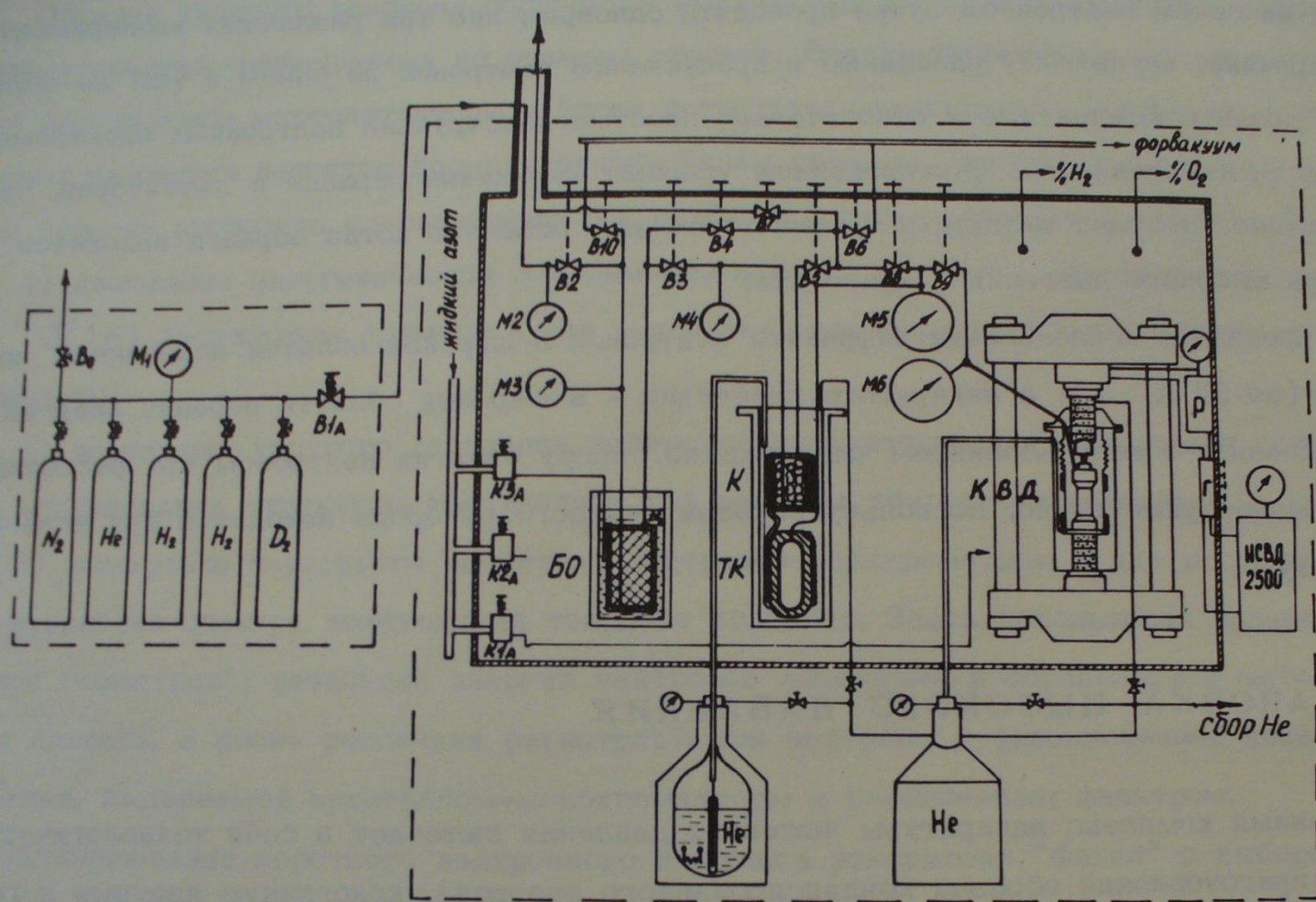
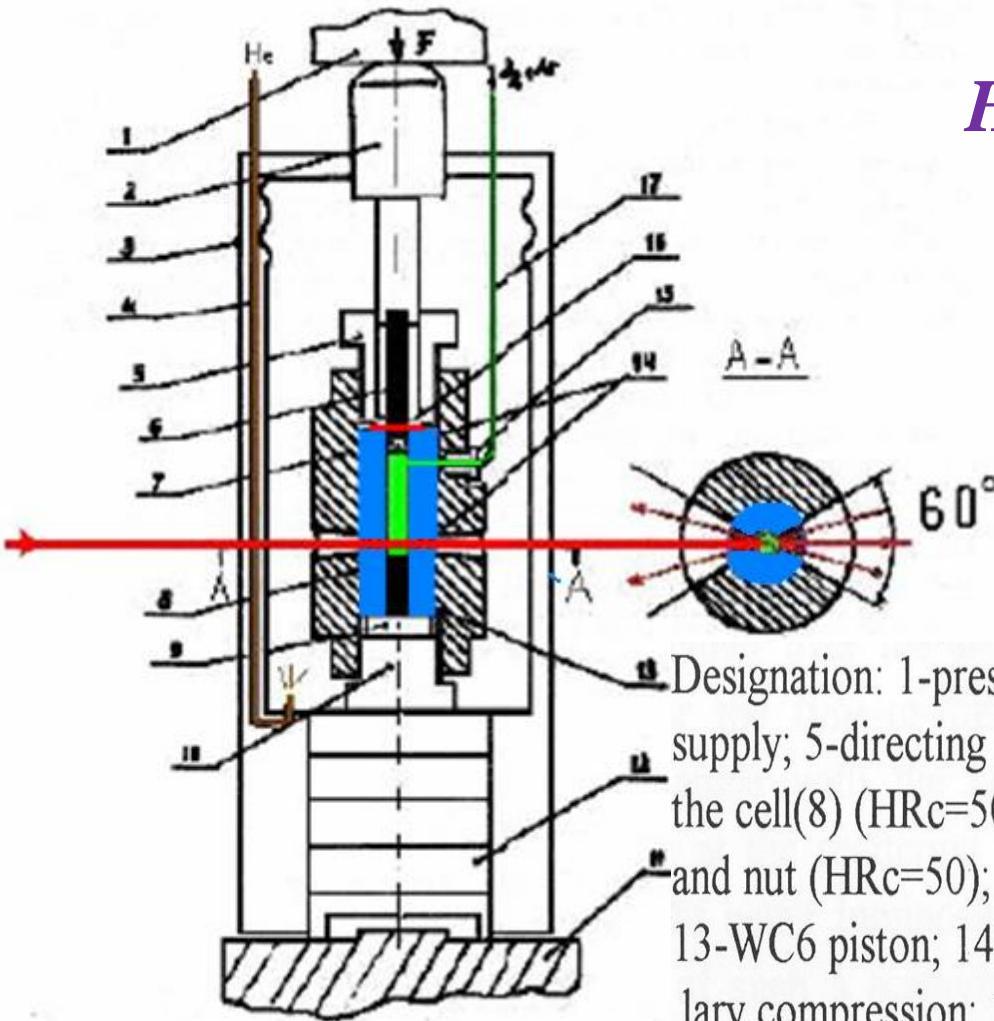


Рис. 3. Технологическая схема работы с водородом: В1-В10 - вентили газовой водородной системы (В1А - с автоматическим приводом); М1-М6 - манометры газовой системы; БО - блок очистки газа; К - орто-пара-конвертор; ТК - термокомпрессор; КВД - камера высокого давления; К1А-К3А - электромагнитные клапаны криогенной азотной системы; Г - управляющие вентили гидравлической системы пресса; Р - ресивер гидравлической системы

Для ряда экспериментов требуется приготовить образцы водорода различного

High pressure apparatus for neutron diffraction investigation of the Strongly compressible substances (H_2 , D_2 , Ar)

*High Pressure Research,
1995, Vol.14, pp199-202.*



Designation: 1-press piston; 2-pusher; 3-cryostat; 4-tube for gas helium supply; 5-directing nut; 6-HPA piston of WC6; 7-alloy steel support of the cell(8) (HRc=50); 8-HPA cell of TiZr alloy; 9,10-alloy steel washer and nut (HRc=50); 11-lower press slab; 12-thermo-insulating column; 13-WC6 piston; 14-compression of piston-fungus type; 15-nut for capillary compression; 16-copper diaphragm; 17- capillary for gas or liquid supply into HPA cell.

Neutron diffraction pattern (TOF) of solid D_2 under pressure $P=24\text{kbar}$ ($T=40\text{K}$) and $P=0\text{kbar}$ ($T=4.2\text{K}$).

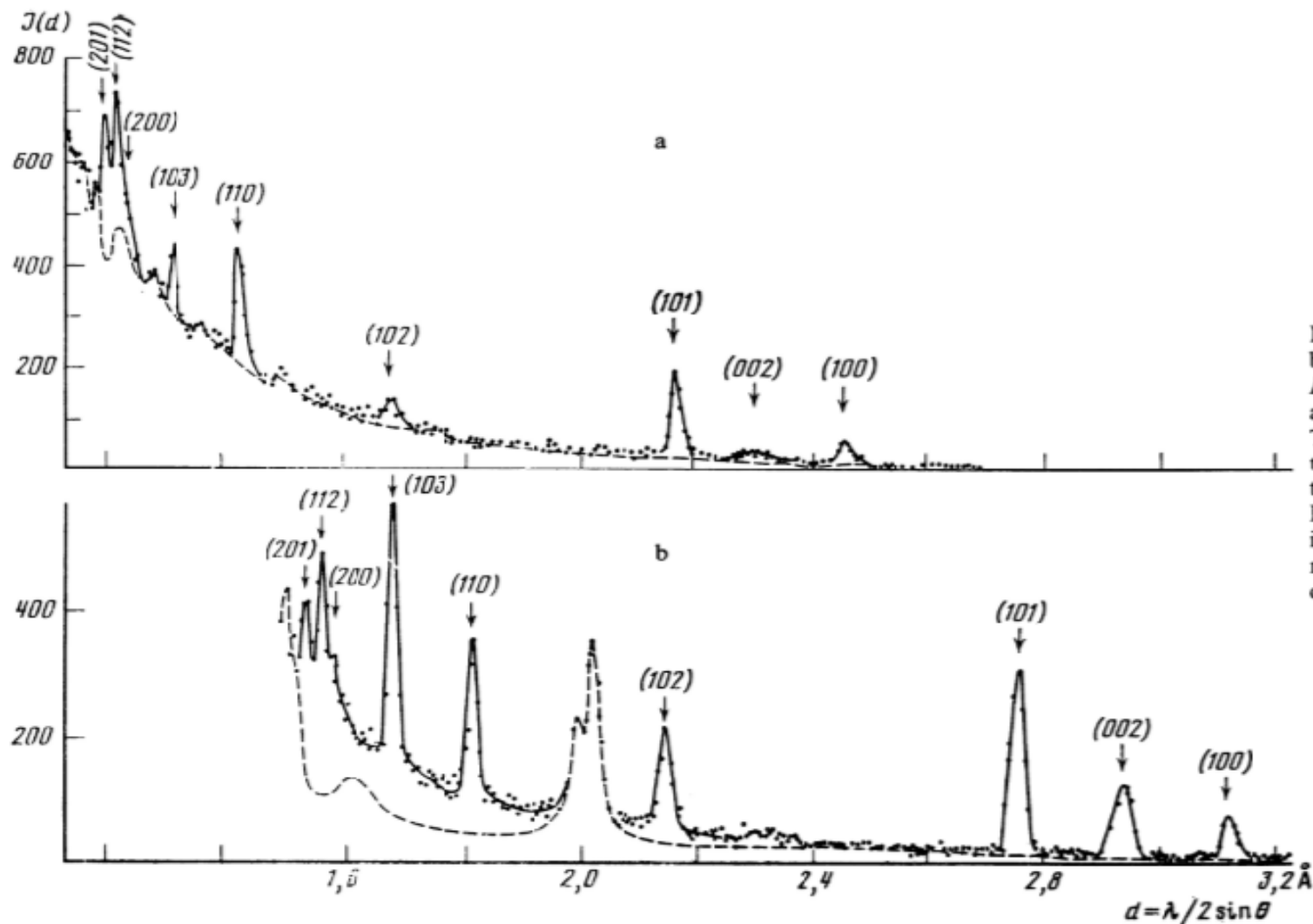


FIG. 1. Neutron diffraction by solid orthodeuterium at $P = 25.3 \text{ kbar}$, $T = 40 \text{ K}$ (a) and $P = 0$, $T = 4.2 \text{ K}$ (b). The arrows indicate the positions of the reflections from the hcp structure of ortho- D_2 . The background scattering from the chamber material is indicated by the dashed lines.



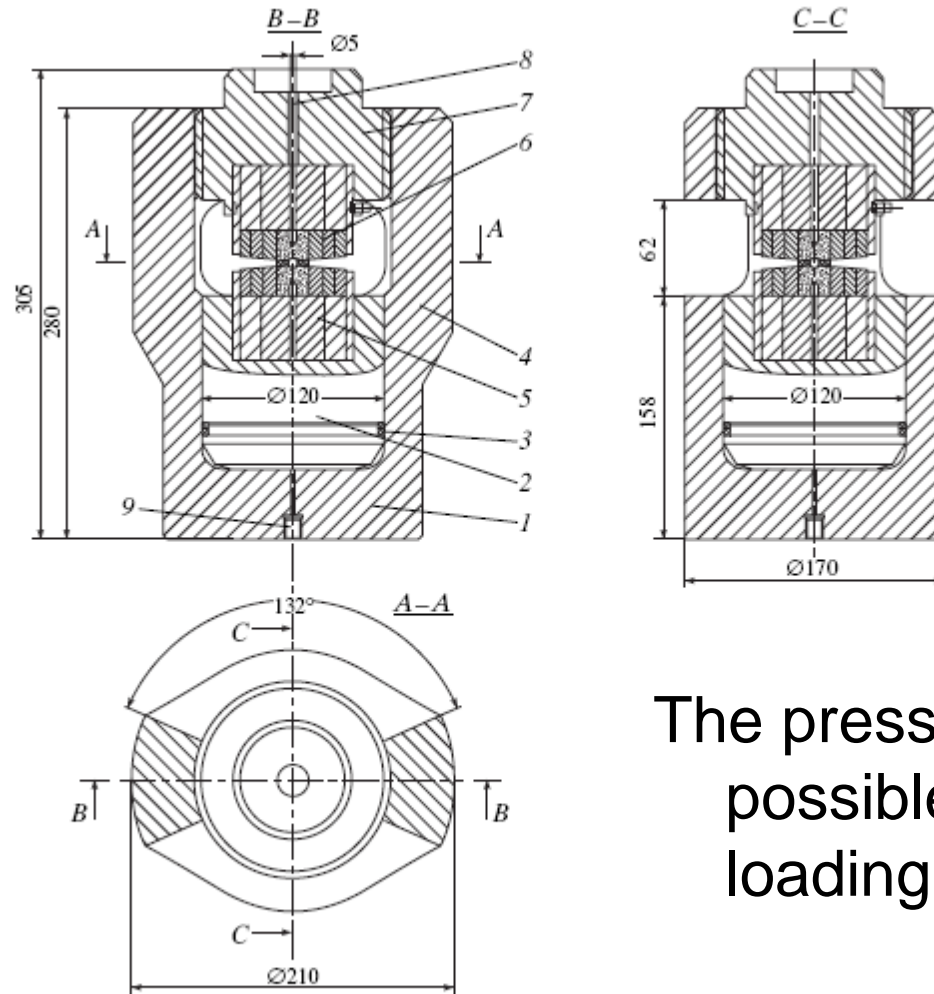
A Compact Hydraulic Press to Use with High-Pressure Devices for Neutron Scattering Studies

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¹ Vereshchagin Institute of High-pressure Physics, Russian Academy of Science, Troitsk, Moscow oblast, 142190 Russia
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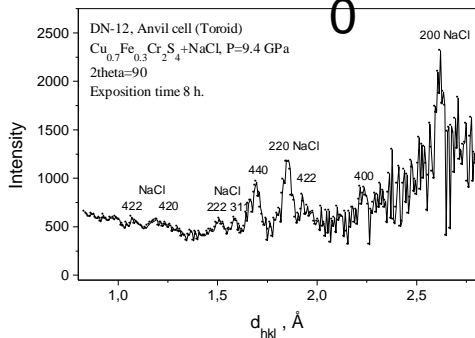
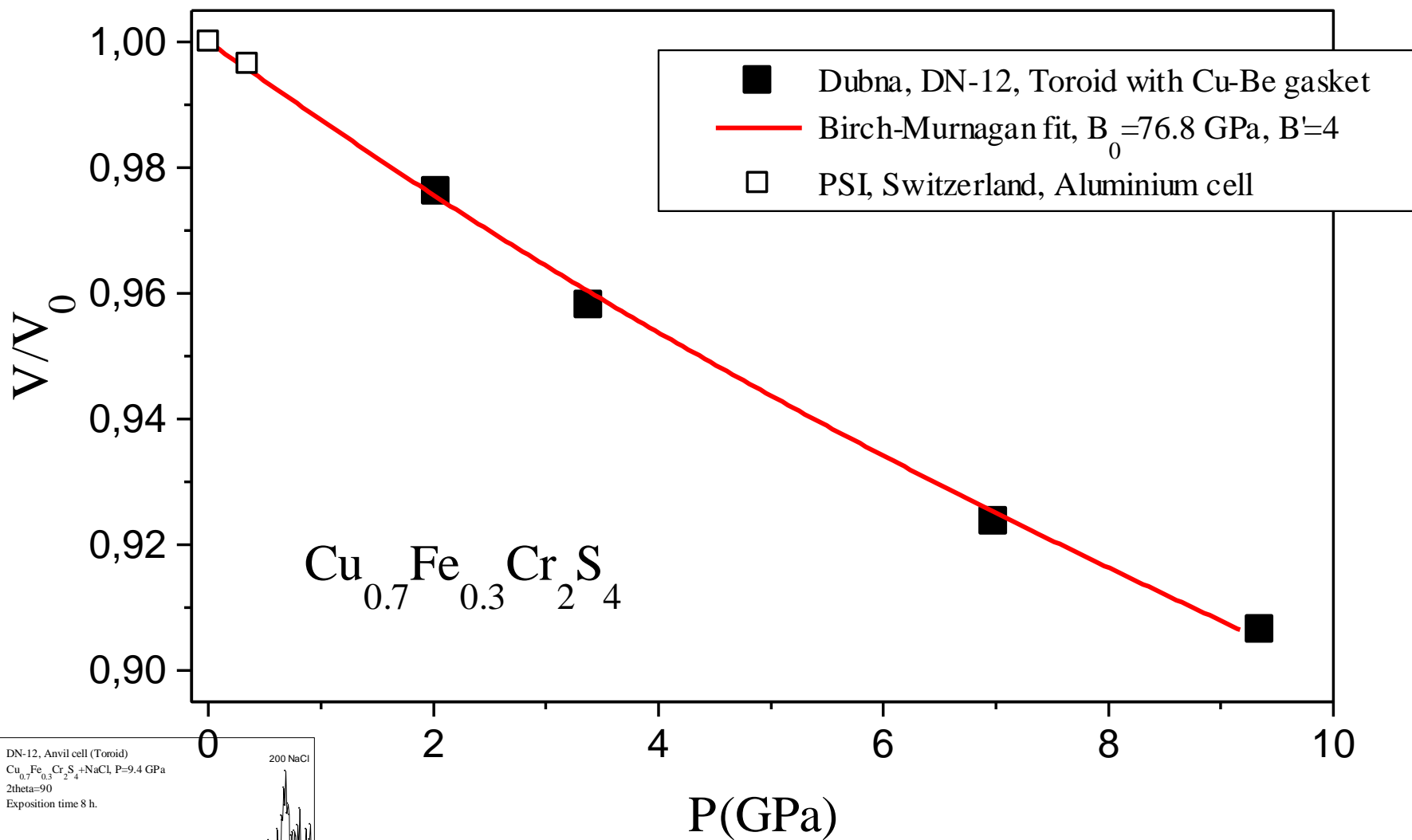
² Los Alamos National Laboratory

Received November 28, 2001



The press makes it possible the maximum loading up to 250 tons

Fig. 1. The compact hydraulic press with a 200-t force: (1) power cylinder; (2) ram; (3) O-shaped rubber sealing ring; (4) supporting frame; (5) supporting block; (6) high-pressure cell; (7) threaded plug; (8) a neutron-beam entrance aperture for experiments in the axial geometry; and (9) hydraulic liquid feed inlet.



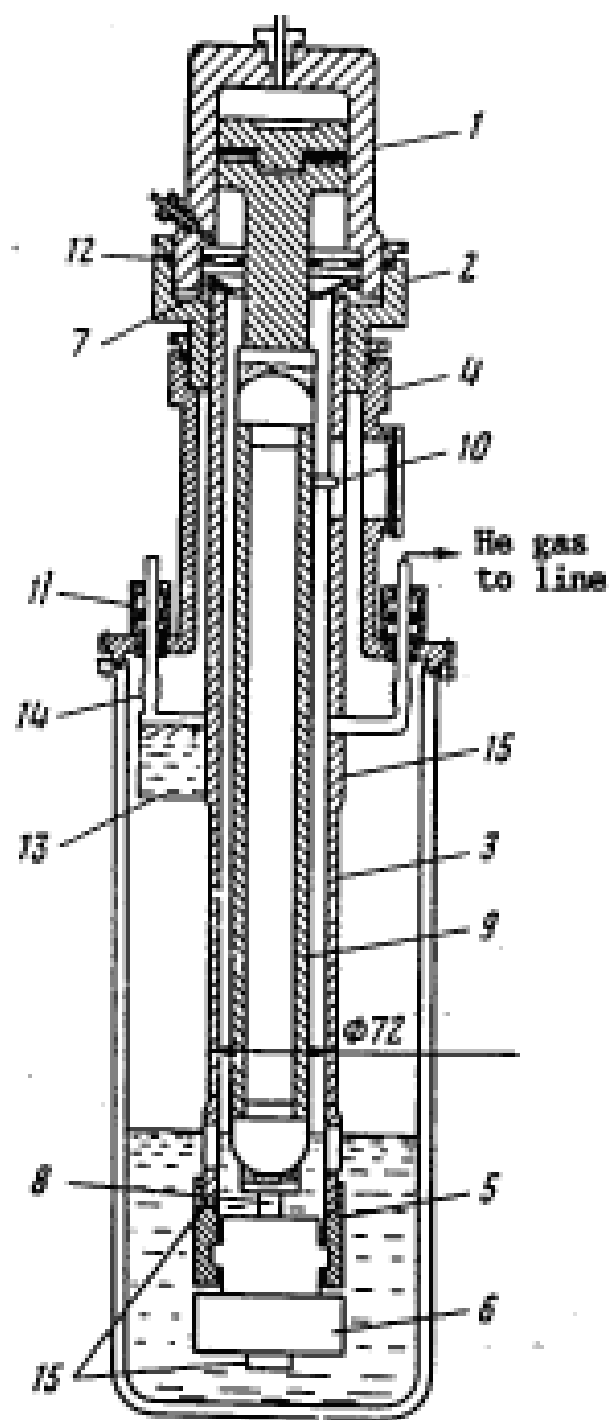
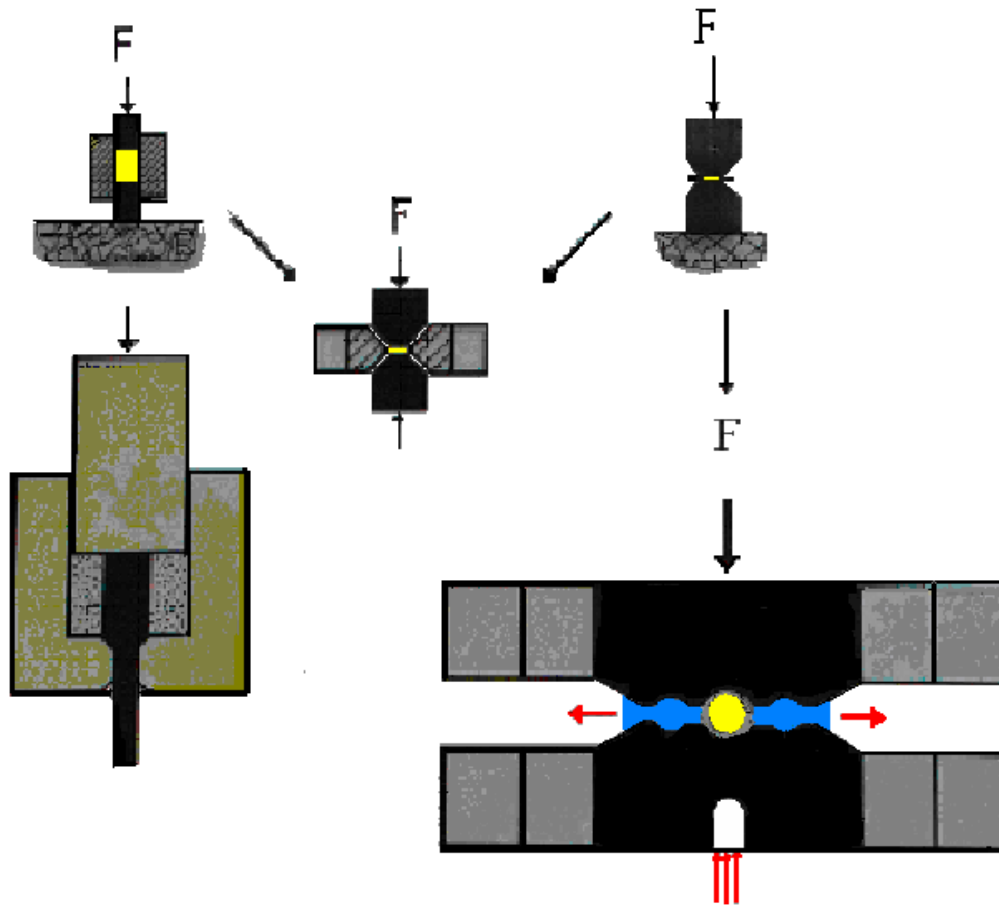


Fig. 1. Press with cryostat: 1) cylinder; 2) coupling; 3) outer tube; 4) part joining press to cryostat; 5) lock; 6) high-pressure chamber; 7) disk spring; 8) shaft of high-pressure chamber; 9) inner tube of press; 10) mark; 11) sleeves; 12) vacuum-rubber gas-gaskets; 13) vessel for liquid nitrogen; 14) bellows; 15) attachment points for thermocouple junctions.

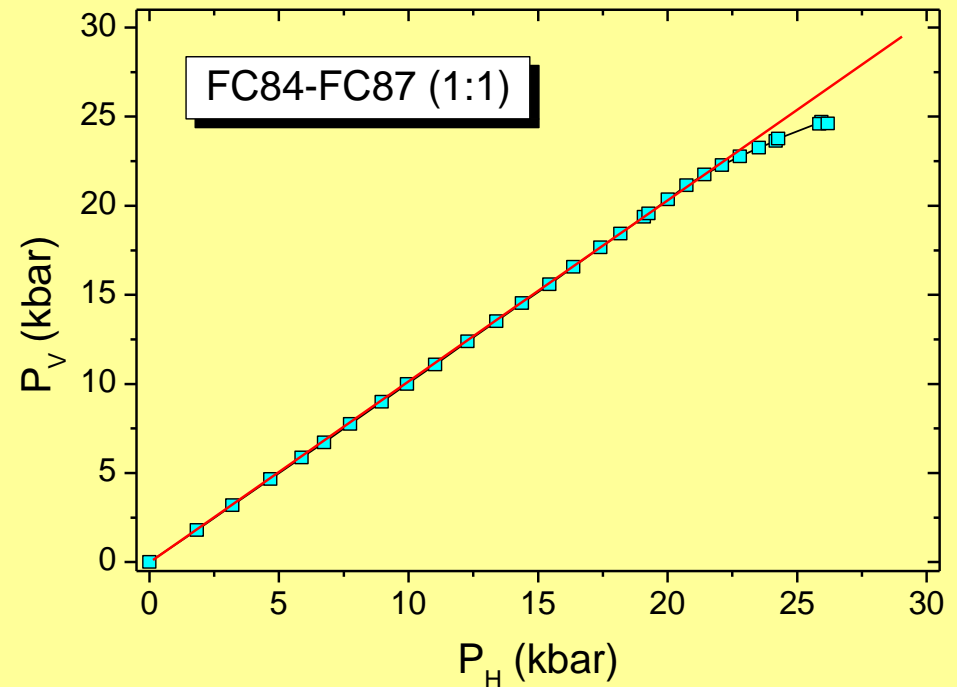
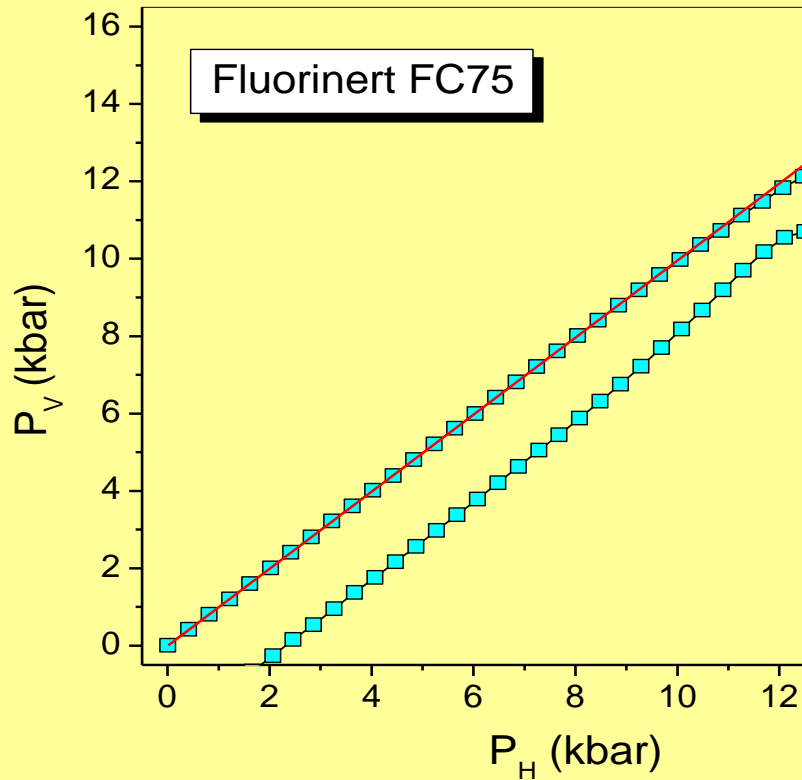
Geomerty of the High Pressure Cells (Type of the High pressure Cells)

1. Piston-cylinder - (up to 60kbar)
2. Belt - (200 kbar)
3. Anvil - (3Mbar)
4. Toroid (Paris-Edinburg)- (250 kbar)

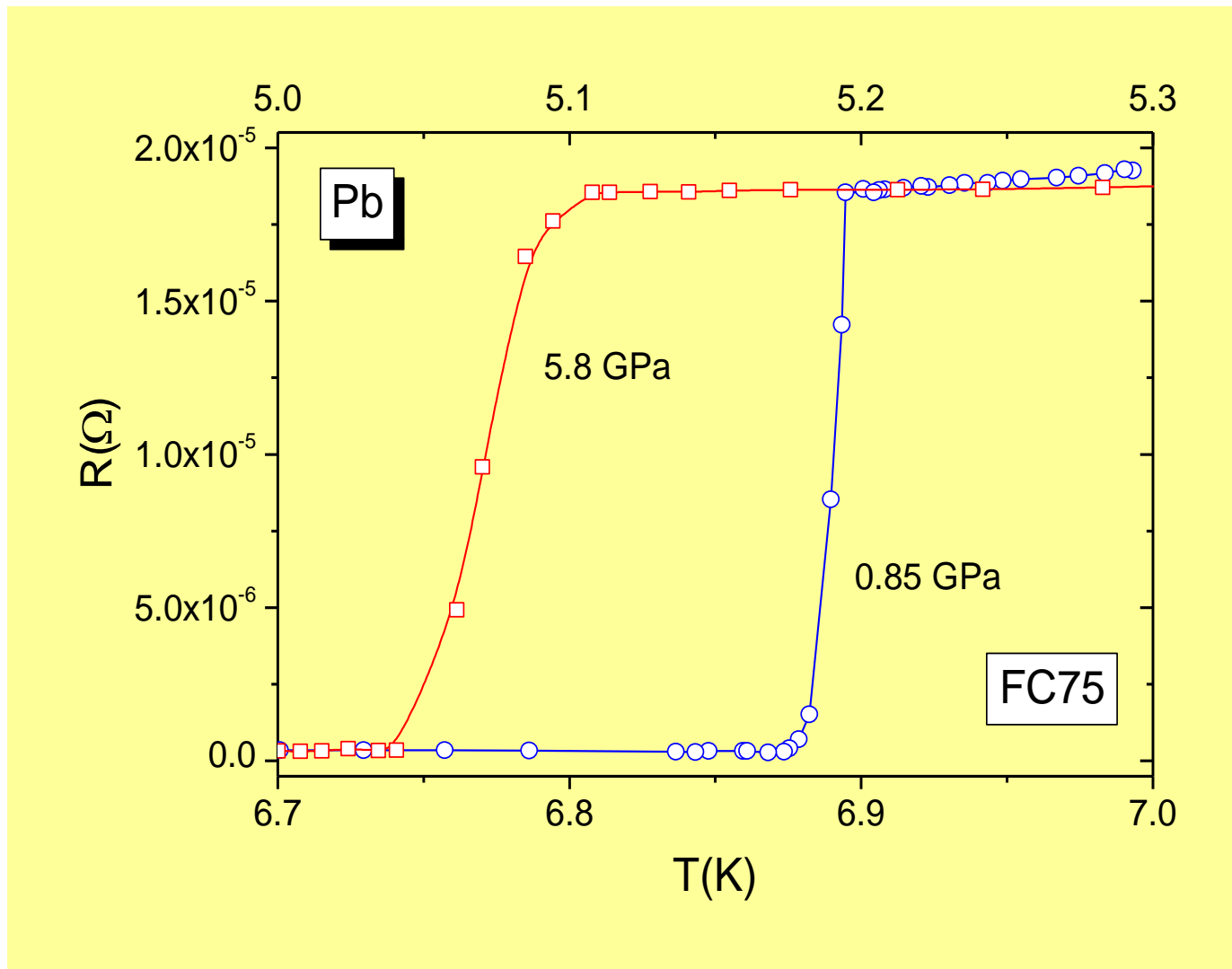


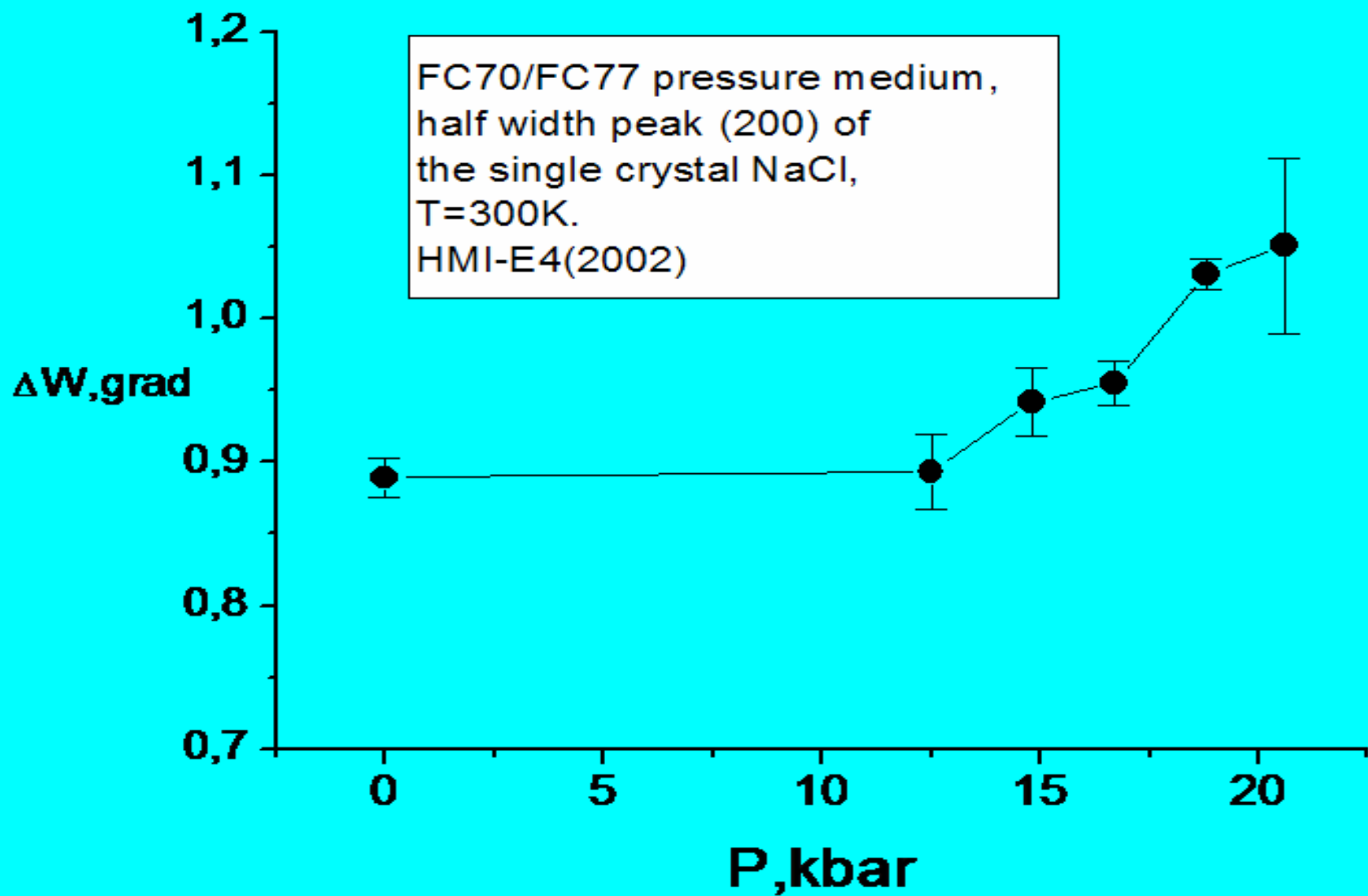
- Fluorinerts - especially mixture (1:1) FC70-FC77 are used for magnetic and transport studies up to 2 GPa in a piston-cylinder cell and up to 8 GPa in a multianvil cubic pressure cell.
- Shear stresses, developed in a liquid, when it solidifies at high pressure - above the hydrostatic limit - may influence strongly the properties of single crystal immersed in it. For this reason the knowledge of hydrostatic limit of liquid in use is of practical importance for interpretation of the results of measurements - sometimes very complicated and time consuming ones.
- In the present study we determined the hydrostatic limits at room temperature of a number of Fluorinert liquids: FC70, FC75, FC77, FC84, FC87 and their mixtures and show how the surpass of this limit produce pressure gradients in the sample, which retains at low temperature. Maximum hydrostatic limit (2.3 GPa) is found for (1:1) mixture of FC84-FC87.

Typical experimental scans of vertical vs horizontal manganin pressure sensor readings for FC75 и FC84-FC87 (1:1). Solidification of liquid takes place in the point, where the the relation $P_H=P_V$ is no more valid.



Температурные зависимости электросопротивления свинцового образца в области сверхпроводящего перехода при различных давлениях. Среда, передающая давление – Fluorinert FC75.

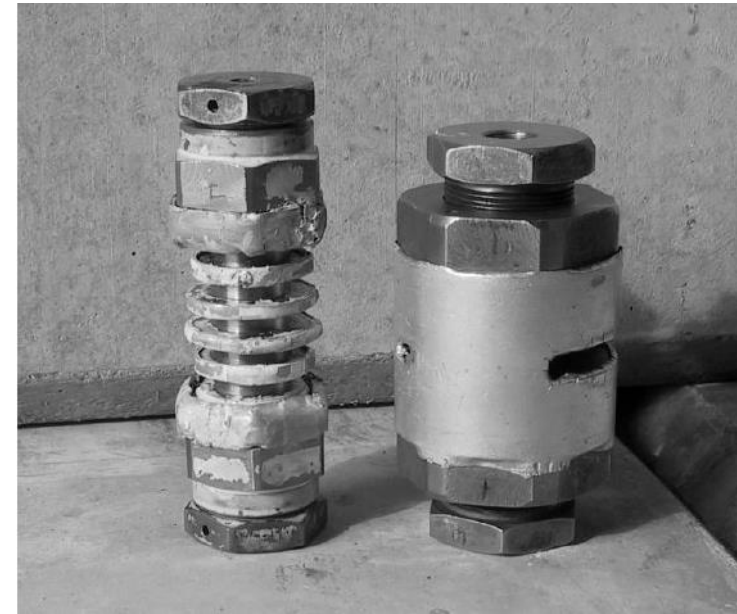
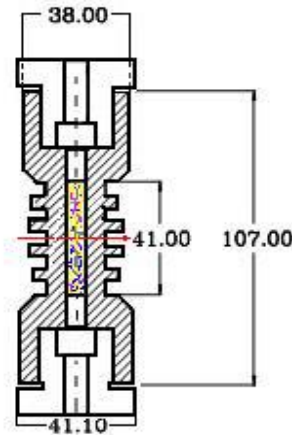
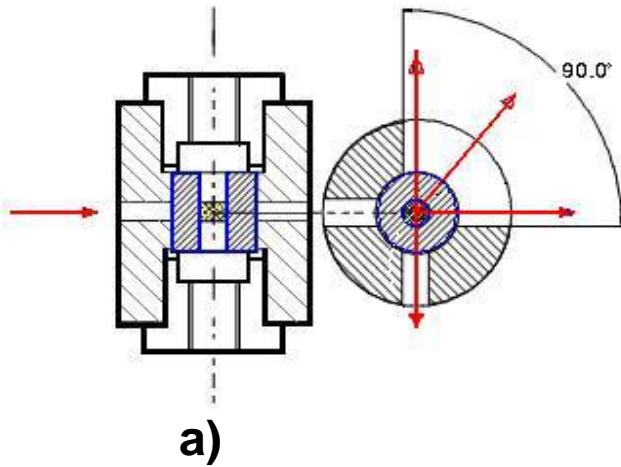




High pressure cells for powder diffraction

TiZr-zero alloy (RT, 10-12 kbar,
with support up to 29 kbar at LT)

High pressure cells-zero **TiZr** alloy (PSI, Swiss)



- a)** The cell up to **17kbar**;
inner part -zero alloy **TiZr**;
b) **TiZr** -alloy cell up to **12kbar**.
Diameters of the inner hole for the
a)-cell is the **10mm** and for the
b)-cell is the **6mm**.
Height of the sample
for the **a)**-cell is the **6mm**.
for the **b)**-cell is the **25mm**

Support and nuts made from hardness steel alloy and pistons made from WC6.

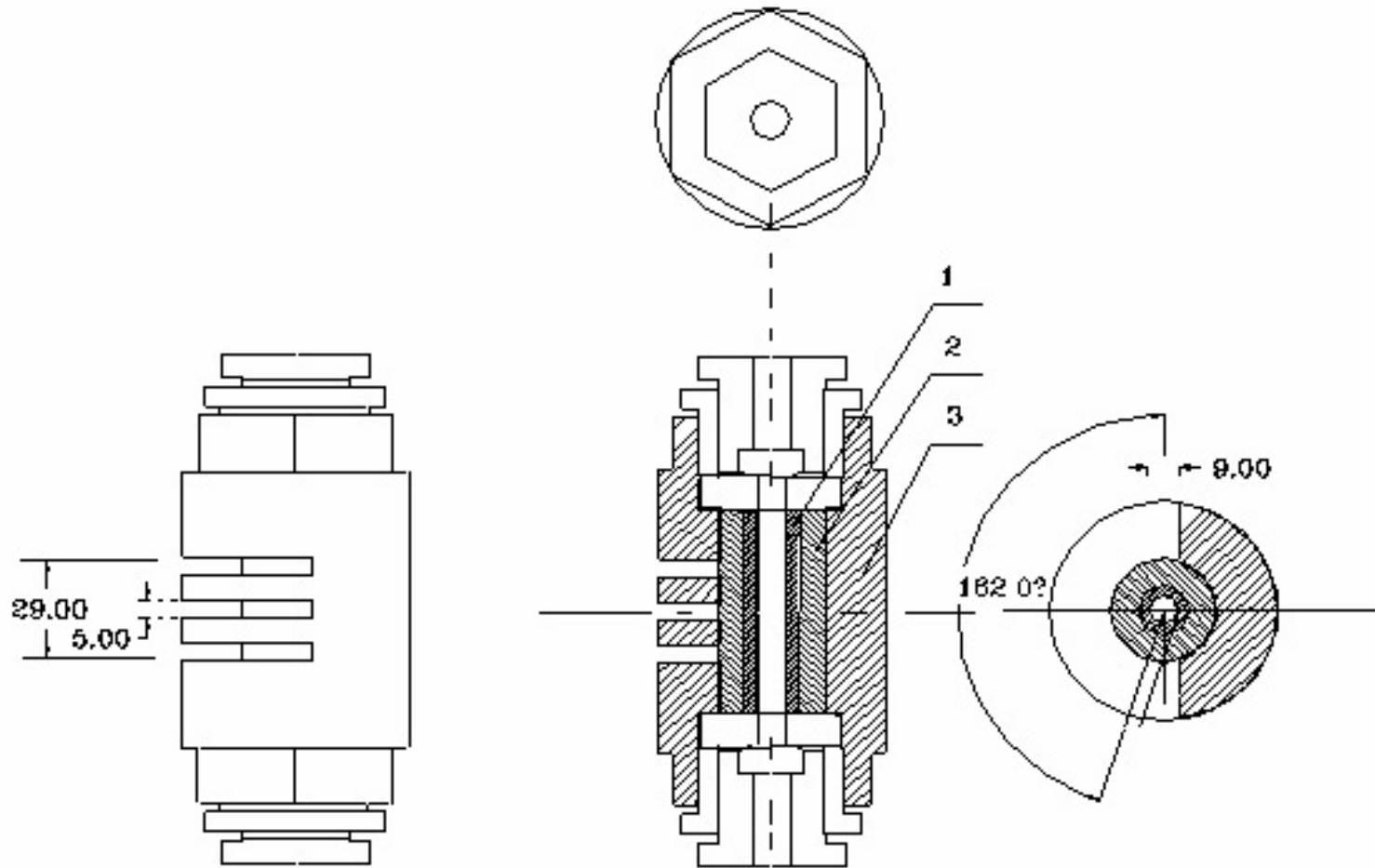
Pictures of the two zero-matrix (**TiZr** alloy) High Pressure Cells used in(PSI-LNS),

left: small cell for use of up to 1.1 GPa(12kbar),

right: big cell for use of up to 1.7 GPa (17kbar) (supported).

High Pressure Cell and Structural Phase Transition (?) In the Itinerant-Electron Ferromagnet MnSi.

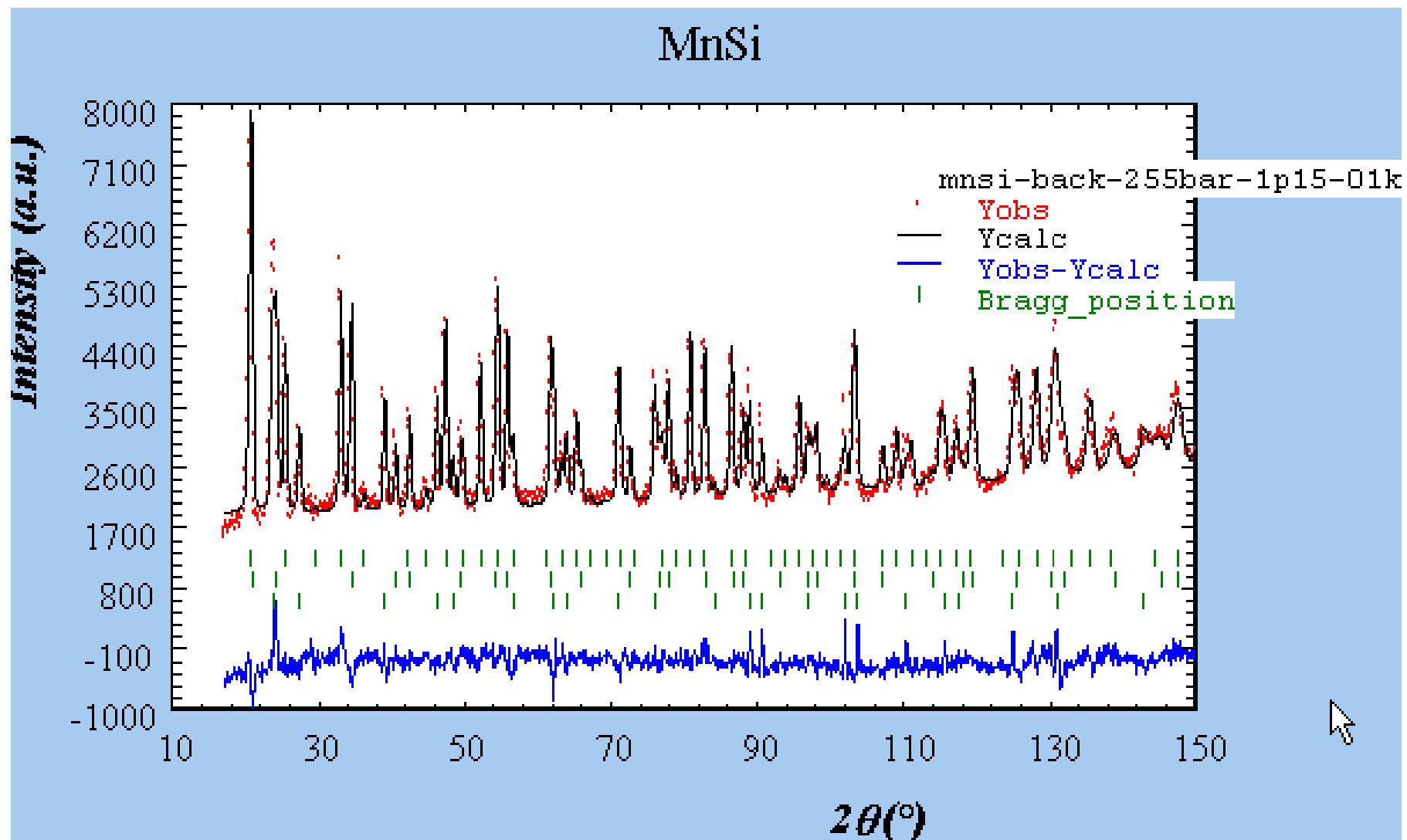
R. Sadykov , B. Fak , and D. Sheptyakov (HPC16-HRPT-PSI)



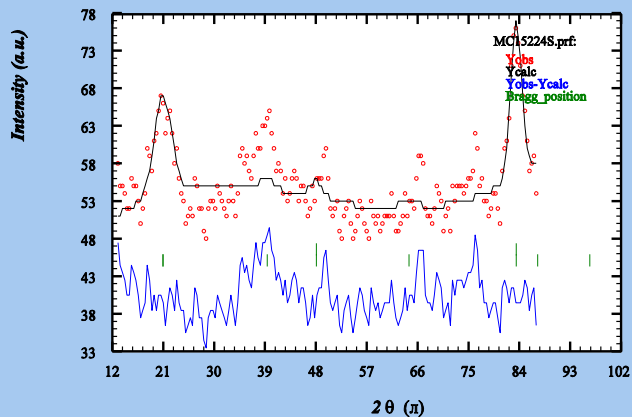
Sketch of the high pressure clamp cell(HPC16) made from zero matrix alloy TiZr ((1,2)and steel support (3).

Neutron diffraction pattern of powder MnSi

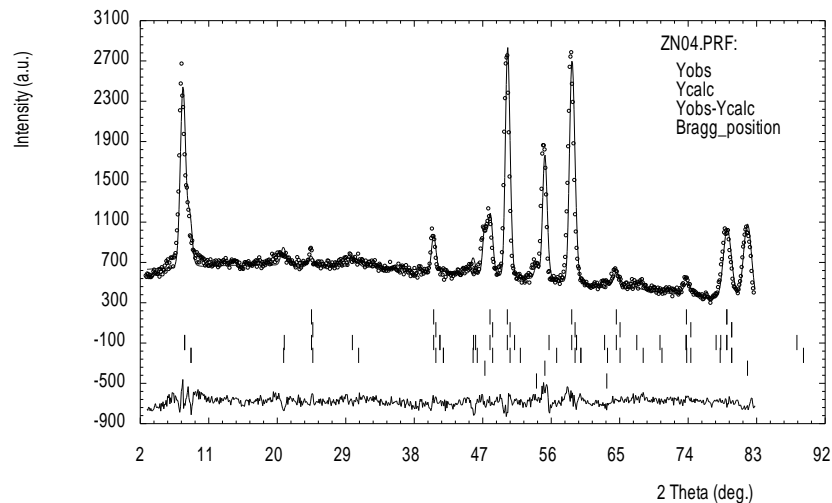
at 14,2kbar and 1.5K.



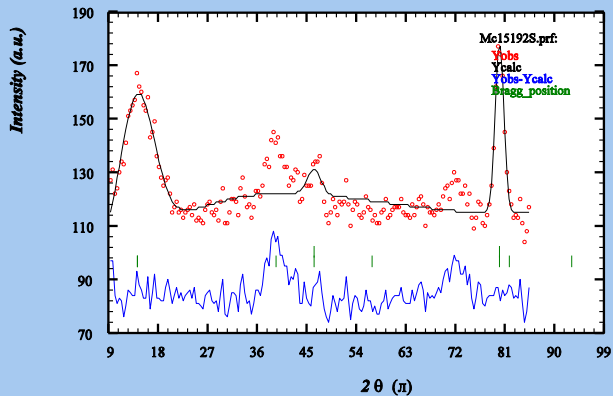
ZnCr₂Se₄; T = 1.5K;



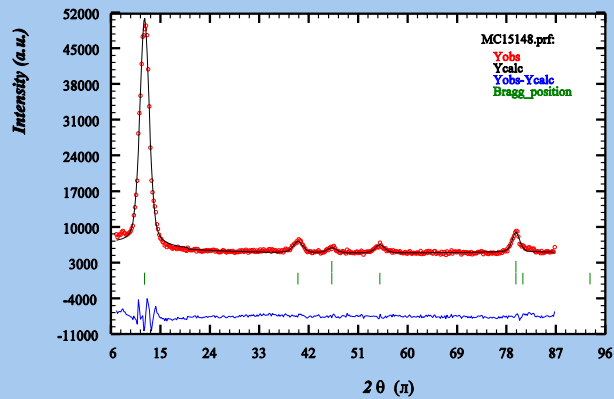
ZnCr₂Se₄+NaCl+Pb+ Pkbar; l = 2.55924Å; T = 4.2K



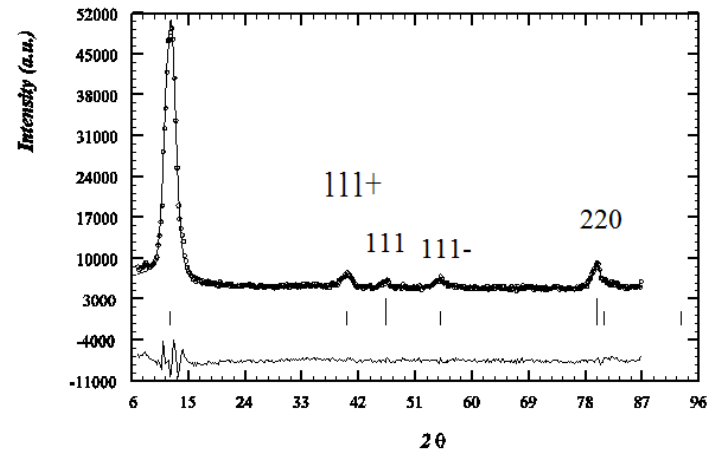
ZnCr₂Se₄; T = 1.5K;

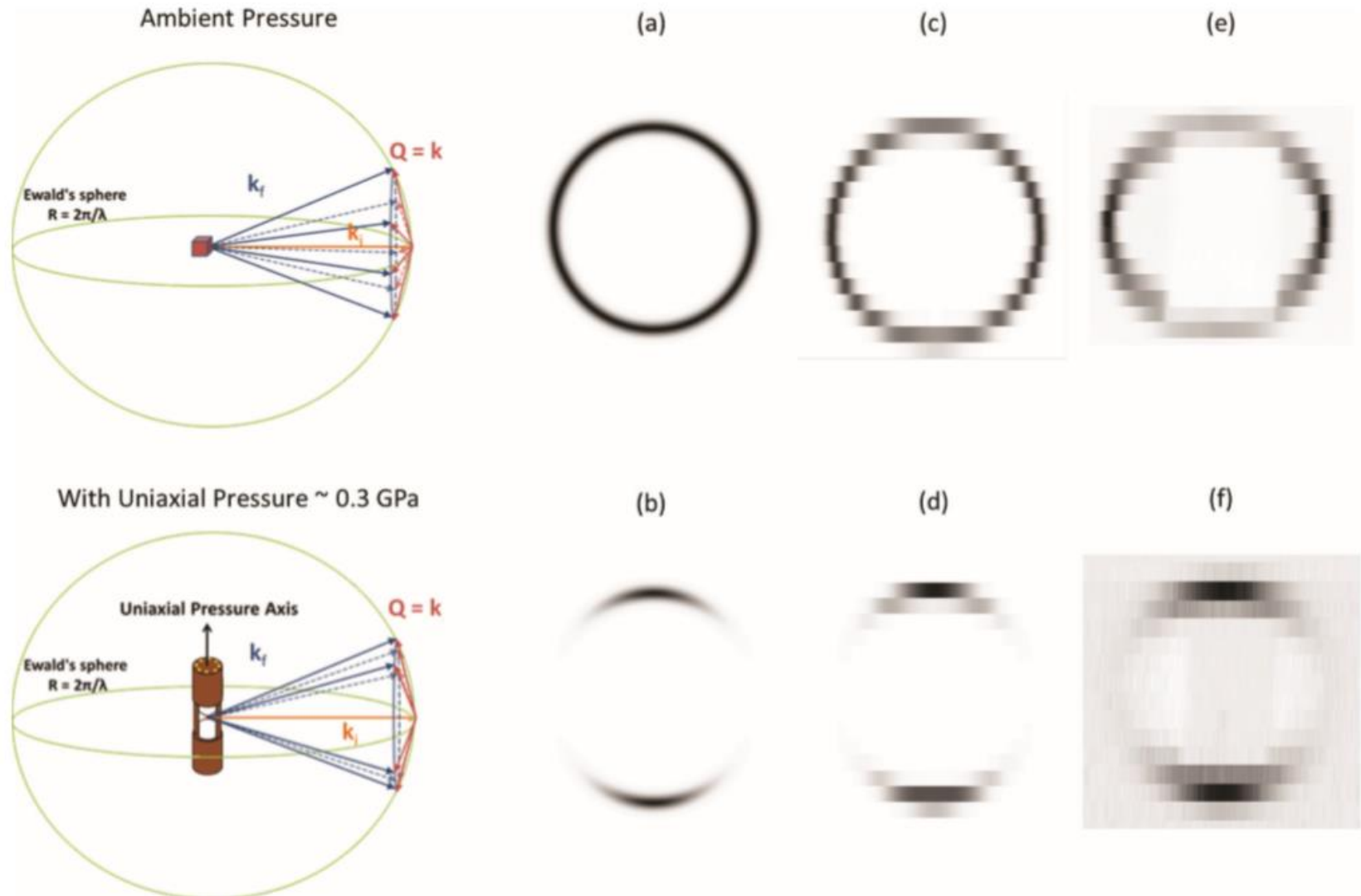


ZnCr₂Se₄; T = 1.5K;



000+- ZnCr₂Se₄, T=1.5K; P=0kbar

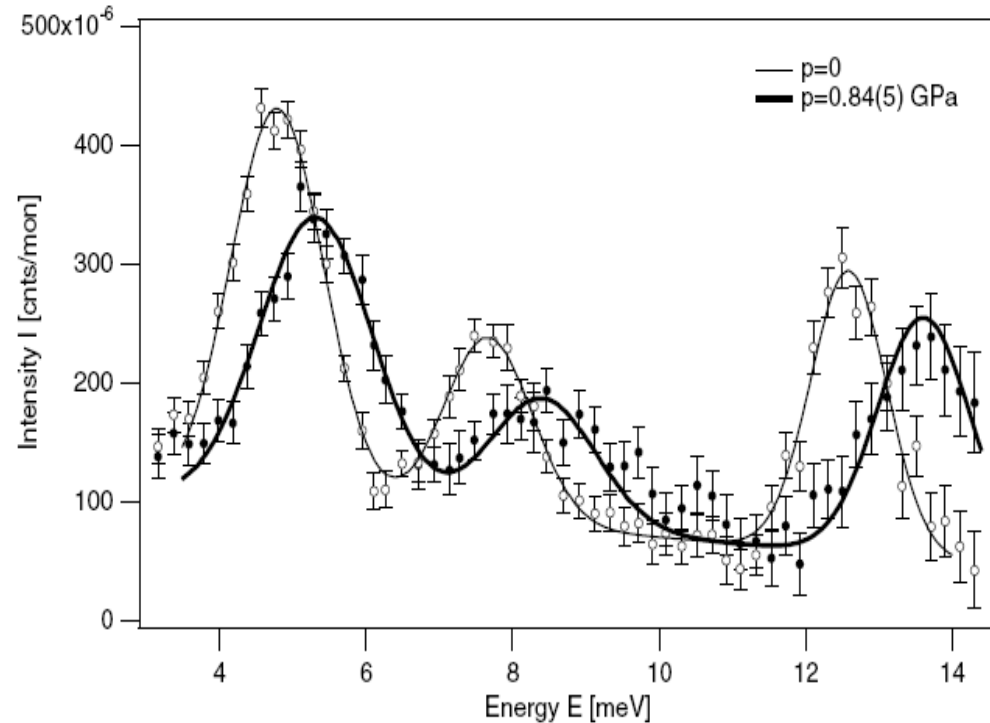
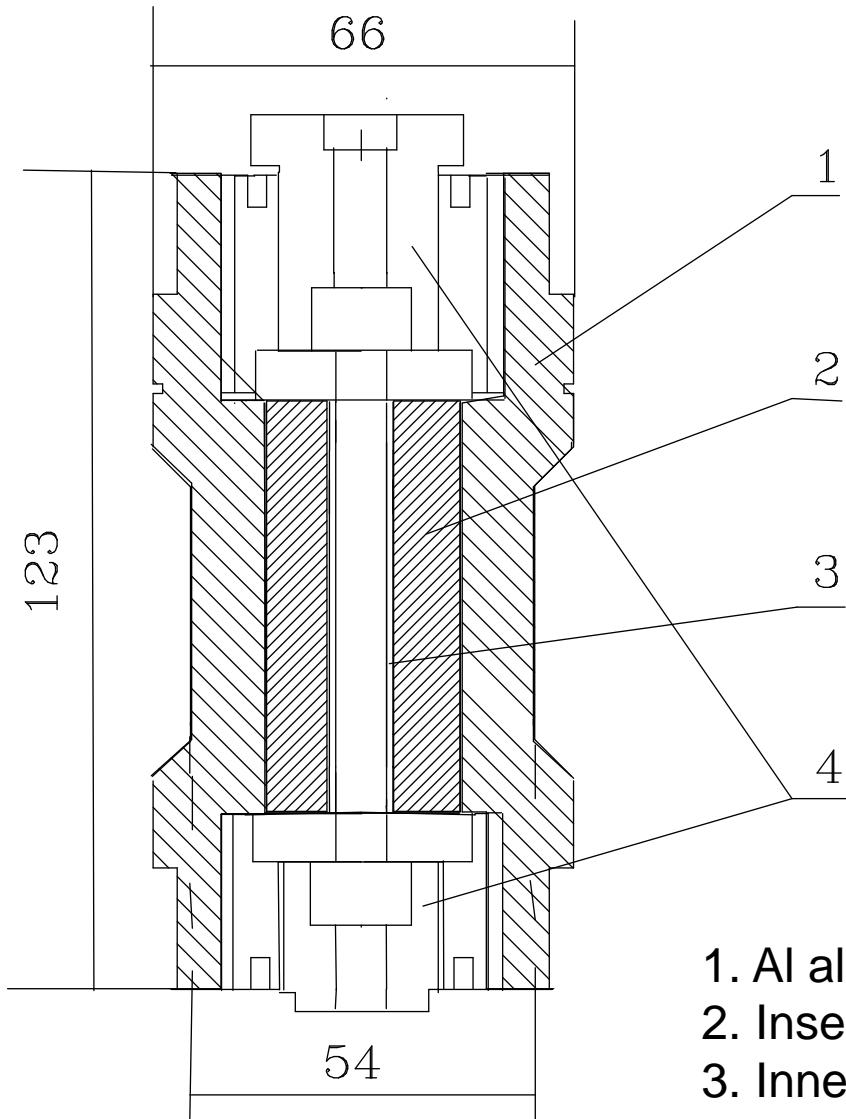




High pressure cells for inelastic neutron scattering studies

Hard Al, Steel and hard HNU (NiCrAl)
alloys

High Pressure Cell (HPC15-AI) up to 15kbar ($T=2-300\text{K}$, $V=1.6\text{cm}^3$) for inelastic scattering neutrons



INS spectra for NdAl_3 at ambient pressure (in the pressure cell) and at $p = 0.84(5)$ GPa ($T = 10$ K).

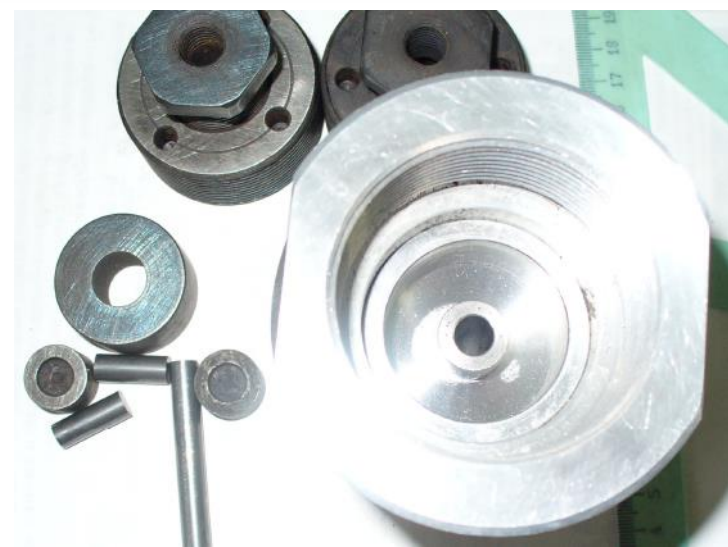
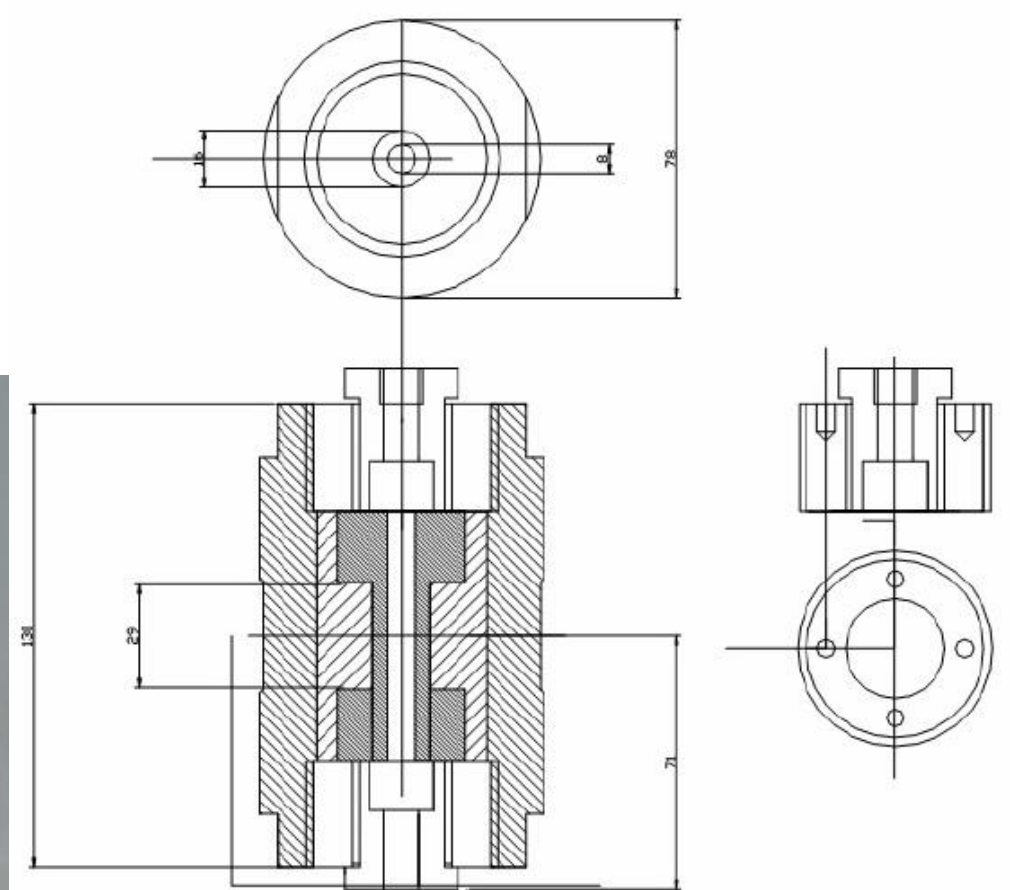
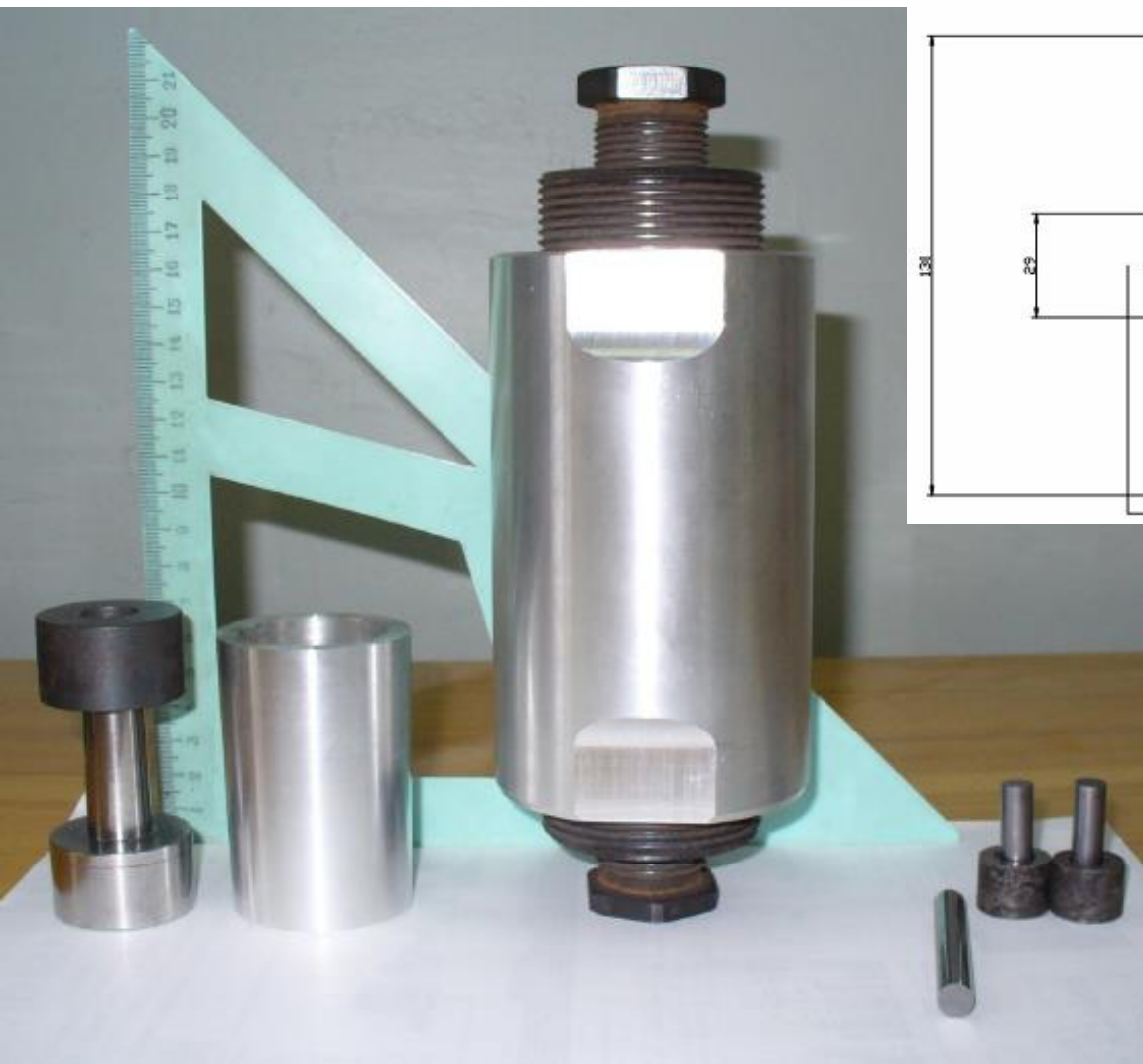
1. Al alloy - B95T
2. Insert part Al alloy- B96T
3. Inner part steel- 45XMHΦA
4. Nuts from steel-45XMHΦA.

Hydrostatic Pressure Cell (HPC15-Al) : $P \leq 1.5 \text{ GPa}$

piston-cylinder clamp cell made of hardened aluminum



HPC17AI(D78/d8)-PSI2006 for single crystal inelastic scattering neutrons



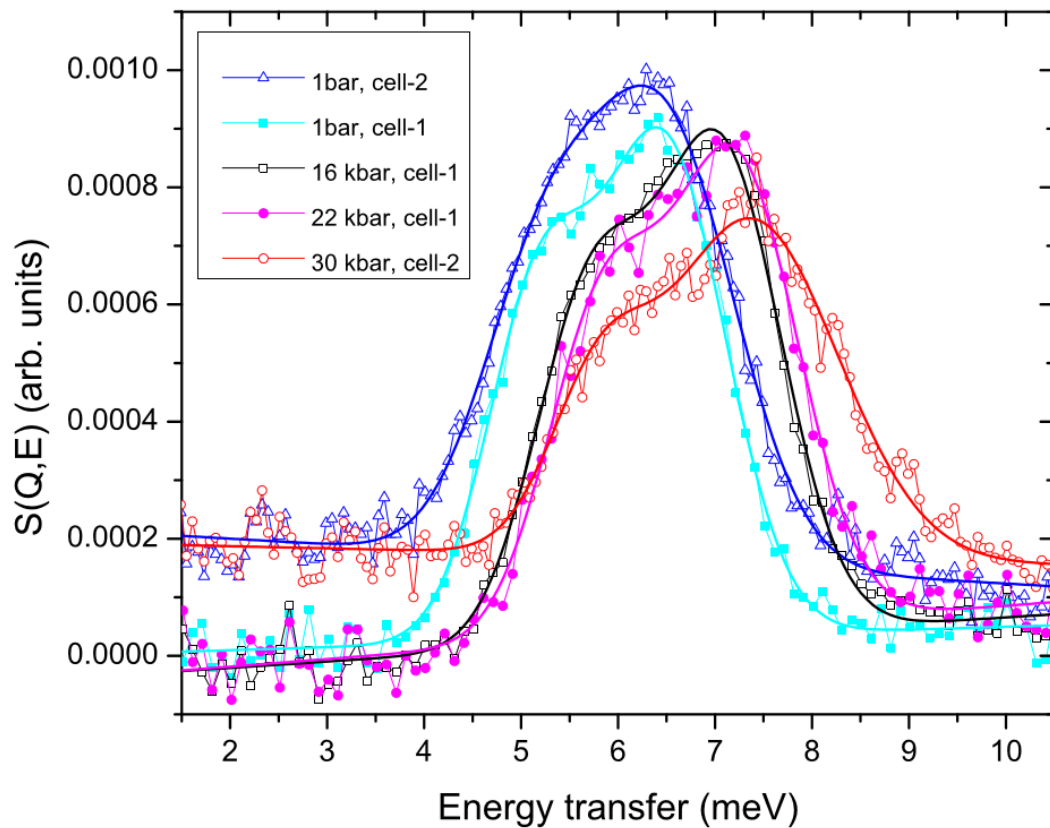
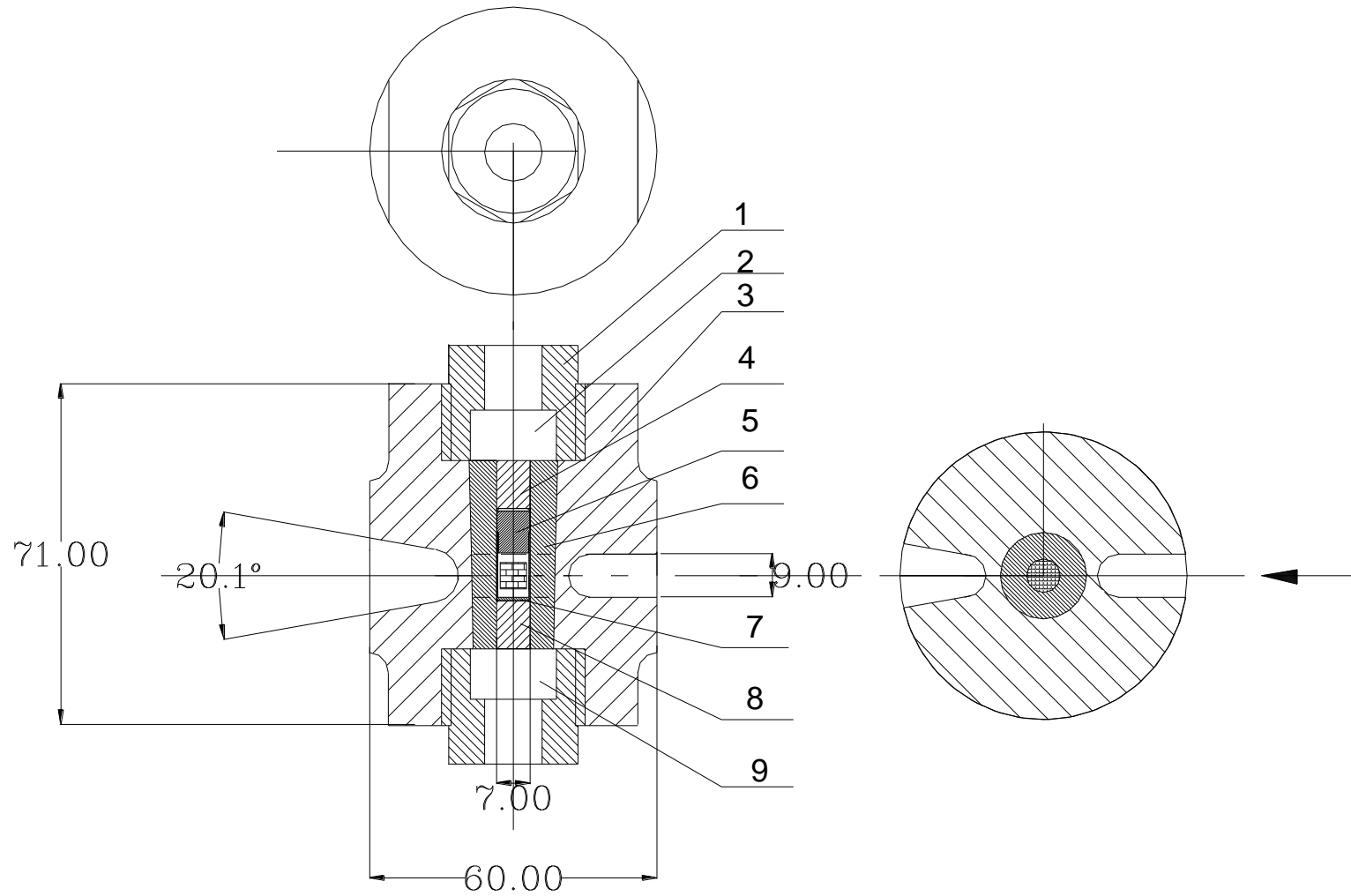


Рис. 18.-INS spectra of α -MnH_{0.07} measured at $T = 1.5$ K and different pressures using the CNCS spectrometer with $E_i = 12$ meV; the background from the high-pressure cell and the cryostat has been subtracted from the data.

1. Pressure effect on hydrogen tunneling and vibrational spectrum in α -Mn.

- A. I. Kolesnikov, A. Podlesnyak, R. A. Sadykov, V. E. Antonov, M. A. Kuzovnikov, G. Ehlers, and G. E. Granroth.
PHYSICAL REVIEW B 94, 134301-5, (2016)

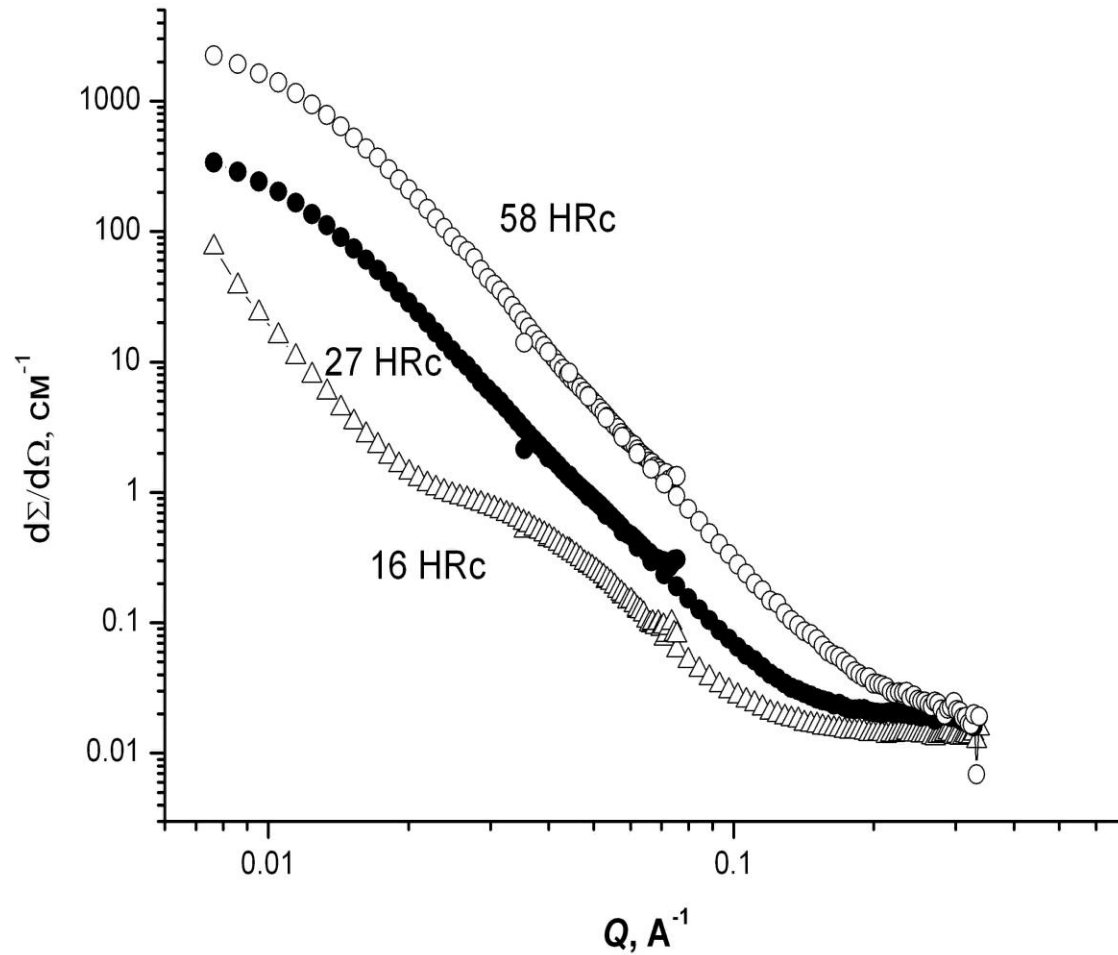
SANS-SPB-PNPI-HPC15, GKSS-Dec.2007 (Germany)



SANS-SPB-PNPI-HPC15, GKSS-Dec.2007 (Germany)



SANS of HNU(NiCrAl)-alloy at different hardness (measured in FRMII).

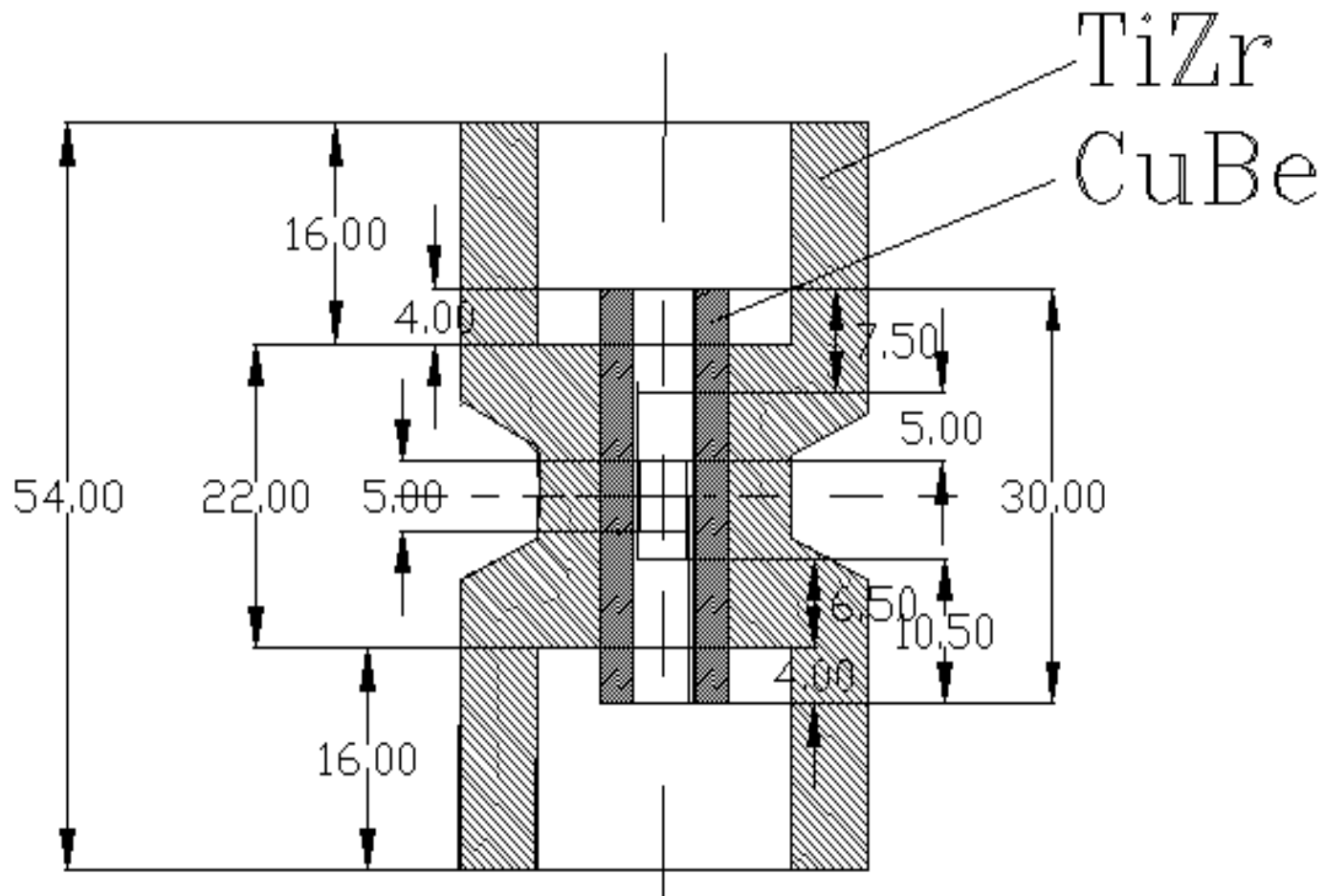


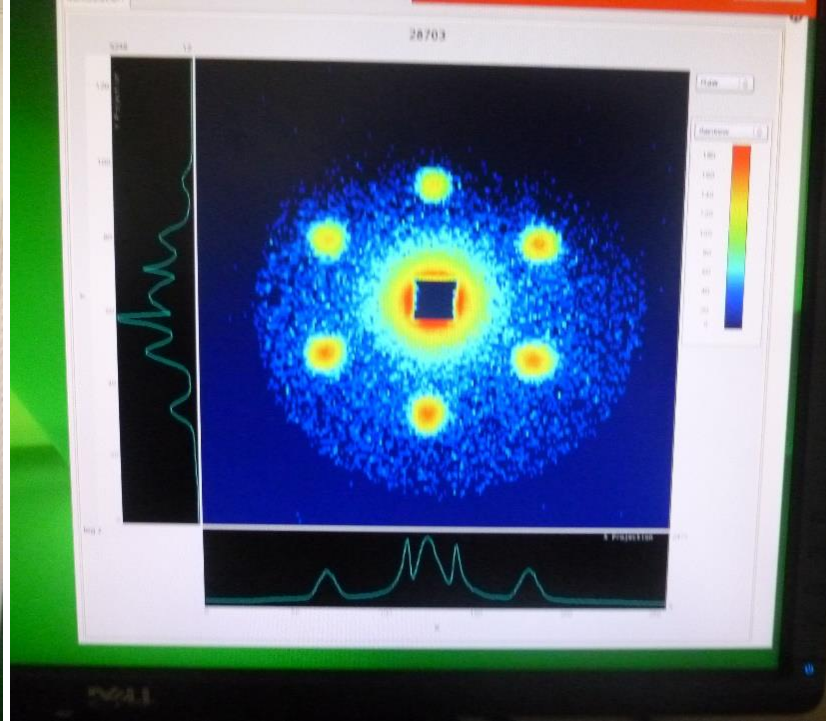
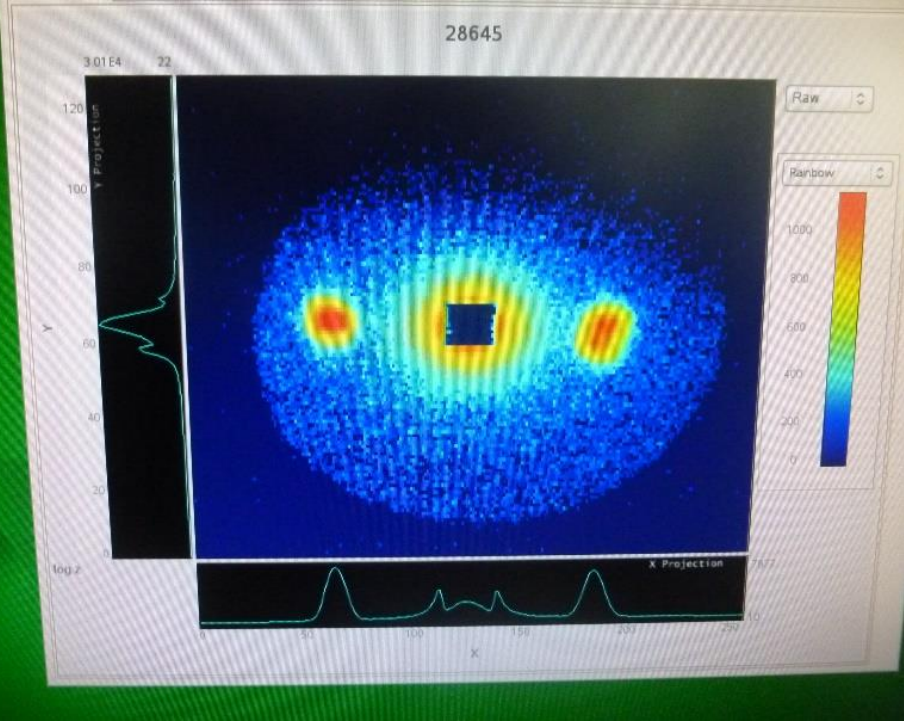
1. HPC-20kbar - TiZr+CuBe for MnSi spin-echo IN15 ILL,
SANS D33,
Sept8-14,2012; May6-23,2013.



2. HPC-20kbar-TiZr+CuBe for MnSi spin-echo IN15 ILL, SANS D33,

Sept8-14,2012; May6-23,2013.

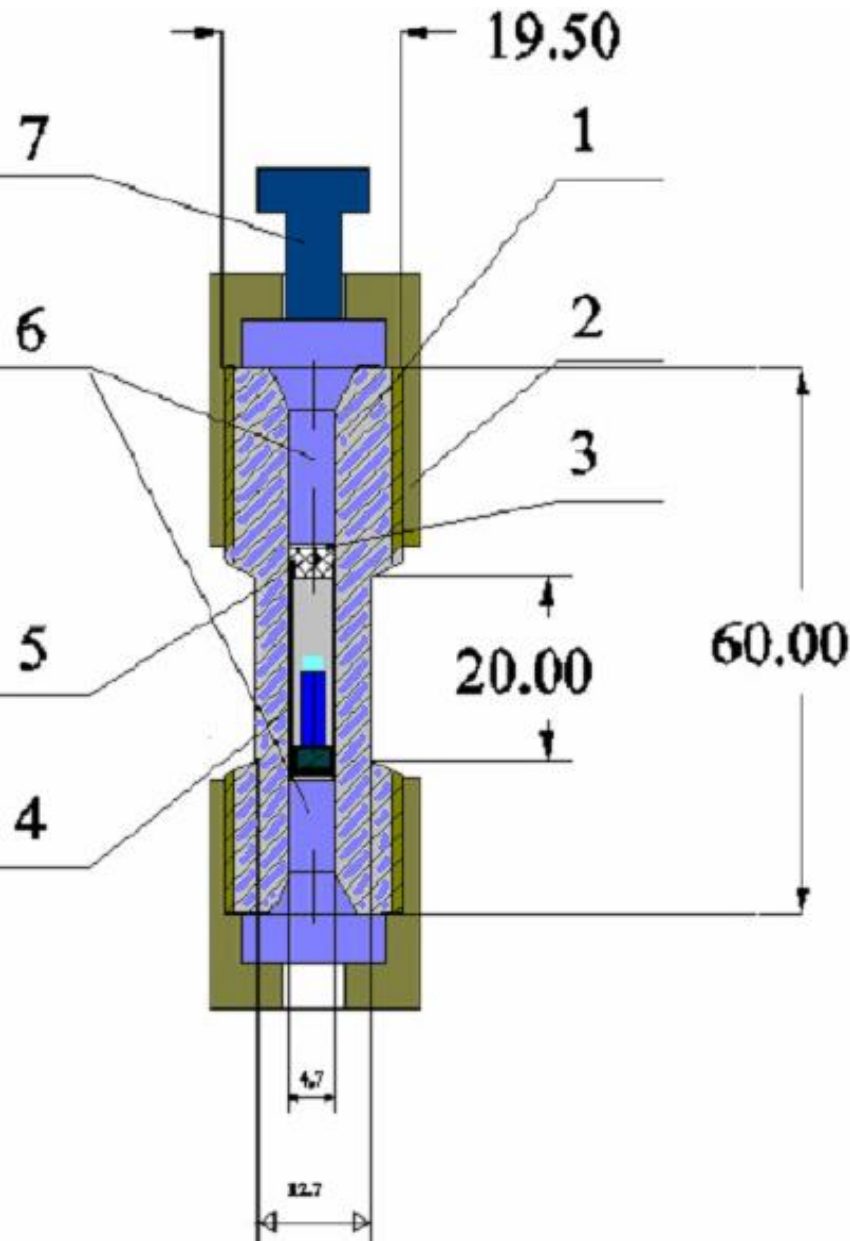




High pressure cells for Single crystal diffraction

TiZr zero alloy, hard Al and hard HNU (NiCrAl)

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

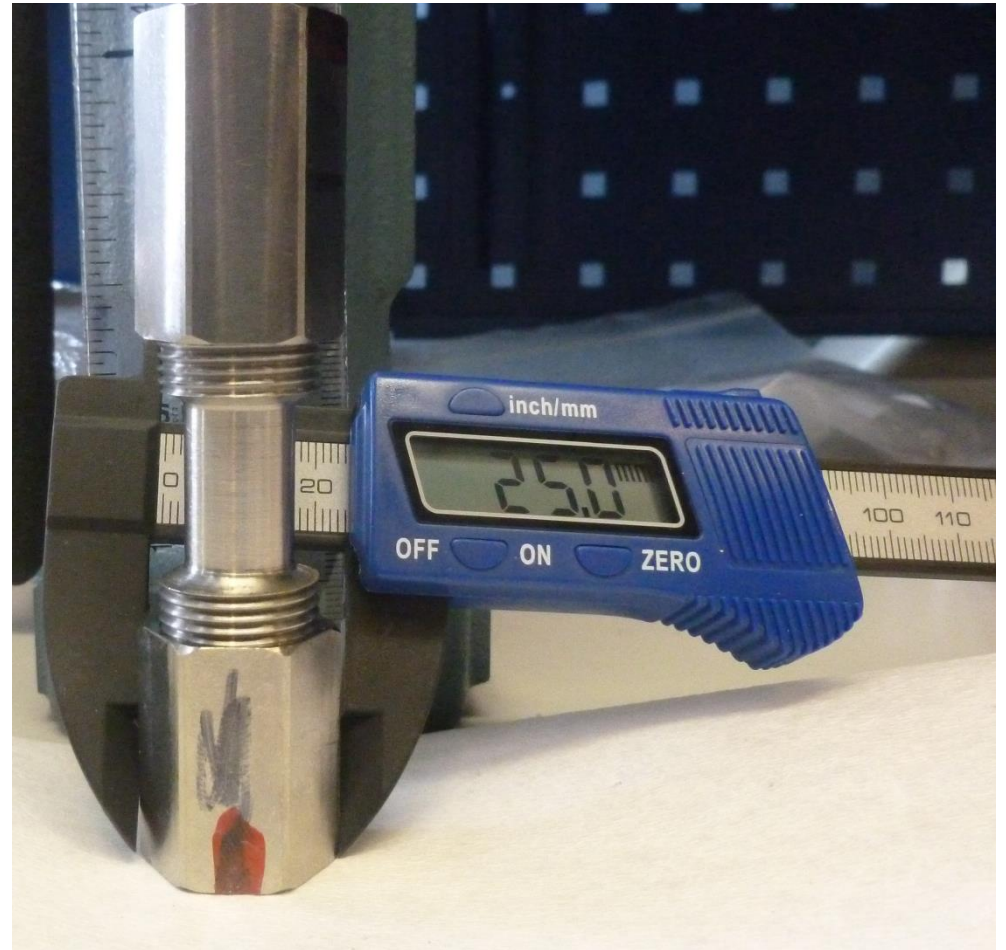


1-cell body-nonmagnetic alloy 40HNU,
2- Nut-Ti alloy,
3-extrusion O-ring-CuBe,
4-sample container-lead(Pb),
5- container caps-Pb,
6-pistons- nonmagnetic alloy 40HNU,
7-pistons for induce pressure.

This is cell nonmagnetic up to low $T=2K$ temperatures and high magnetic fields $H=9,5T$, because we have not changing diffraction patterns from cell.

40HNU=40XH10(NiCrAl)

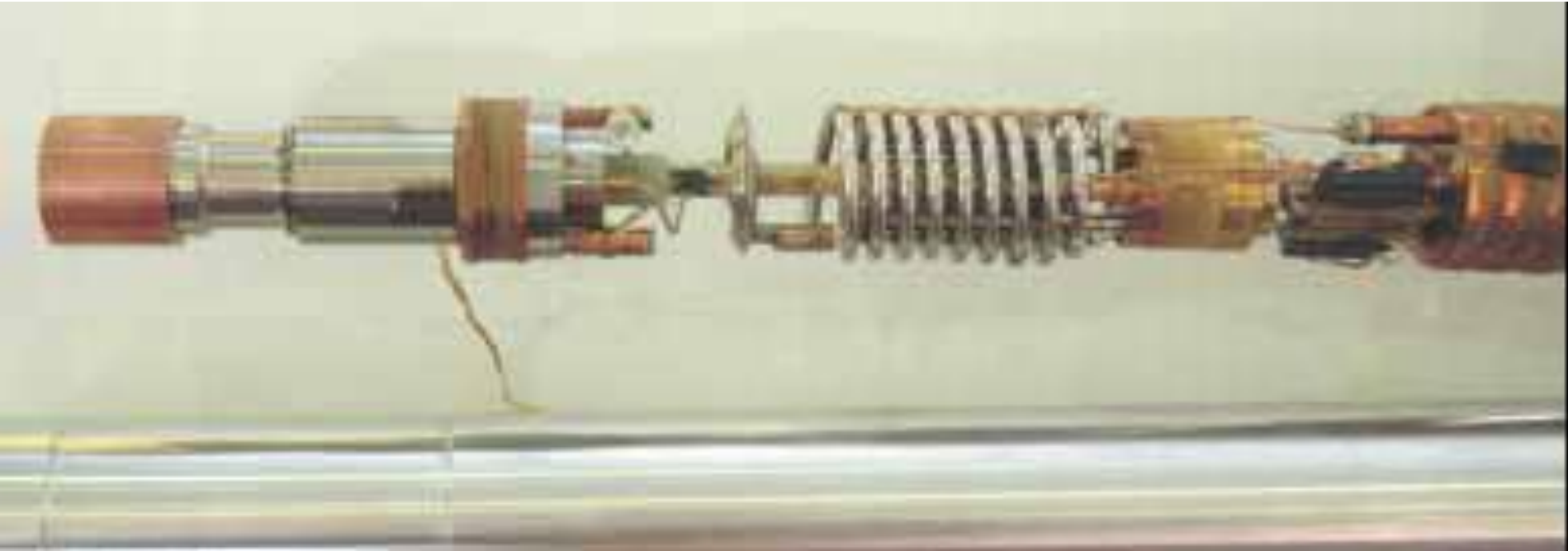
SNS, CNCS, Feb. 2013



Quantum melting in magnetic metals

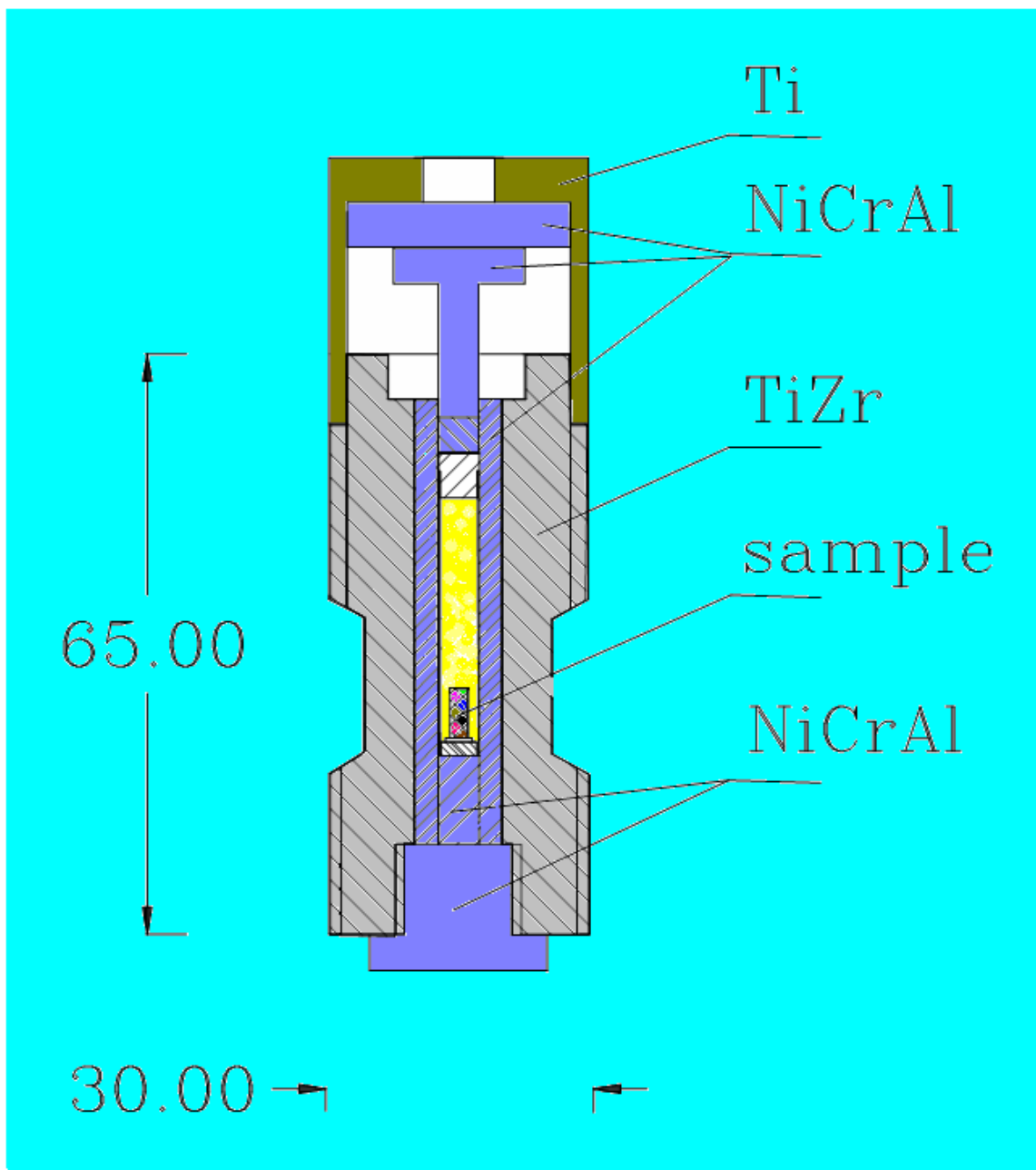
MJ Bull (*ISIS*), SS Saxena (*University of Cambridge*),

RA Sadykov (*Institute for High Pressure Physics, Troitsk, Russia*),
CD Frost (*ISIS*)

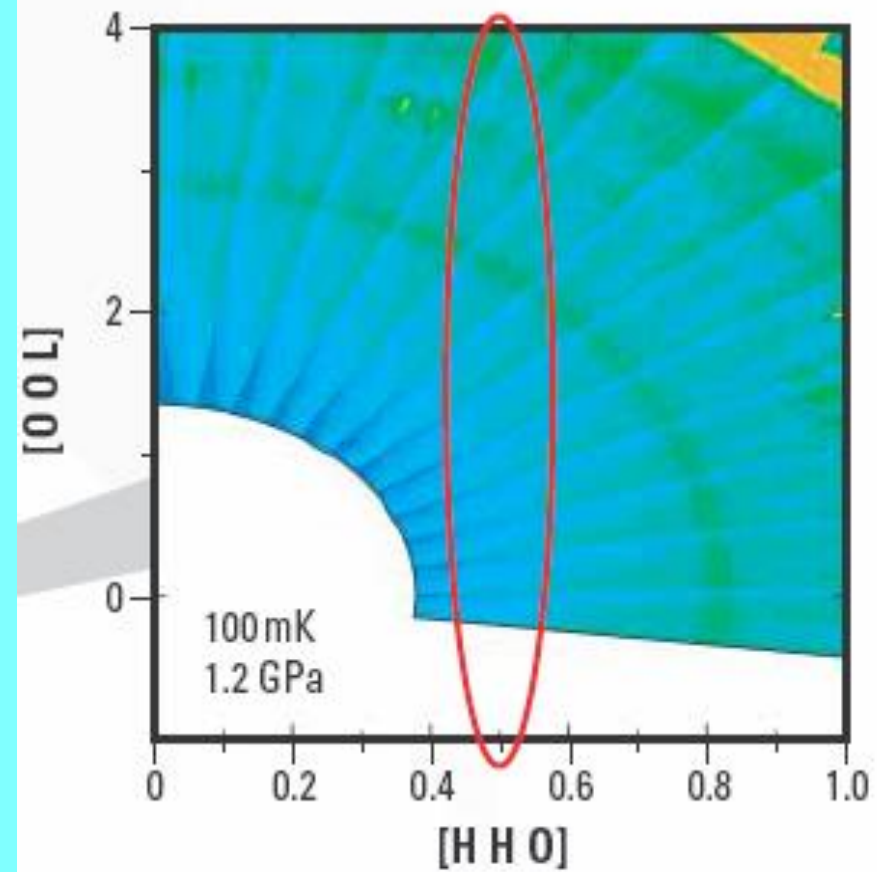
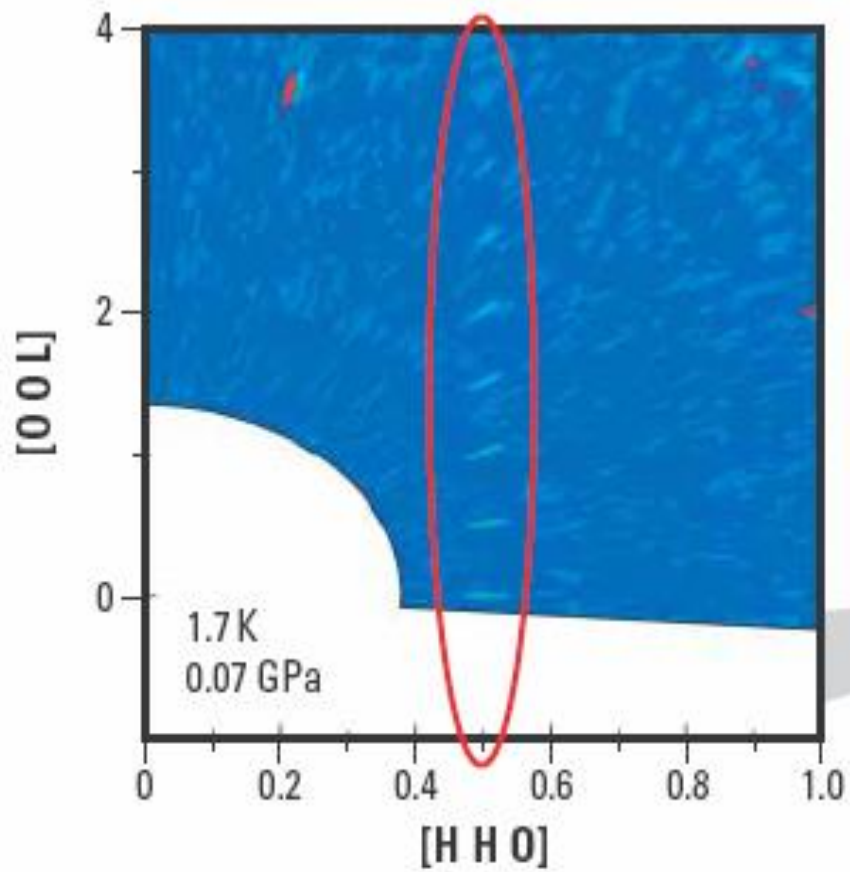


The large bore TiZr + NiCrAl alloys piston cell mounted on the dilution fridge insert. The cell can accept a crystal up to 4,7mm in diameter and operates at pressures up to 2.5GPa at low temperature.

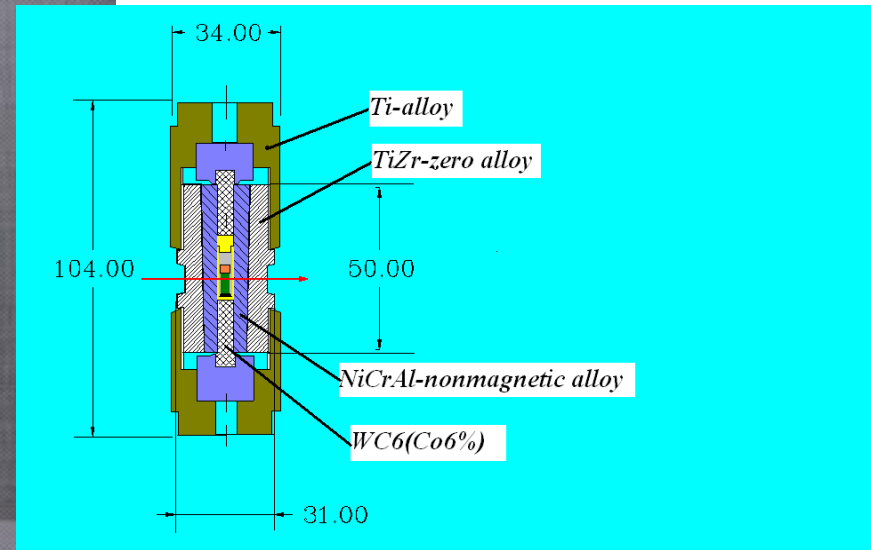
HPC25(100mk) for the *dilution fridge insert*. PRIZMA-ISIS-UK-2001



Quantum melting in magnetic metals (CeRh₂Si₂)



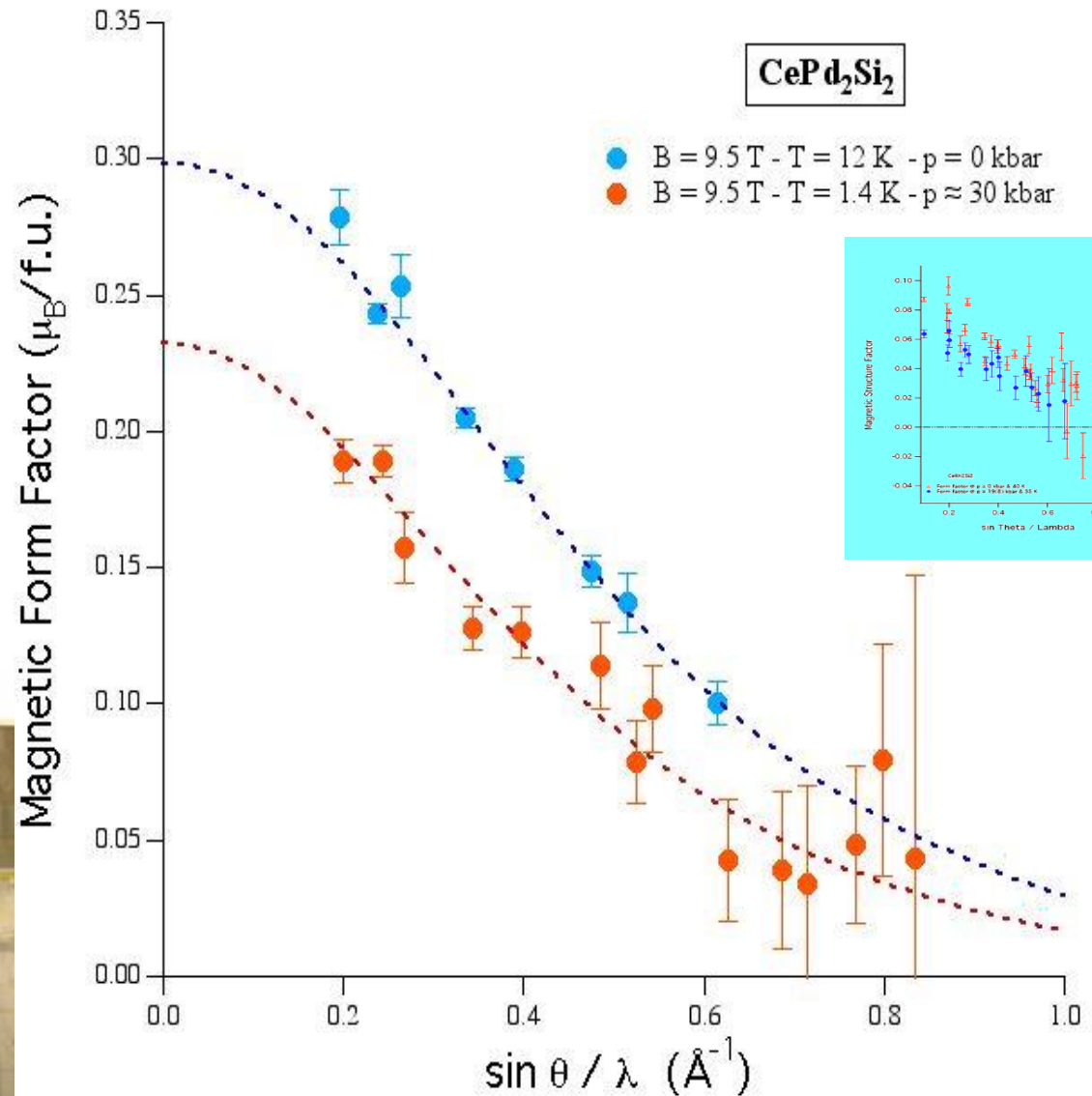
Нейтроннографическая немагнитная камера высокого давления типа поршень-цилиндр до 40кбар



Photographs of the P=40kbar non-magnetic clamp cell designed at the Institute for High Pressure Physics RAS and used in the 10T cryomagnet on the polarized neutron diffractometer D3.

Magnetic form factors measured in the CePd₂Si₂ The Spin Polarised Hot Neutron Beam Facility D3(ILL)

paramagnetic states:
above 10K at P=0 kbar (blue circles) and at the lowest accessible temperature (1.4 K) at p ≈ 30 kbar (red circles). Preliminary refinements within the dipolar approximation are shown as dotted lines.



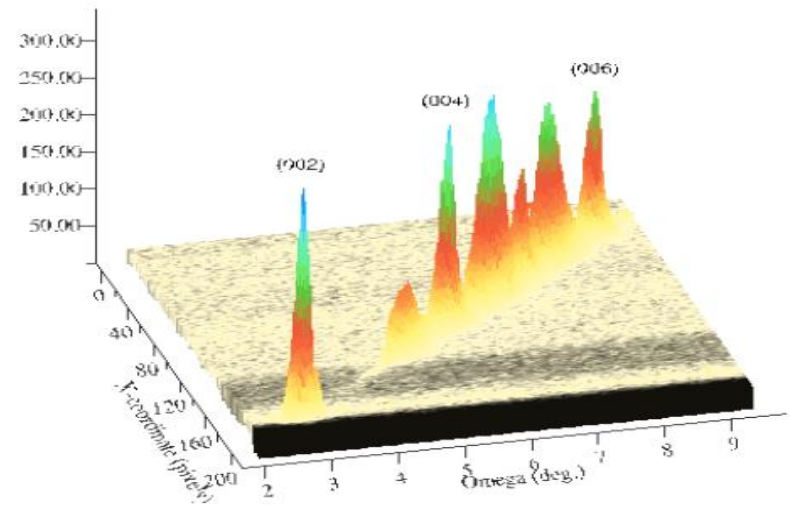
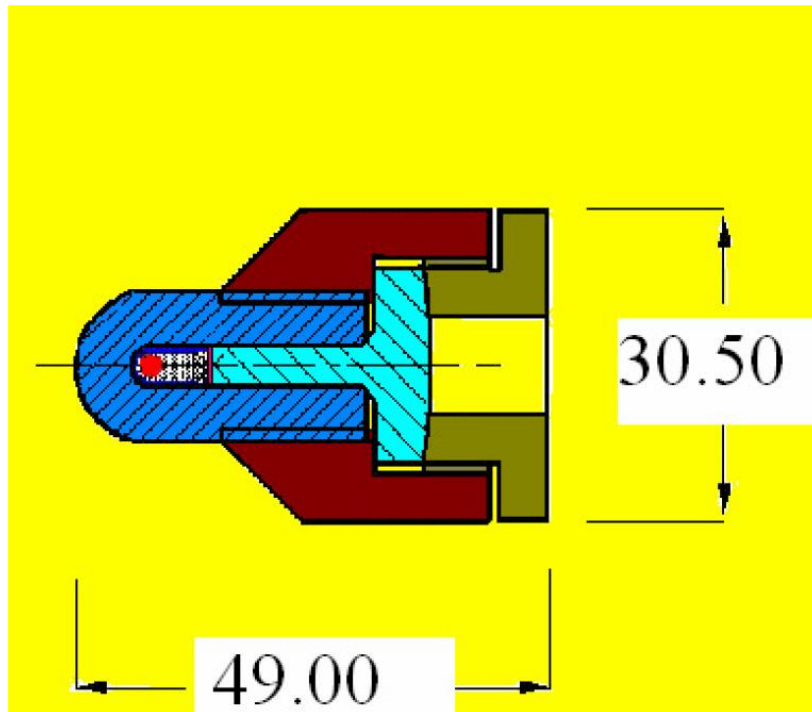
A NEW SINGLE-CRYSTAL PRESSURE CELL FOR TriCS UP TO 3GPa

R.Sadykov¹, D.Sheptyakov², O.Zaharko², Th.Strässle², J.Schefer²

¹Vereshchagin High-Pressure Physics Institute RAS, 142092 Troitsk, Moscow region, Russia

²Laboratory for Neutron Scattering, ETH Zurich & PSI Villigen, CH-5232, Switzerland

Ba-hexaferrite $\text{BaFe}_{8.8}\text{Co}_{1.6}\text{Ti}_{1.6}\text{O}_{19}$

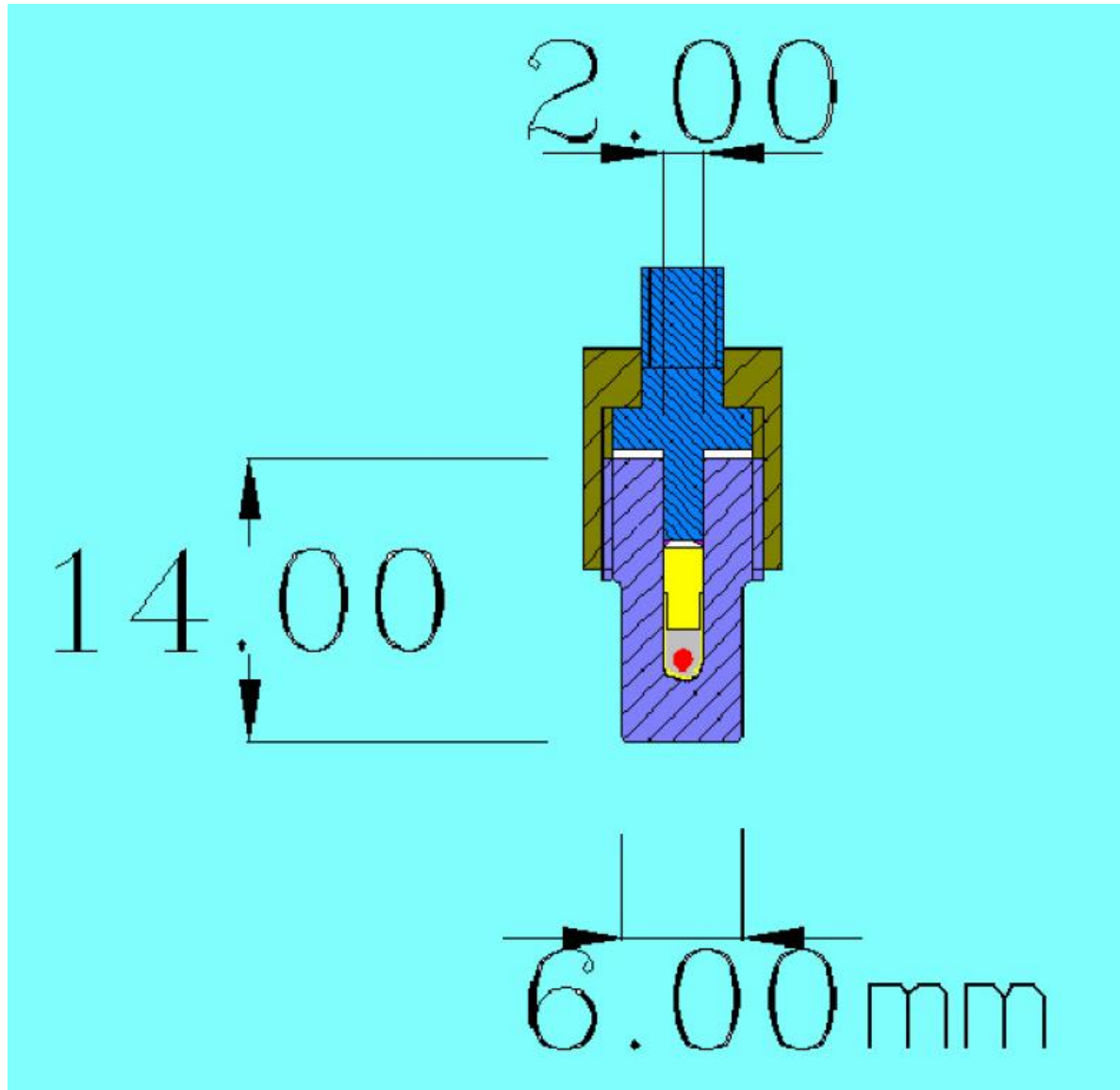


Bragg peaks and magnetic satellites from the sample measured with the 2D detector at $p=3\text{GPa}$, $T=17\text{K}$ (scans in omega presented as a sequence of 1D projection onto one of the detector axes). (sample from: R.Sadykov et al., *Sov.Phys.Solid State* 23, 1865 (1981))

SNS-Jan-2015



hpc20(D10+VIVALDI) - ILL-2006,
volume for sample $h=5\text{mm}$, $d=2\text{mm}$.



Min 42

Max 3276

xf=1632, yf=815

Overlay on

Bg inc.

Contrast



Colour Black on white

Mag x4

PS

Zoom

T = 2 K



Min 600

Max 3276

xf=1612, yf=1013

Overlay on

Bg inc.

Contrast

Colour Black on white

Mag x4

PS

Zoom

