

Типы камер высокого давления для исследования конденсированных сред методом INS

(Nonmagnetic high pressure clamp cells for studies condensed matter by INS)

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A number of the high pressure **clamp cells** that was made from nonmagnetic **TiZr** zero alloy, hard **Al** and hard HfU (**NiCrAl**) **alloys** are presented.

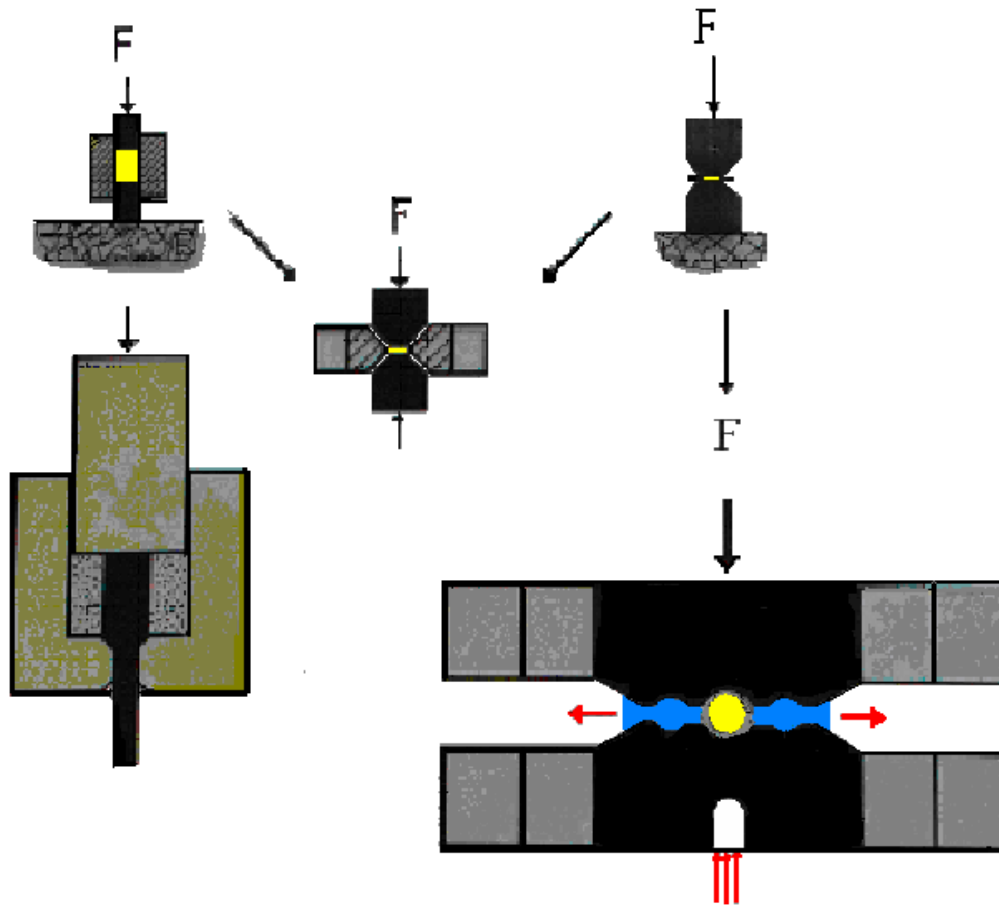
All this is cells as for powder and single crystal as for diffraction and inelastic neutron scattering studies **possible putting in the typical cryostats** (on the dilution fridge insert) and high magnetic fields cryostat up to 6-10T.

The a new **nonmagnetic** composite piston-cylinder type cells was made from TiZr+HfU up to **4GPa**.

Single crystal or powder **NaCl** (for a pressure calibration) and **Fluorinert** (a pressure medium) were used in all experiments on the neutron sources.

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)



Nonmagnetic Materials for Neutron scattering high pressure cells and its limits of the pressure

Alloys-Polycrystalline

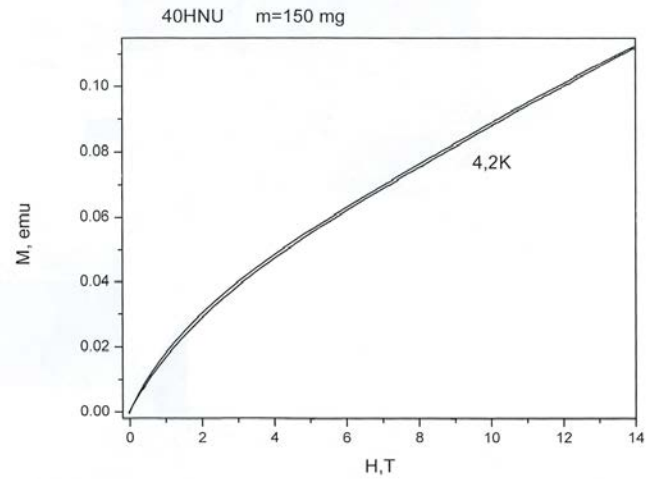
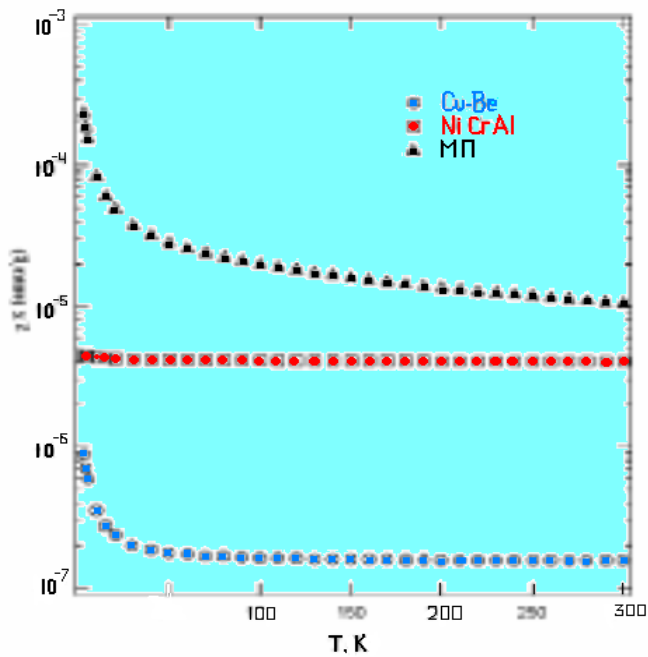
1. Al-alloy (8kbar, with support up to 20 kbar)
2. CuBe-alloy (17-21 kbar)
3. TiZr-zero alloy (10-12 kbar, with support up to 29 kbar at LT)
4. NiCrAl alloy (with support up to 40 kbar at LT)

Ceramics-Polycrystalline

5. Alumina Al_2O_3 (with support up to 27 kbar)
6. Sintering BNmet (anvils up to 150 kbar-d=3mm)
7. Sintering diamonds (anvils up to ? kbar)

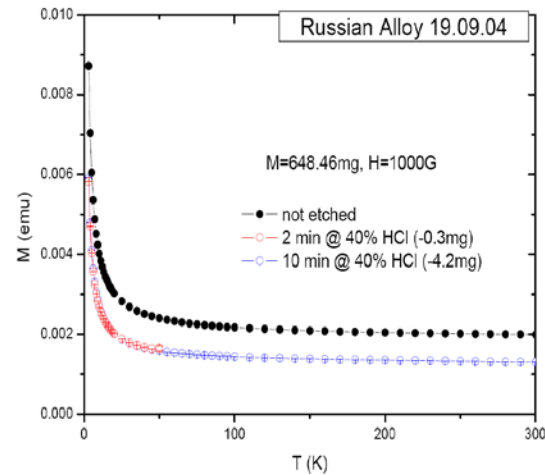
Single crystals:

1. Diamond (anvils up to 1000 kbar)
2. SiC- (anvils up to 200 kbar) муассанит
3. Sapphire (anvils up to 90 kbar) Al_2O_3



Dependence of magnetization on the high magnetic field

Temperature dependent susceptibility of MP35N alloy, Ni-Cr-Al (40HNU=40XHIO) alloy and Cu-Be alloy.



Dependence of magnetization on the temperature measured in the SQUID.

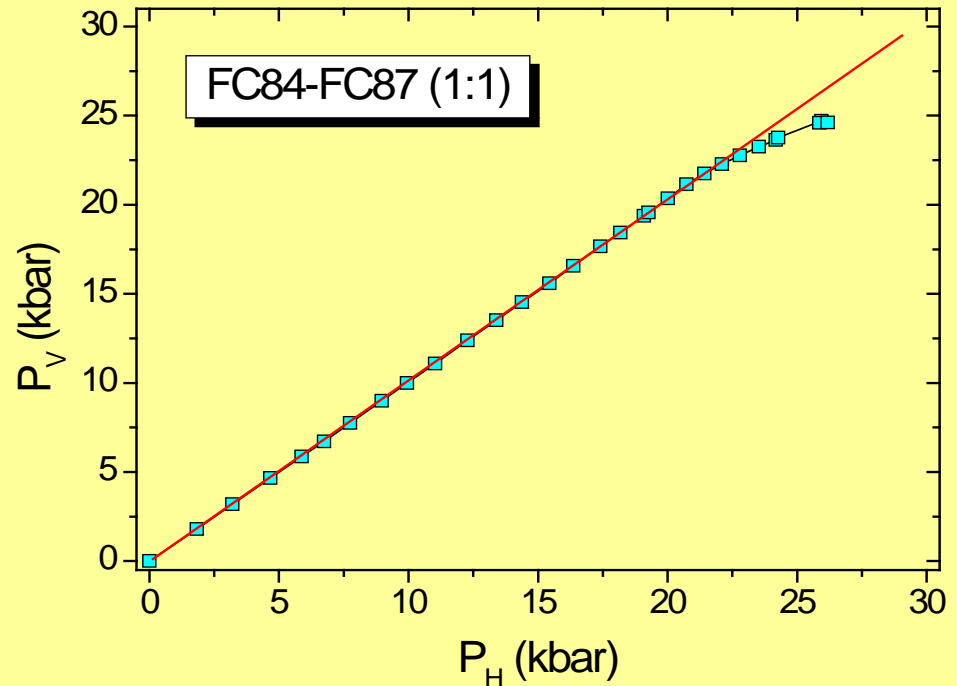
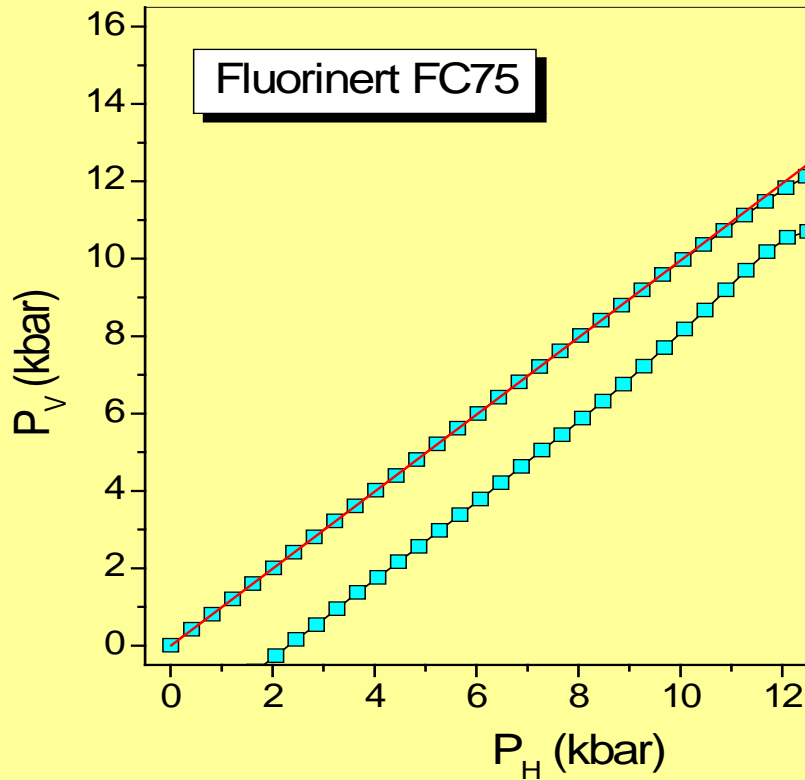
Hydrostatic limits of Fluorinert liquids used for neutron and transport studies at high pressure,

V A Sidorov and R A Sadykov,
J. Phys.: Condens. Matter 17 (2005) S3005–S3008.

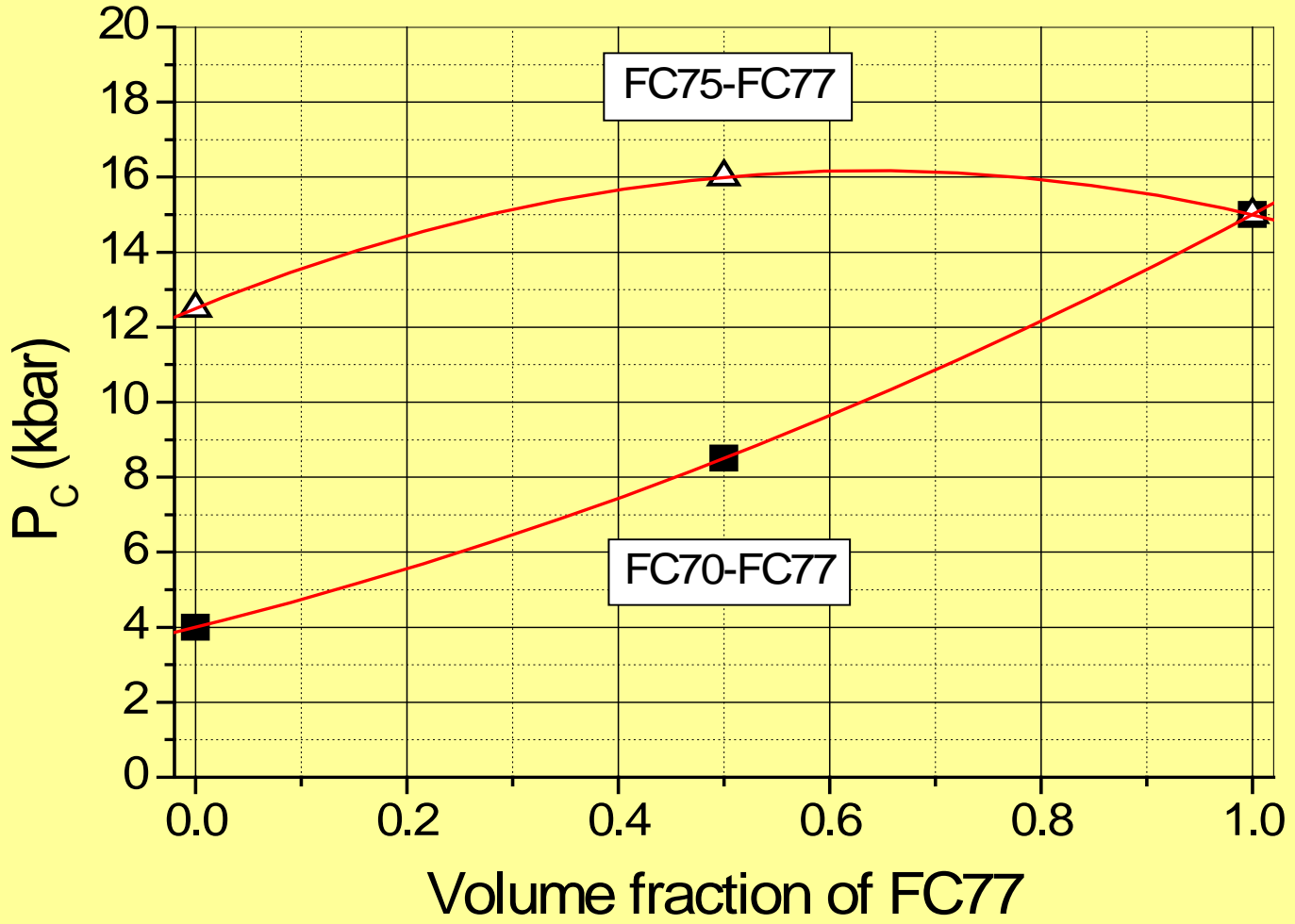
Fluorinert liquids are the analogues of hydrocarbons in which hydrogen is substituted for fluorine. These liquids are widely used for neutron studies at high pressure as a pressure transmitting media, since they exhibit small incoherent scattering of neutrons.

- 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.

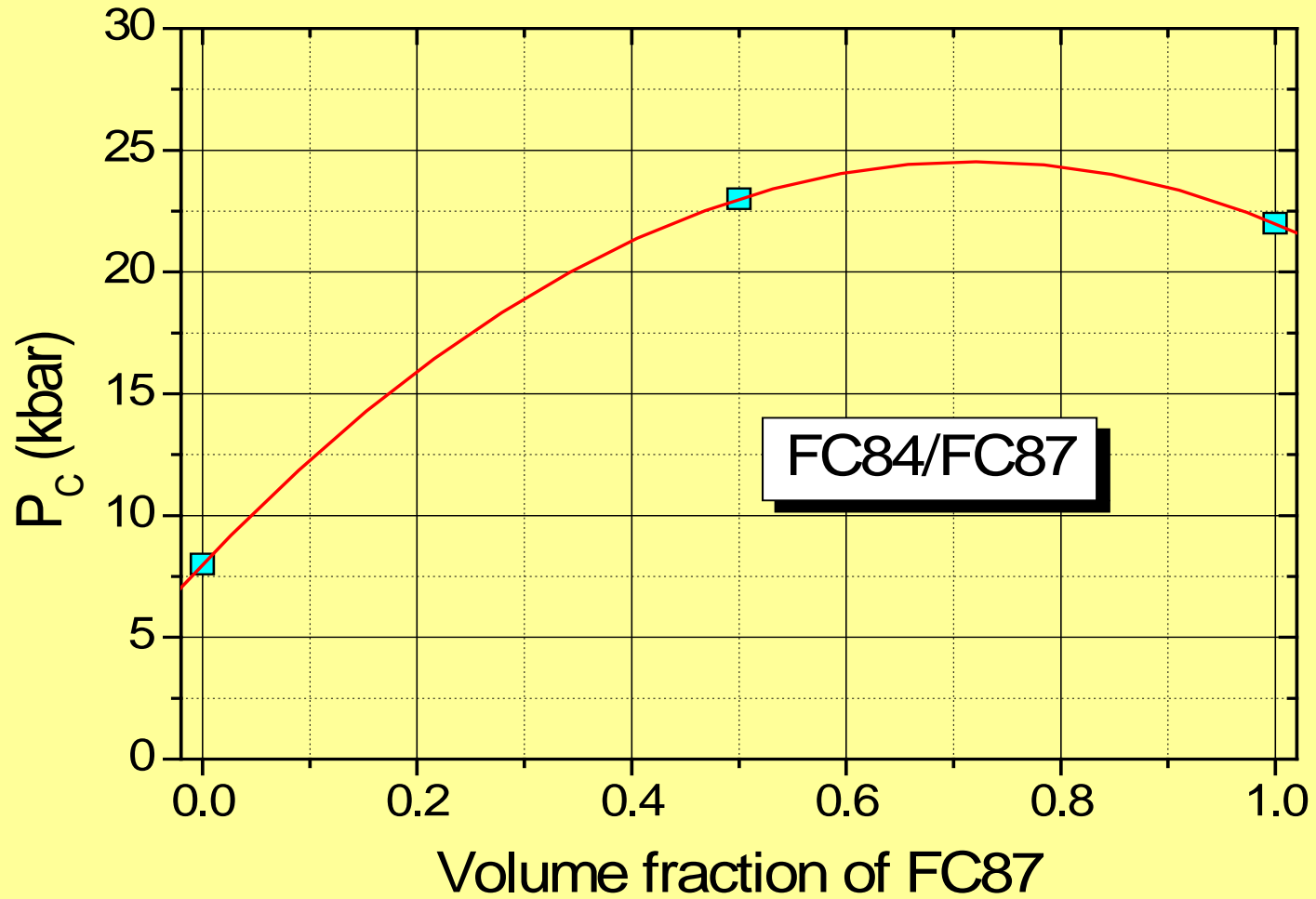
Typical experimental scans of vertical vs horizontal manganin pressure sensor readings for FC75 и FC84-FC87 (1:1). Solidification of liquid takes place in the point, where the the relation $P_H=P_V$ is no more valid.



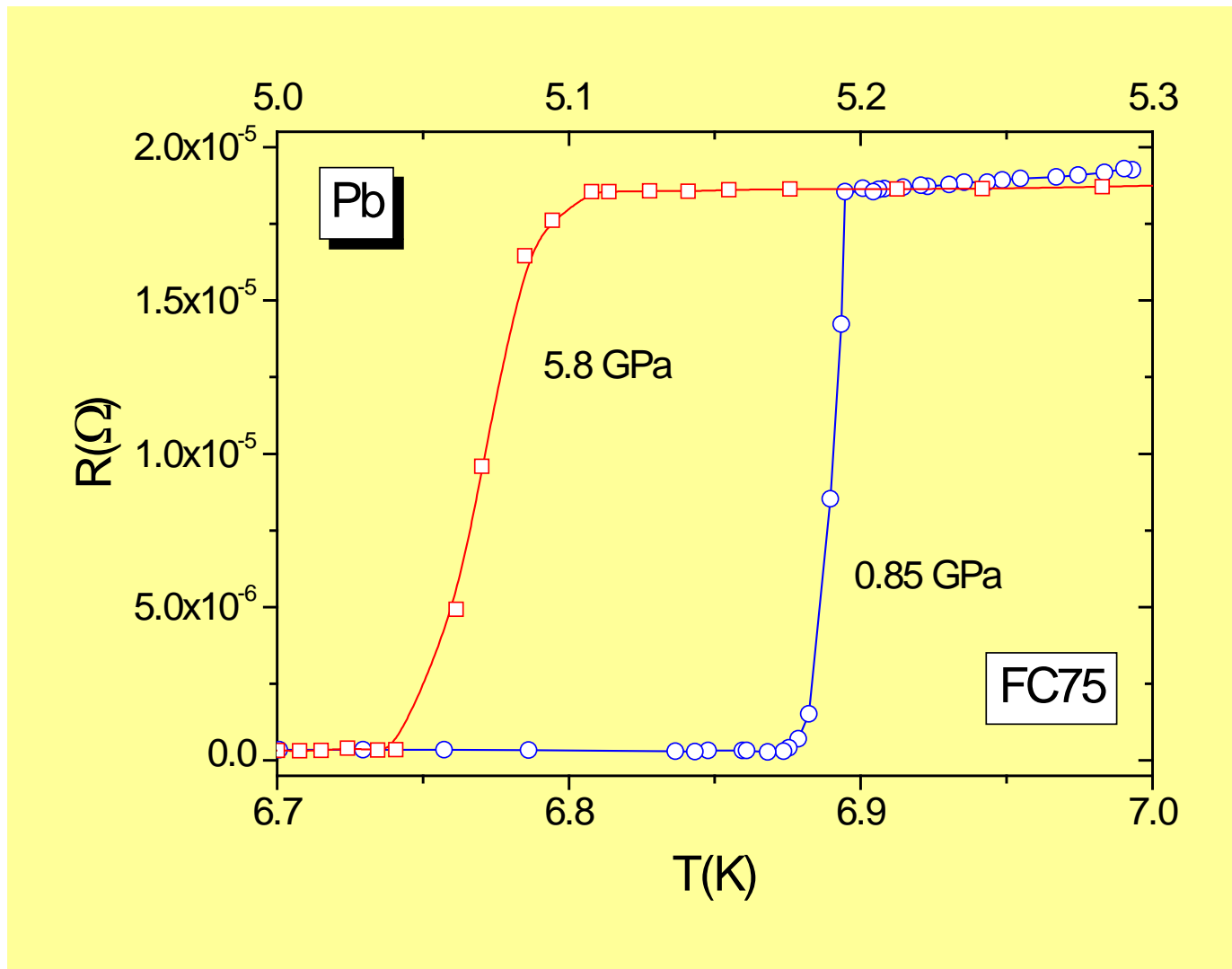
Hydrostatic limits for Fluorinert FC70-FC77 and FC75-FC77



Hydrostatic limits for Fluorinert FC84-FC87

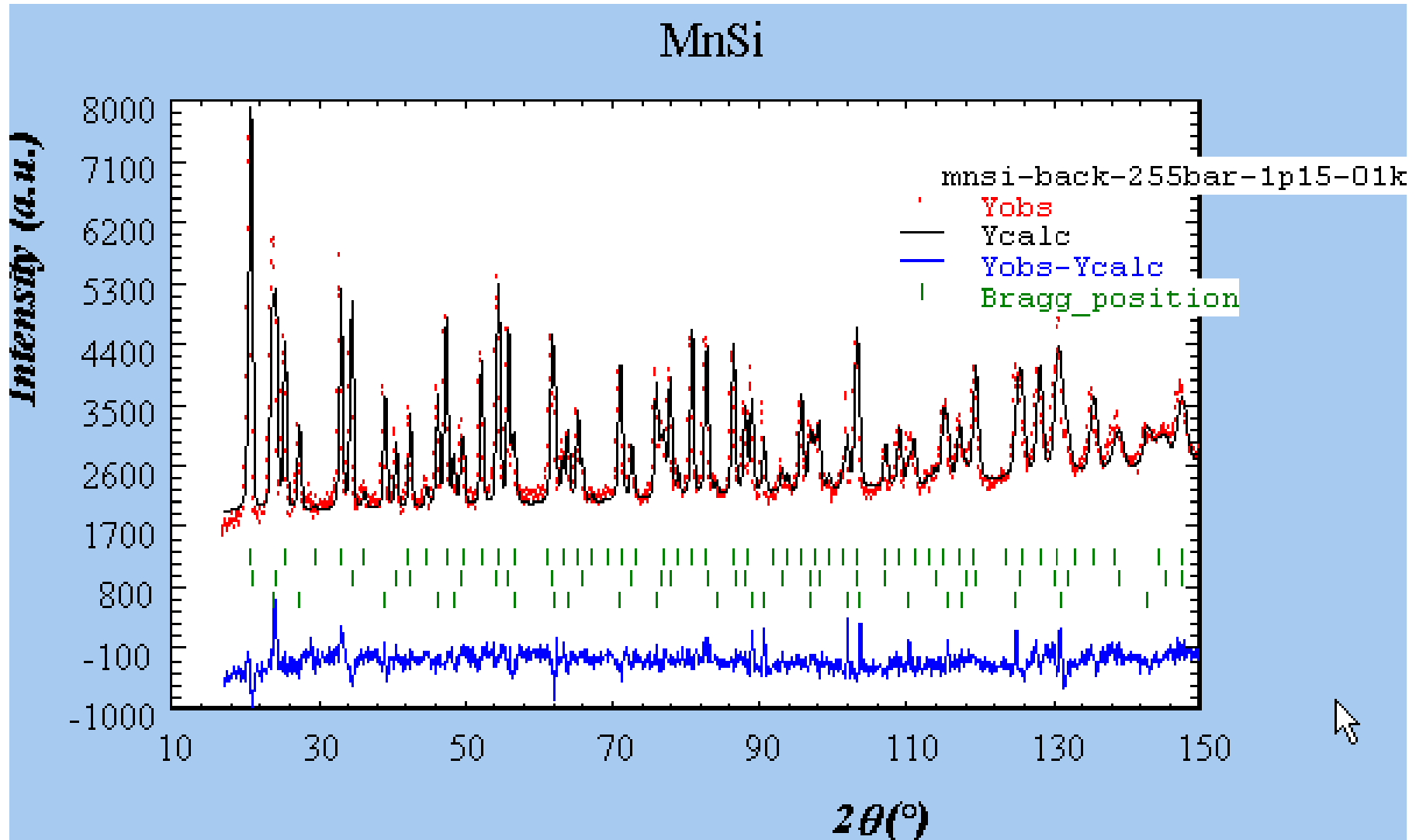


Температурные зависимости электросопротивления свинцового образца в области сверхпроводящего перехода при различных давлениях. Среда, передающая давление – Fluorinert FC75.

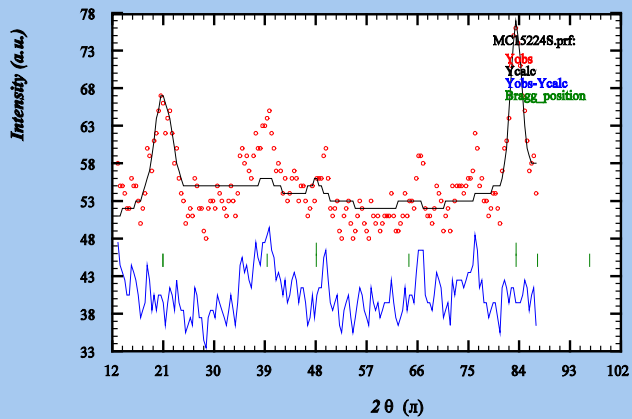


Neutron diffraction pattern of powder MnSi

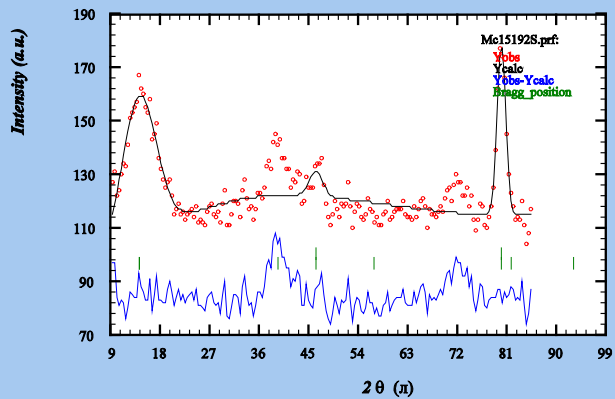
at 14,2kbar and 1.5K.



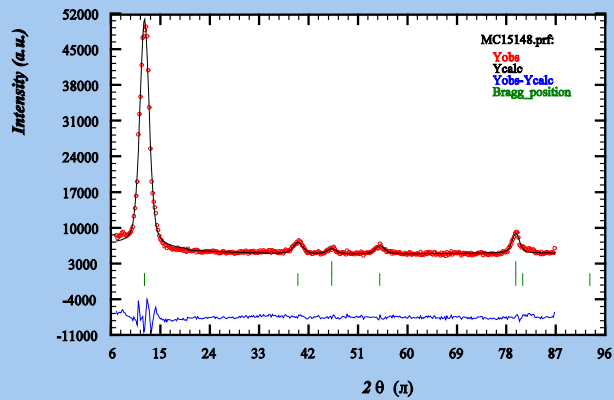
ZnCr₂Se₄; T = 1.5K;



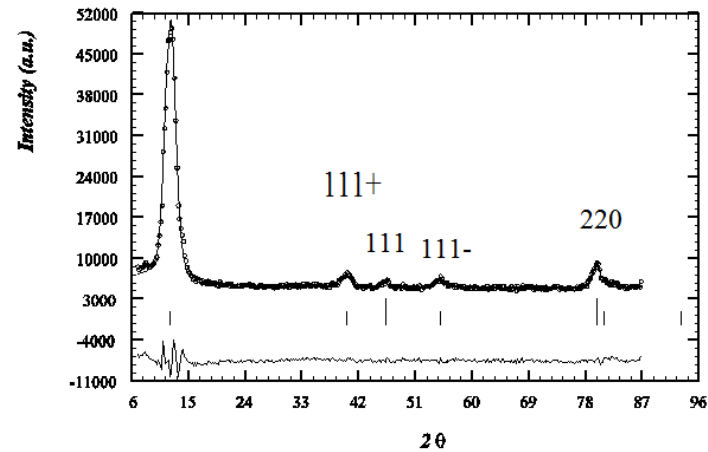
ZnCr₂Se₄; T = 1.5K;

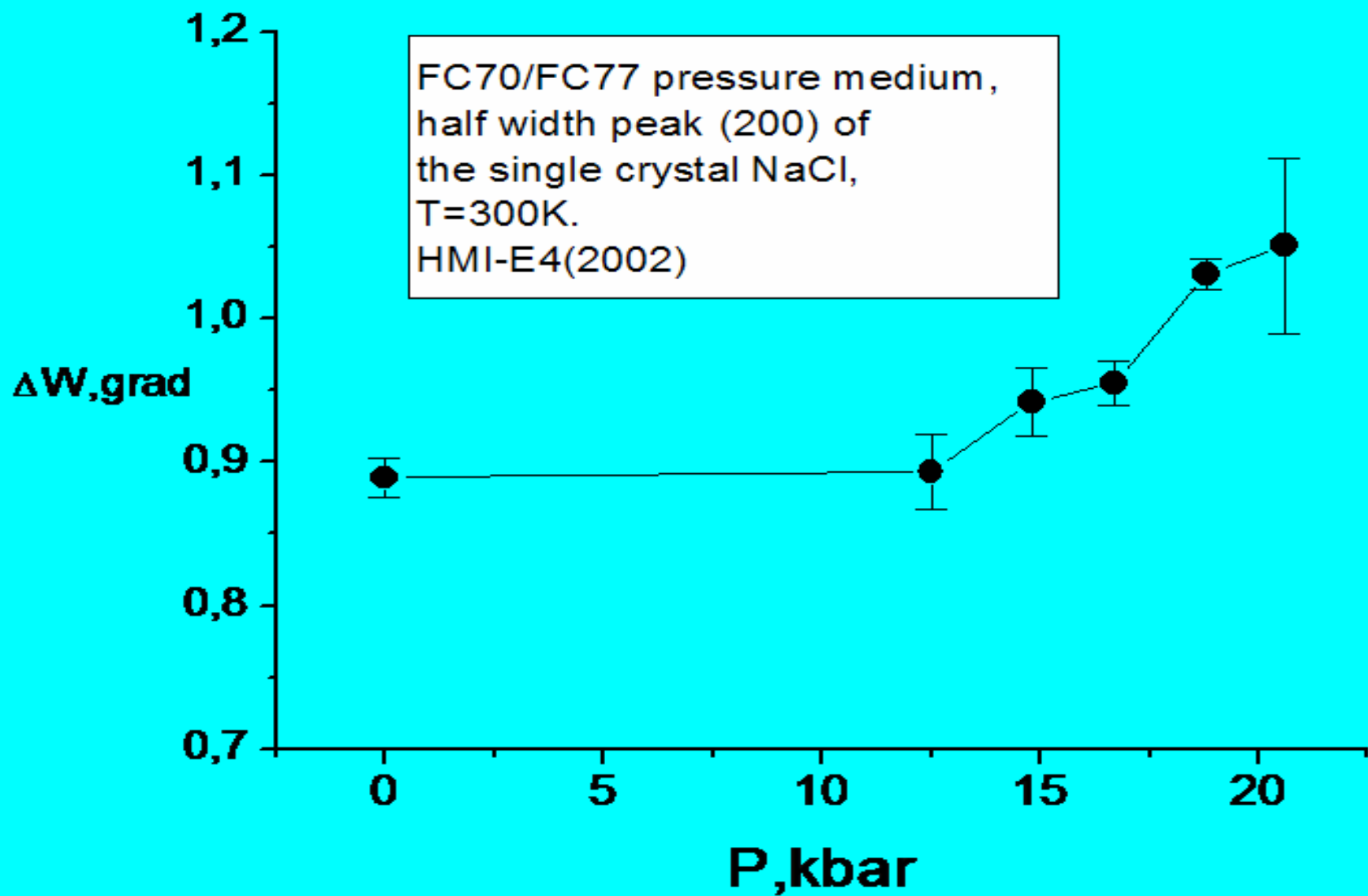


ZnCr₂Se₄; T = 1.5K;

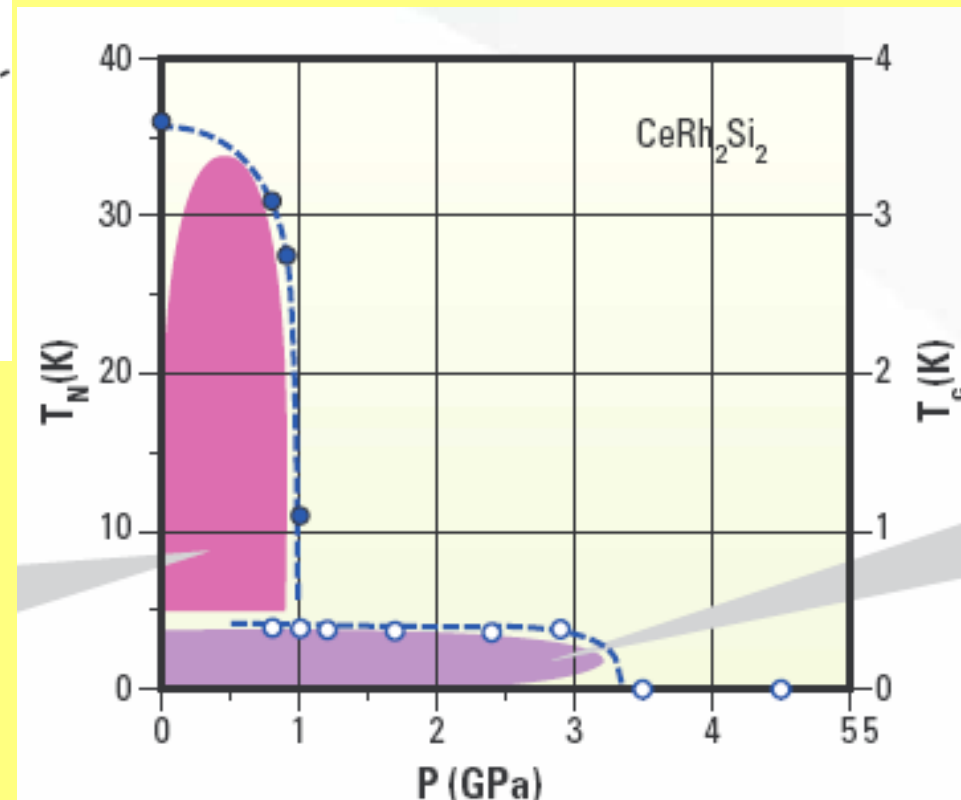
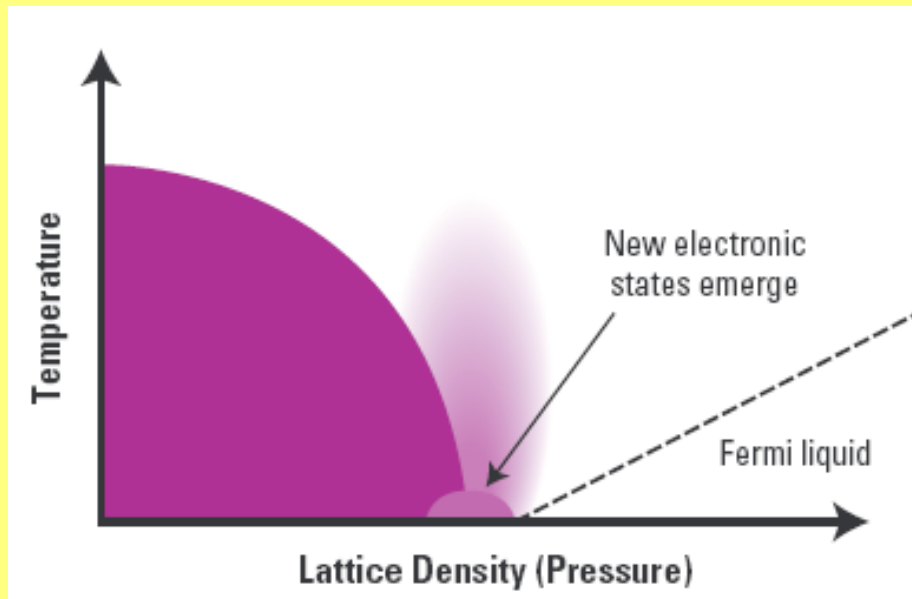


000+ ZnCr₂Se₄, T=1.5K; P=0kbar

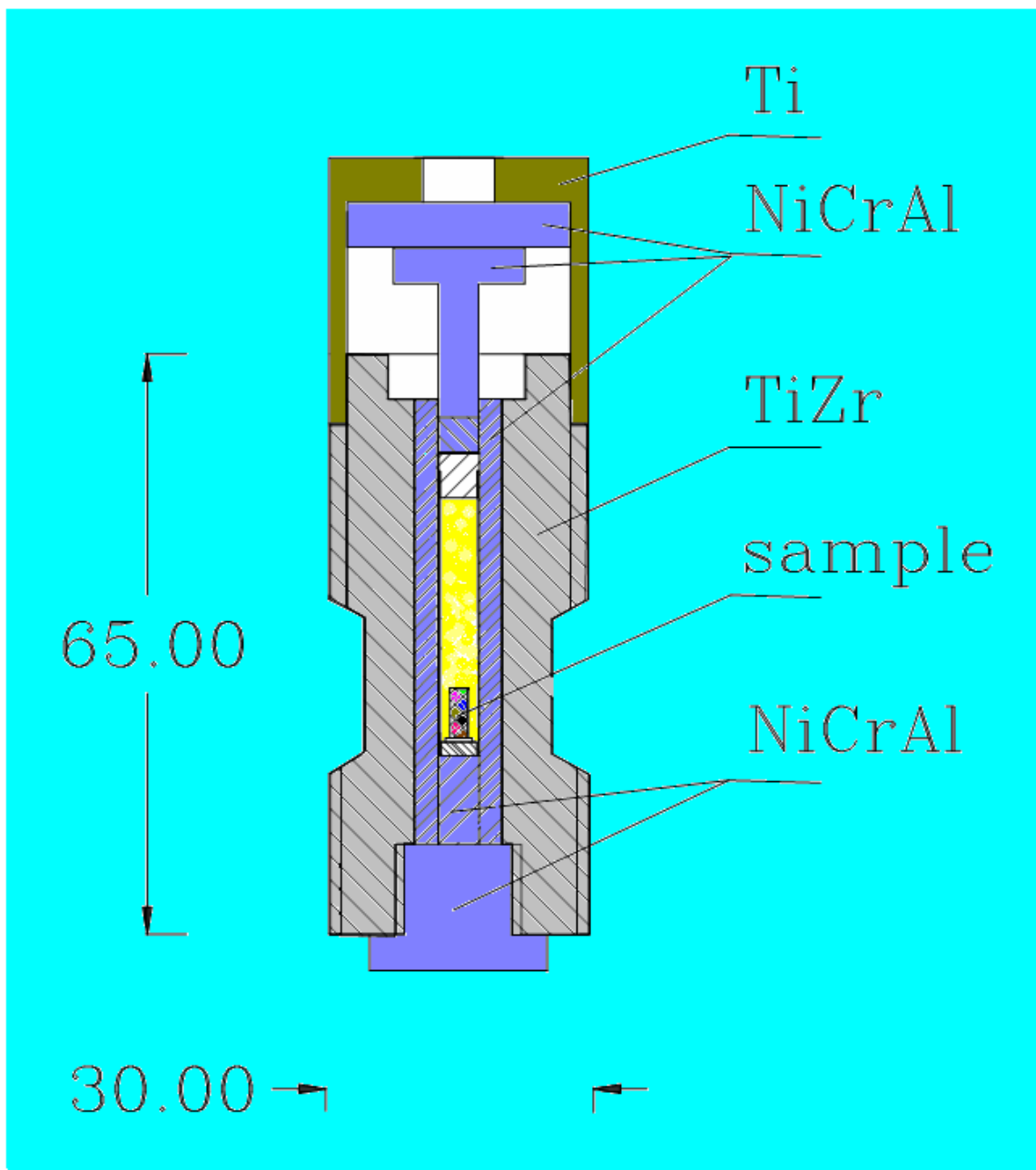




Quantum melting in magnetic metals (CeRh_2Si_2)



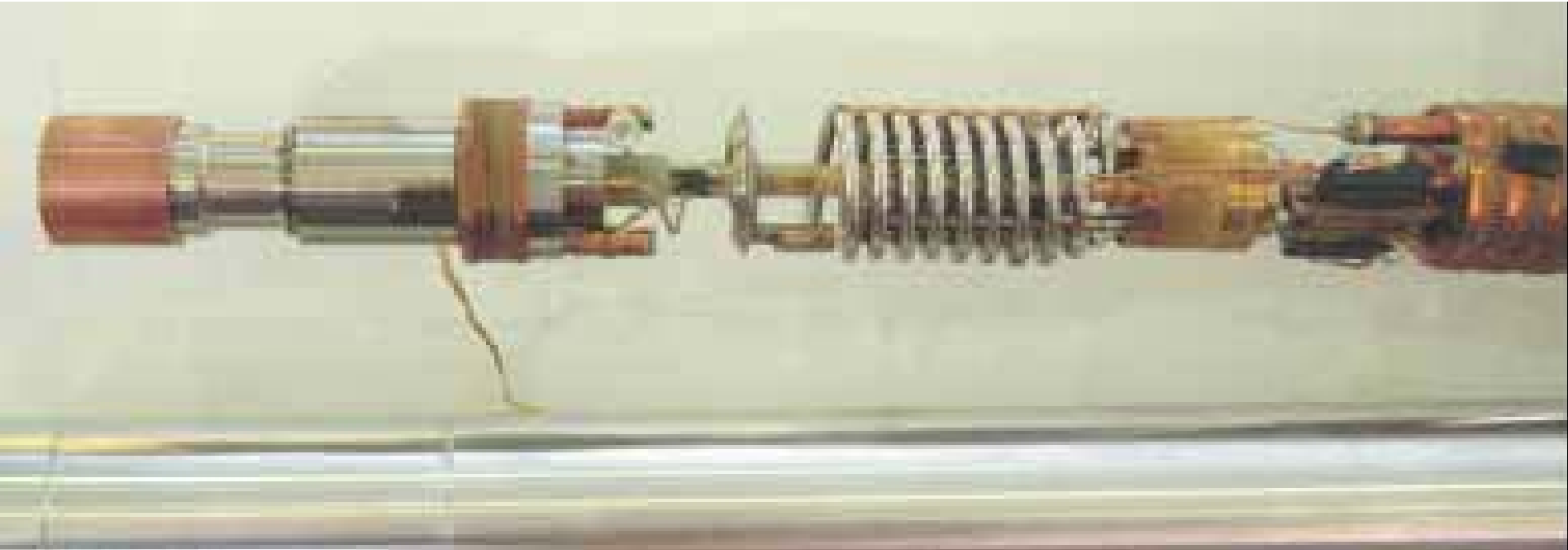
HPC25(100mk) for the *dilution fridge insert*. PRIZMA-ISIS-UK-2001



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.

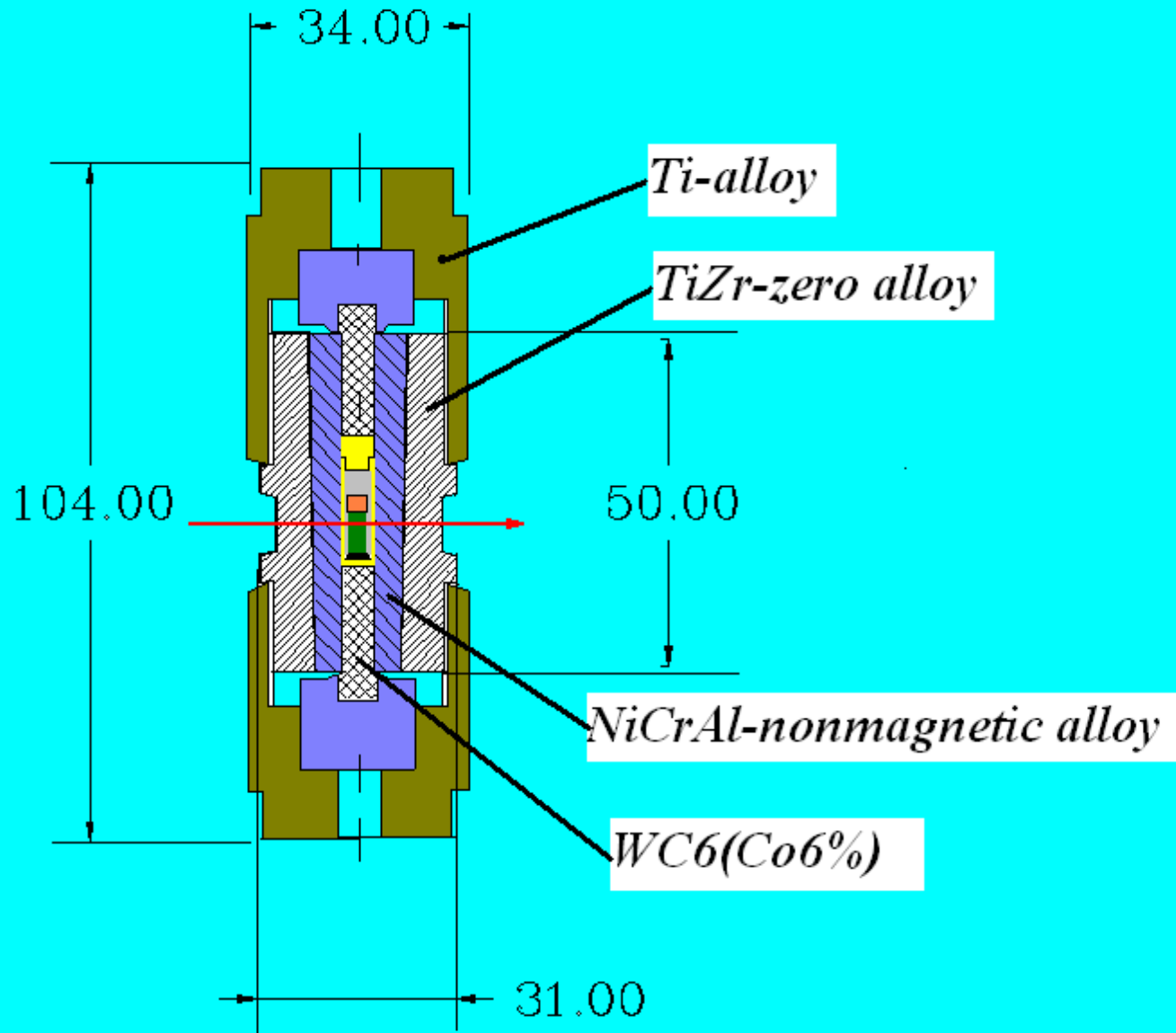
Magnetization distribution under pressure in the pressure induced superconductor CePd_2Si_2 .

N. Kernavanois, R. Sadykov, E. Ressouche, S. Raymond, P. Lejay and J. Flouquet, ILL Annual Report 2004/highlights/.



Photographs of the $P=40\text{kbar}$ 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.

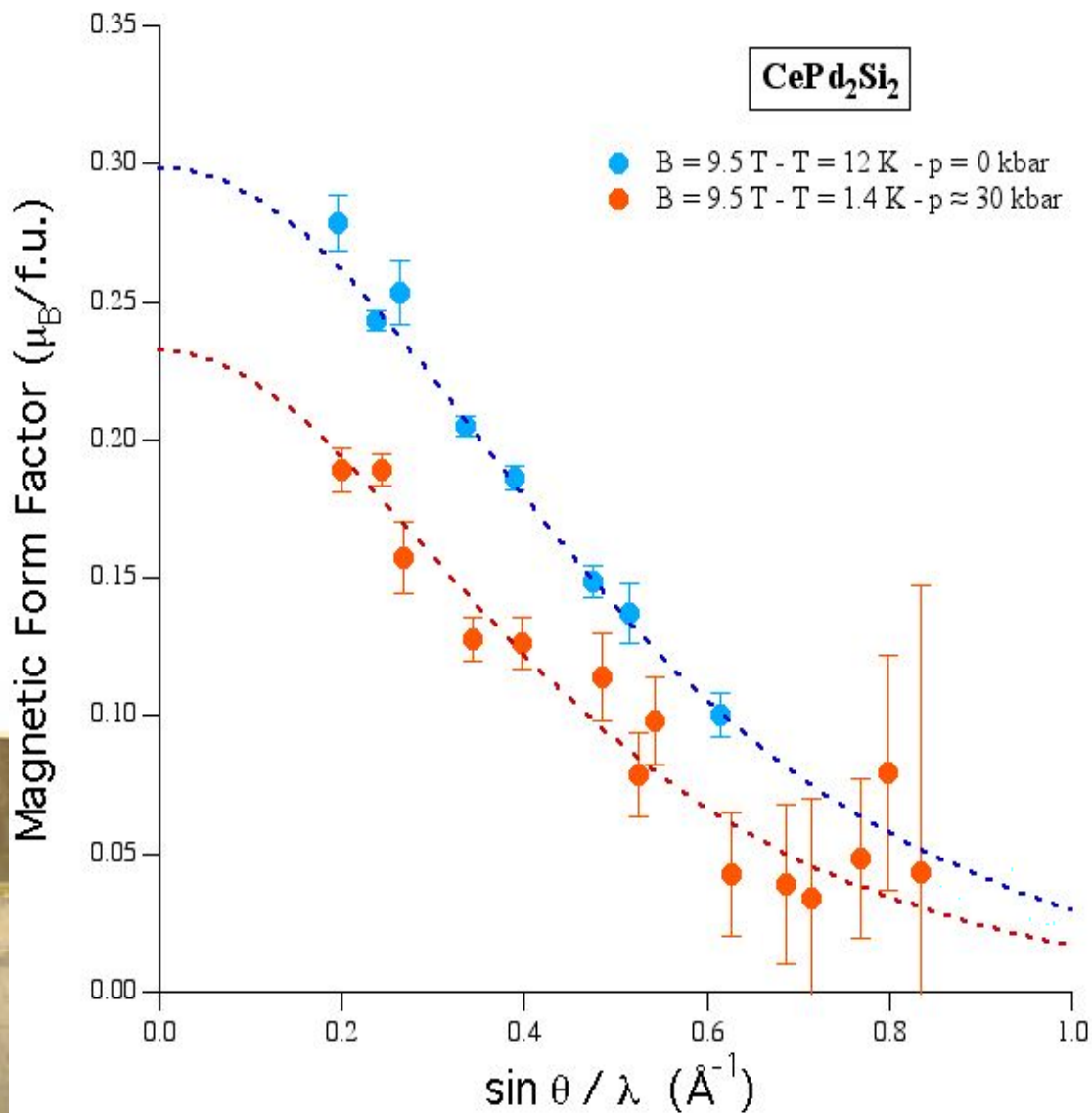
HPC40-ILL-D3(2004)



Magnetic form factors Ce 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.



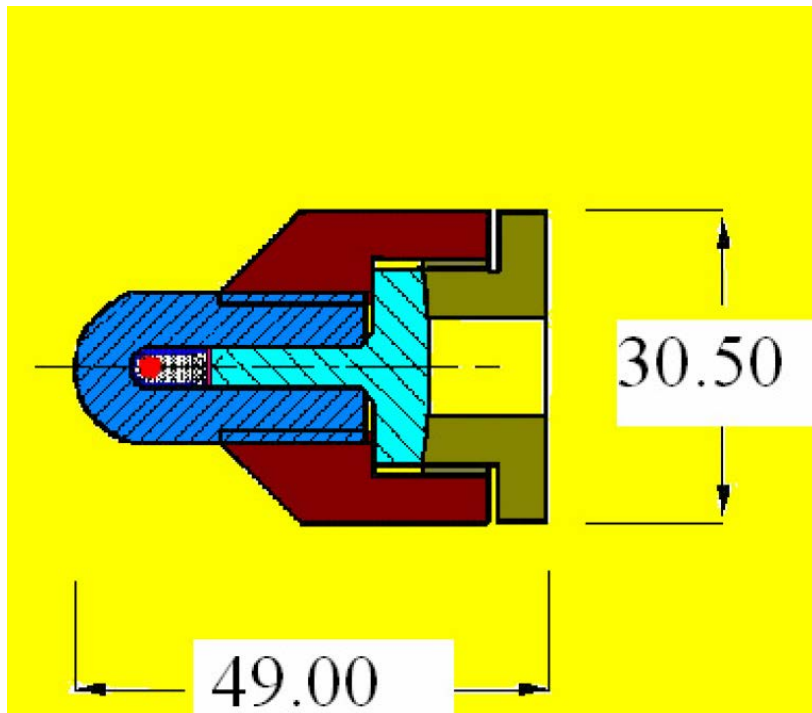
A new single-crystal pressure cell for TriCS up to 3 GPa.

R. Sadykov, D. Sheptyakov, O. Zaharko, Th. Strässle and J. Schefer.

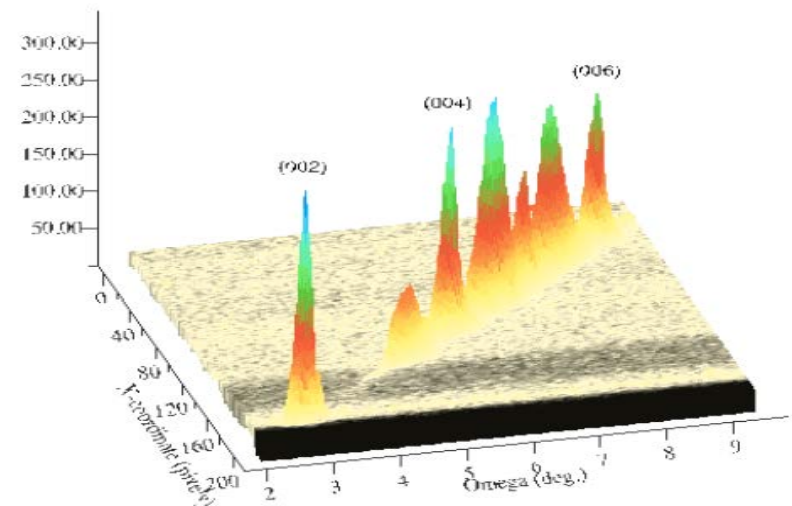
PSI, Scientific Report 2001 Volume III Condensed Matter Research with Neutrons, ISSN 1423-7326 March 2002, p.111 WWW-Version: FUN.WEB.PSI.CH

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

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



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

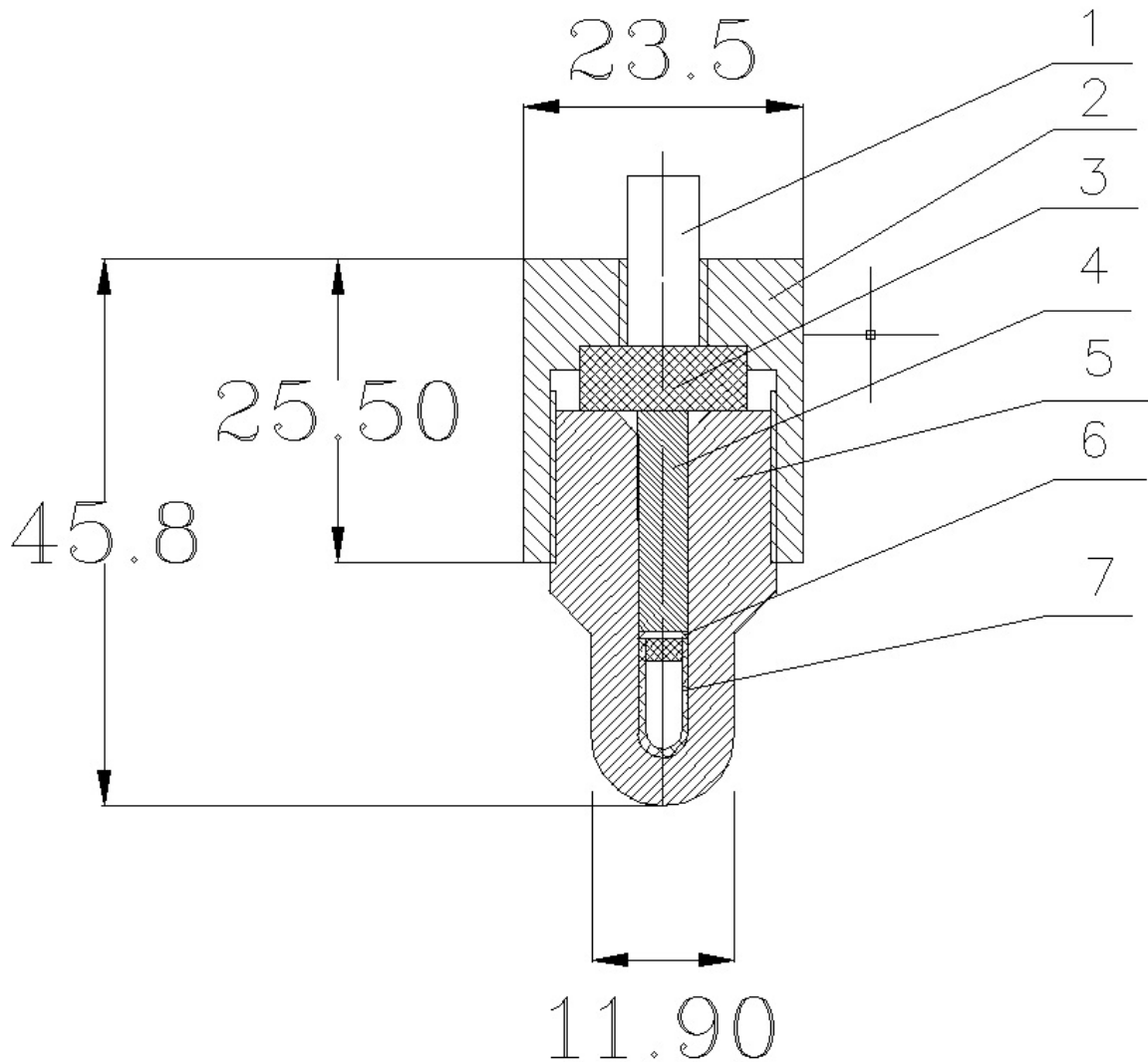


Bragg peaks and magnetic satellites from the sample measured with the 2D detector at $p=3\text{GPa}$, $T=17\text{K}$ (scans in omega presented as a sequence of 1D projection onto one of the detector axes).

(sample from: R.Sadykov et al., *Sov.Phys.Solid State* 23, 1865 (1981))

4-20-SNS2015

с принадлежностями:



1 -Толкатель, каленая
твердая сталь 45хн2мфа -
1 шт.

2-Гайка, титановый сплав
BT8 – 2шт .

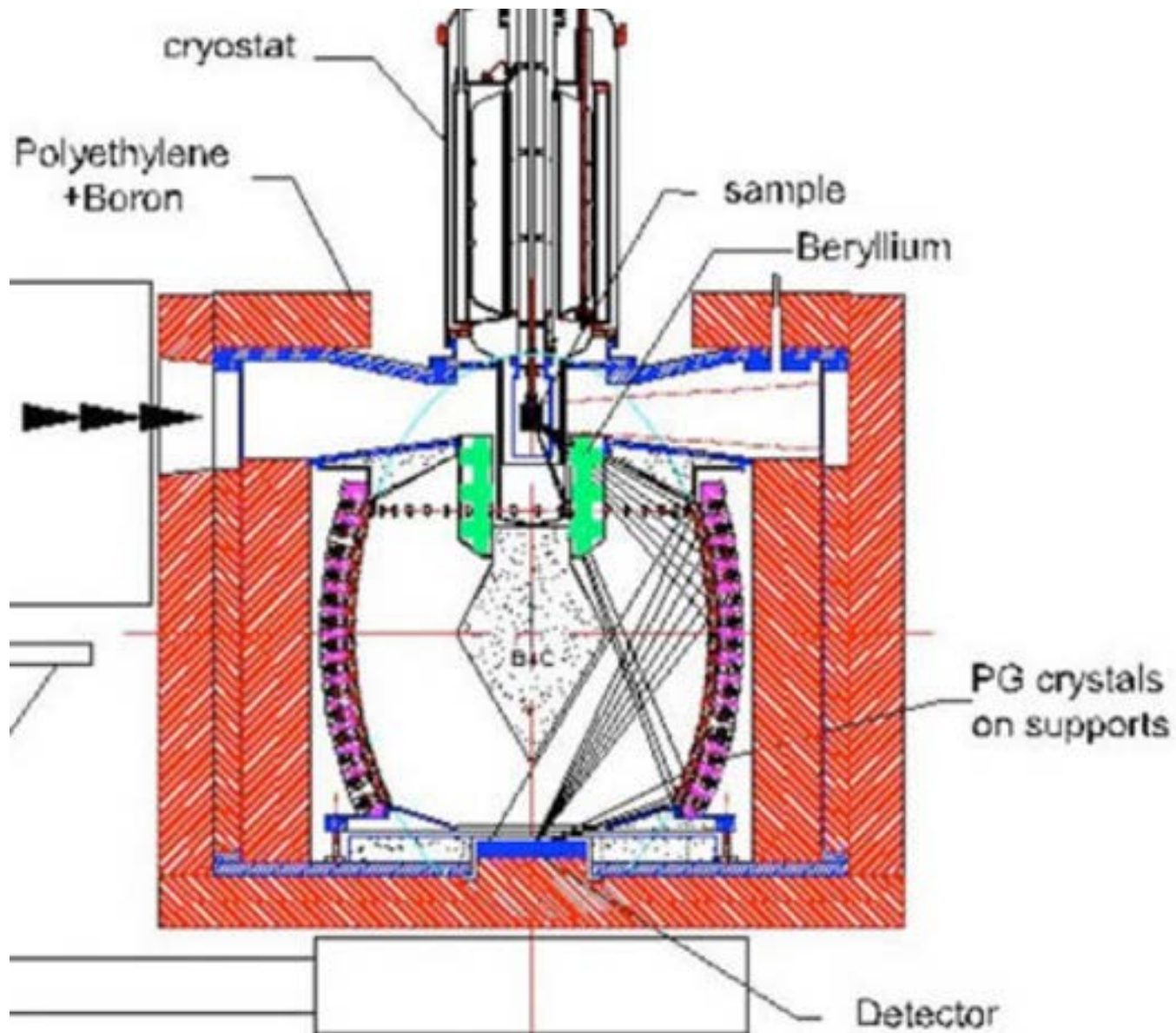
3- Опора , каленый
твердый сплав 40хню –
3шт.

4- Поршень, стальные или
твердый сплав WC6 – 2шт.

5- корпус камеры высокого
давления(пресс-формы),
сплав 40хню-2шт.

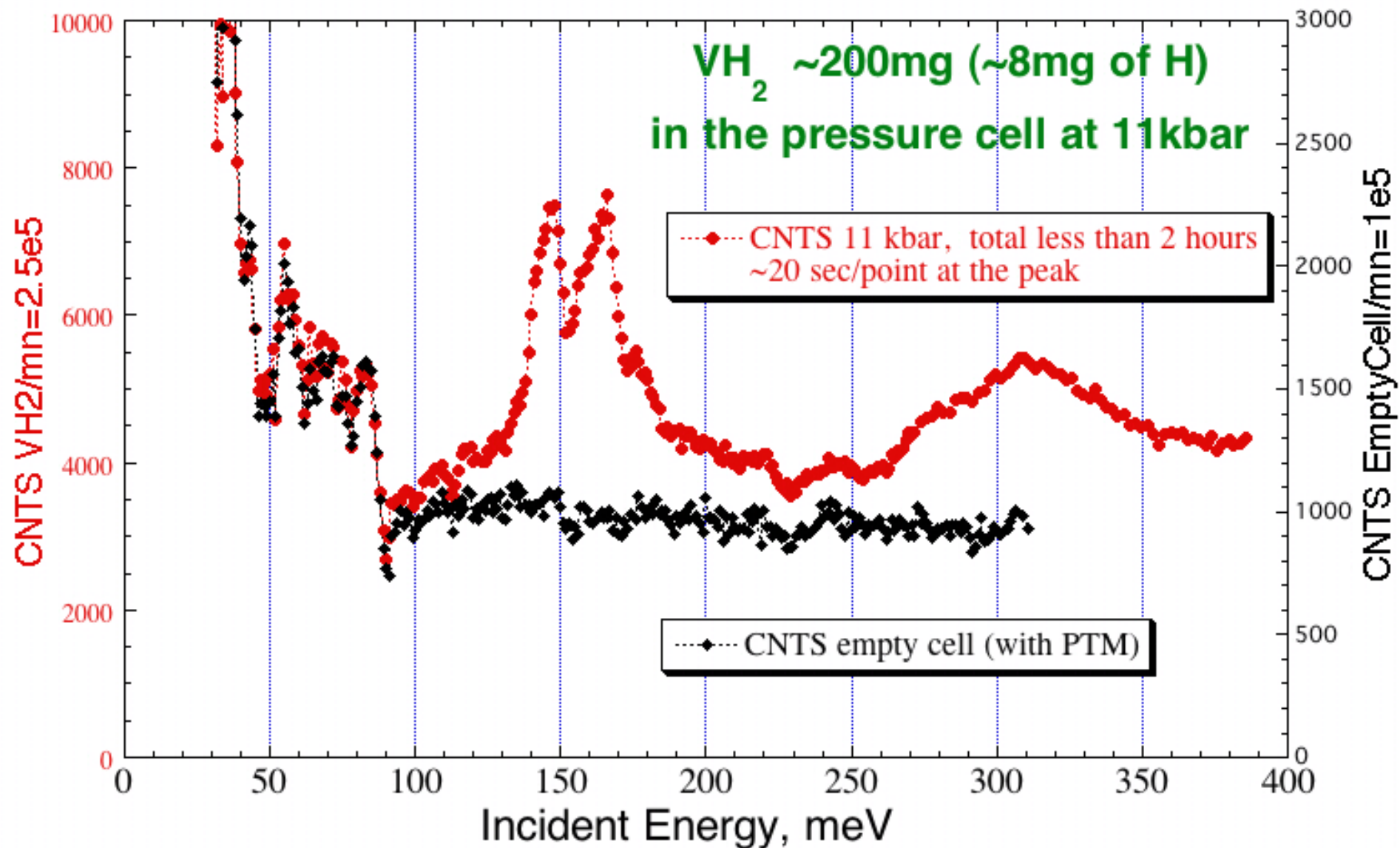
6- Уплотнительное кольцо,
сплав БрБ2 – 8 шт.

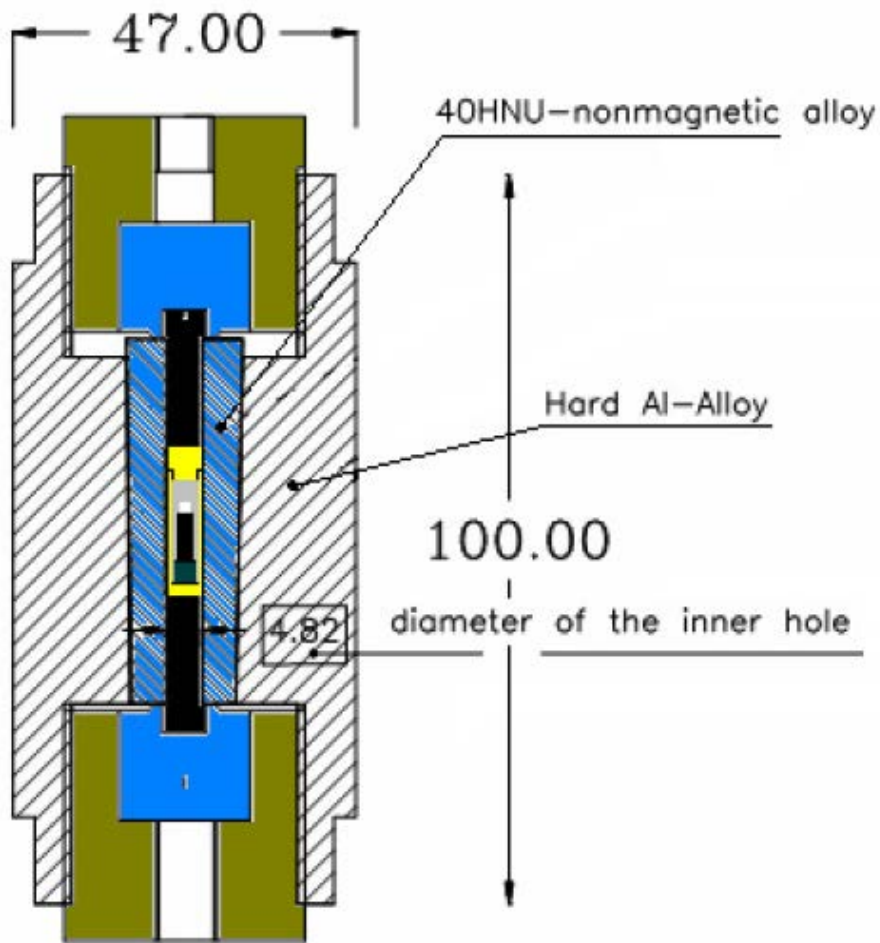
7- тефлоновая кювета – 10
шт.



**Hot neutron three-axis spectrometer IN1 - TAS/LAGRANGE
(Large Area GRaphite ANalyser for Genuine Excitations)**

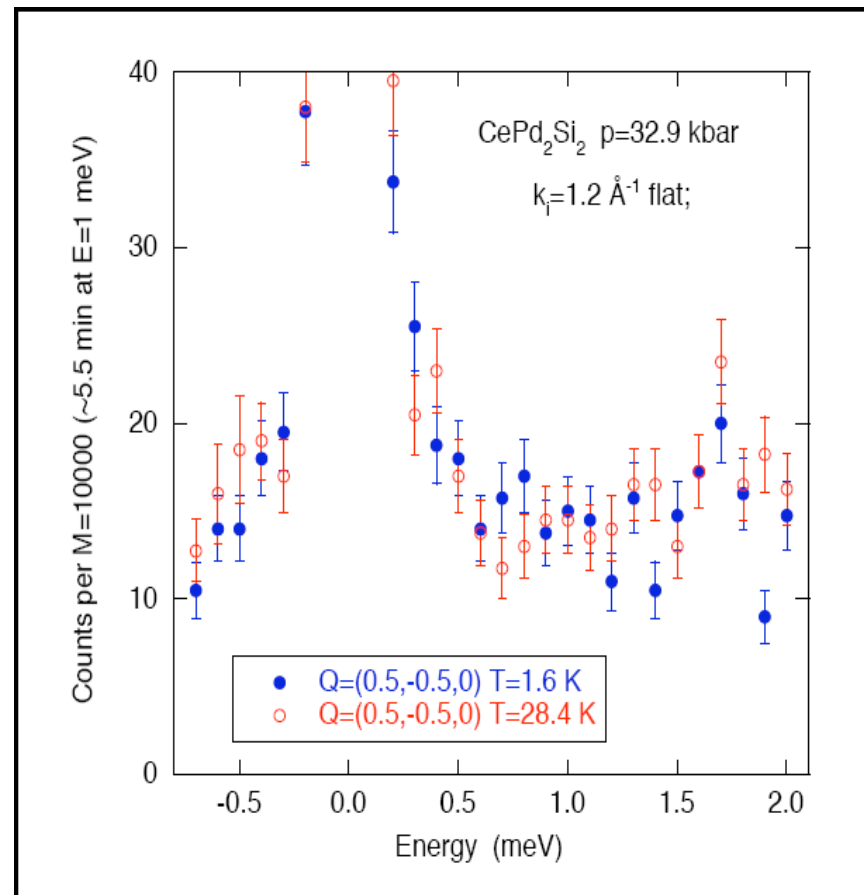
IN1-Lagrange 18-20.05.2013 Cu220 2D-foca





$$P_{RT}(T=300K)=30.5\text{kbar}$$

$$P_{LT}(T=1,5K)=32.9\text{kbar}$$

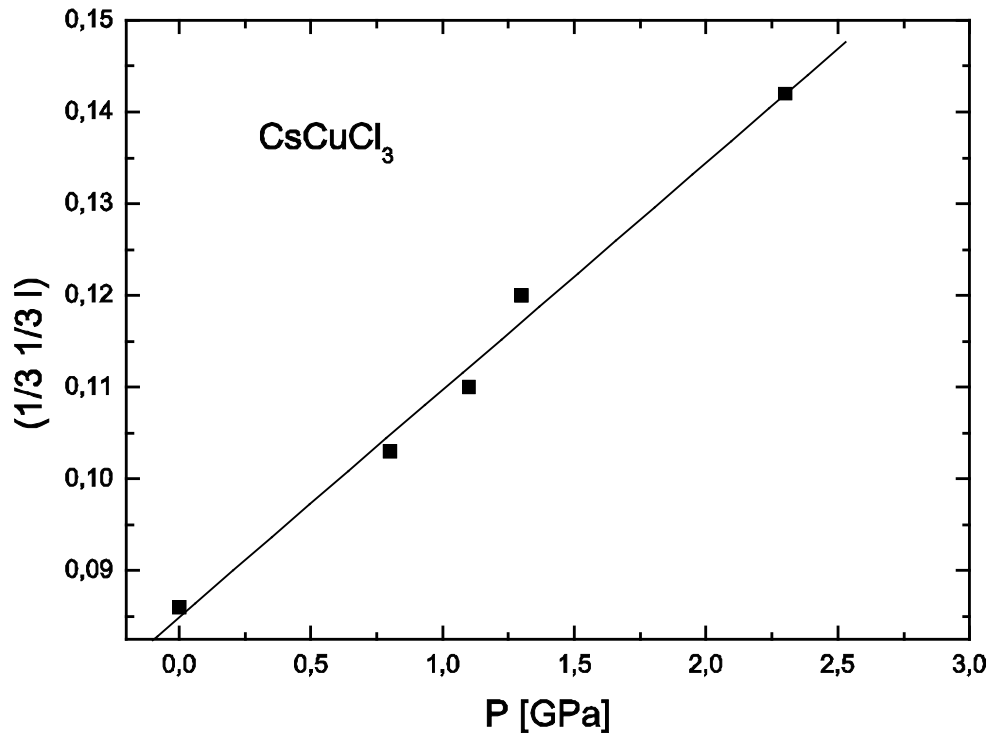


Inelastic scattering at $p = 32.9$ kbar, where temperature-dependent spin fluctuation scattering is expected.

Magnetic structure in CsCuCl₃ at high pressures

HMI, Date(s) of Experiment 15.4.-23.4.2005*

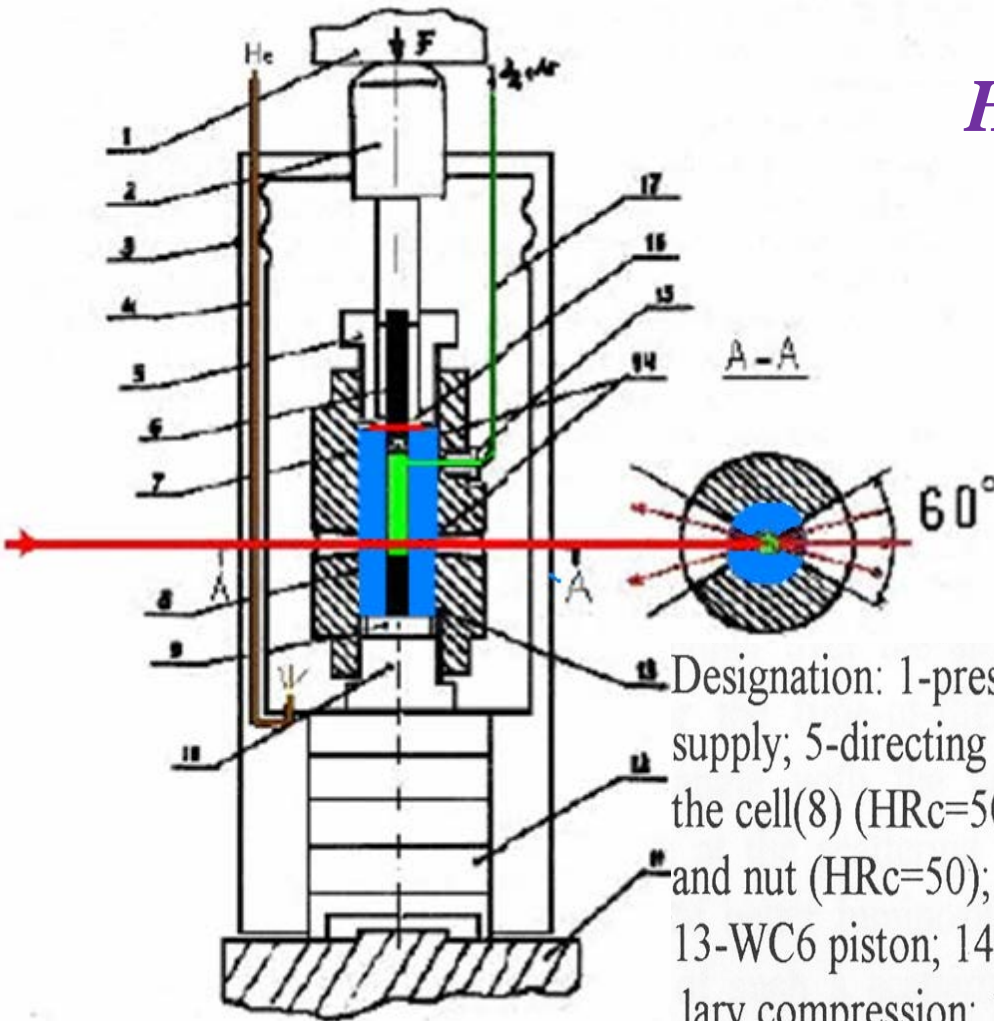
Principal Proposer: Norbert Stüßer, HMI Experimental Team: Ravil Sadykov, Inst. of high pressure physics, RAS Russia, Andreas Hoser, FZ Jülich/RWTH Aachen



The turning angle of adjacent spins along c changes from 5.1° at zero pressure to 8.5° at 2.3 GPa.

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 $P=0\text{kbar}$ ($T=4.2\text{K}$).

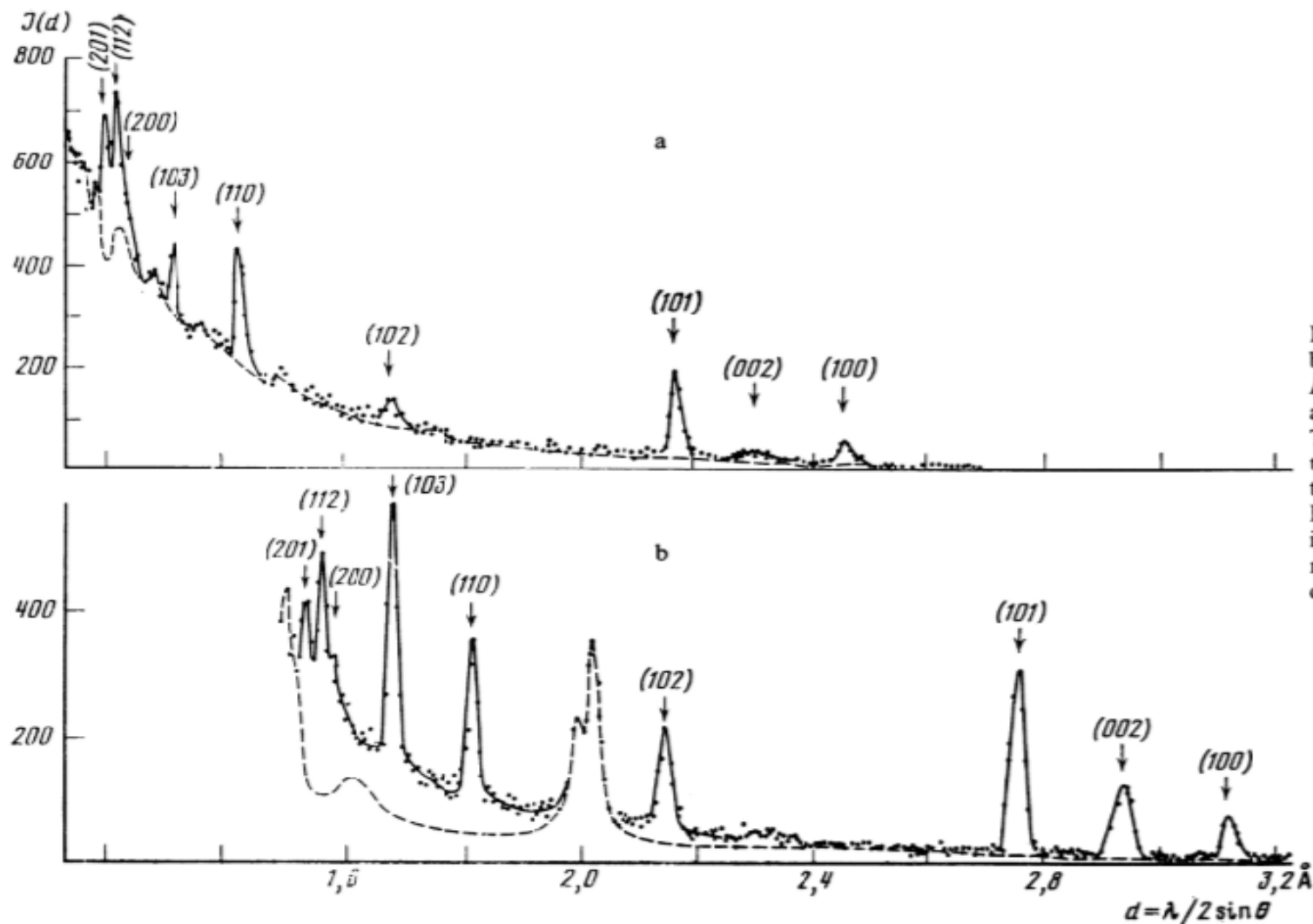
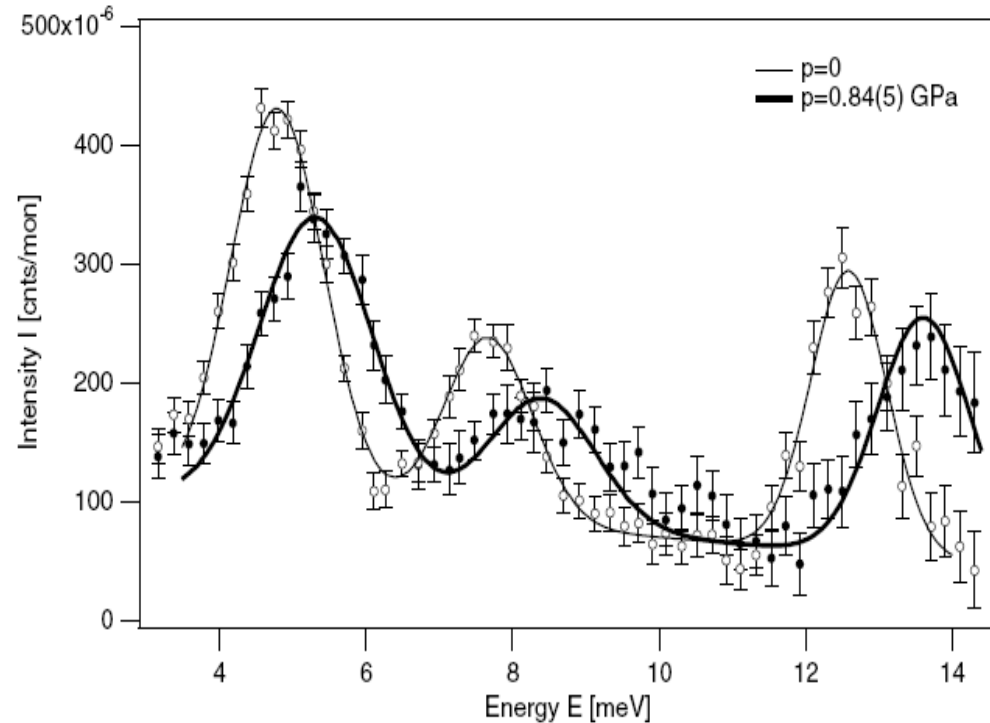
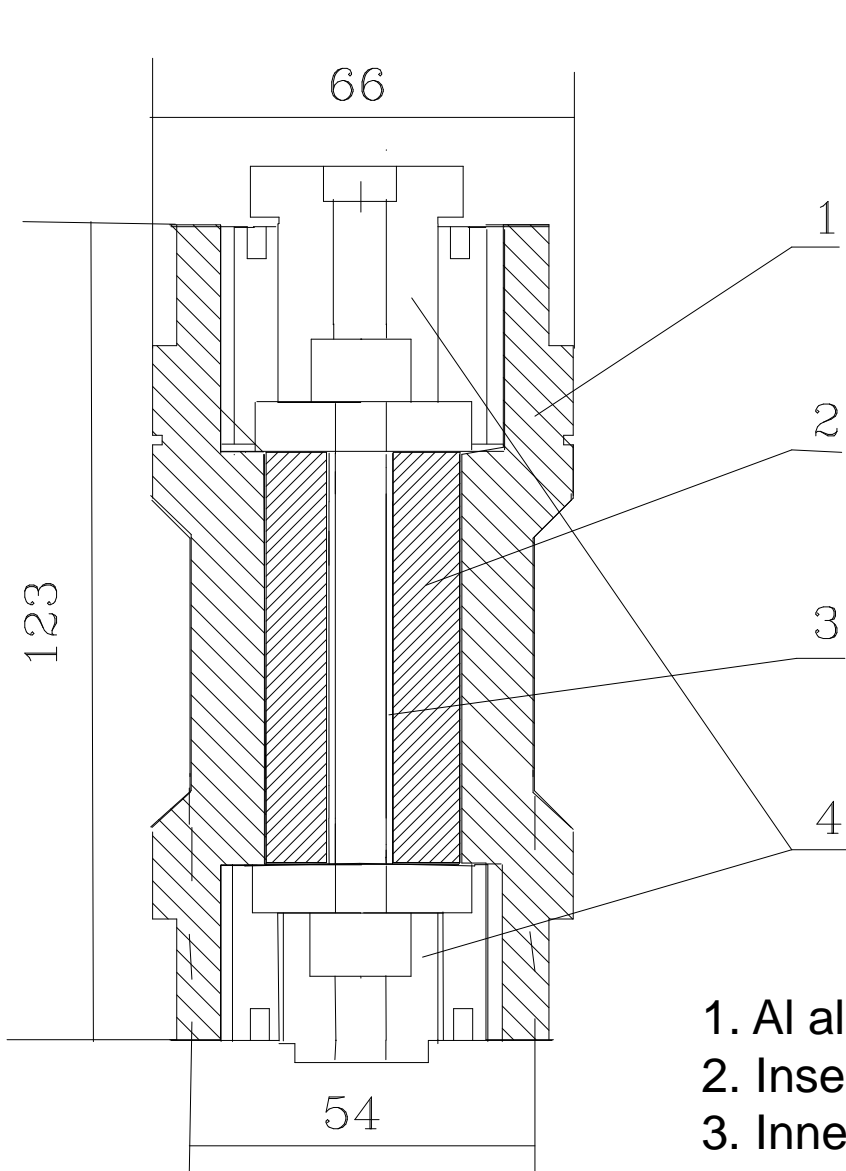


FIG. 1. Neutron diffraction by solid orthodeuterium at $P = 25.3 \text{ kbar}$, $T = 40 \text{ K}$ (a) and $P = 0$, $T = 4.2 \text{ K}$ (b). The arrows indicate the positions of the reflections from the hcp structure of ortho- D_2 . The background scattering from the chamber material is indicated by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

High pressure cells for inelastic neutron scattering studies

Hard Al, Steel and hard HNU (NiCrAl)
alloys

High Pressure Cell (HPC15-Al) 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).

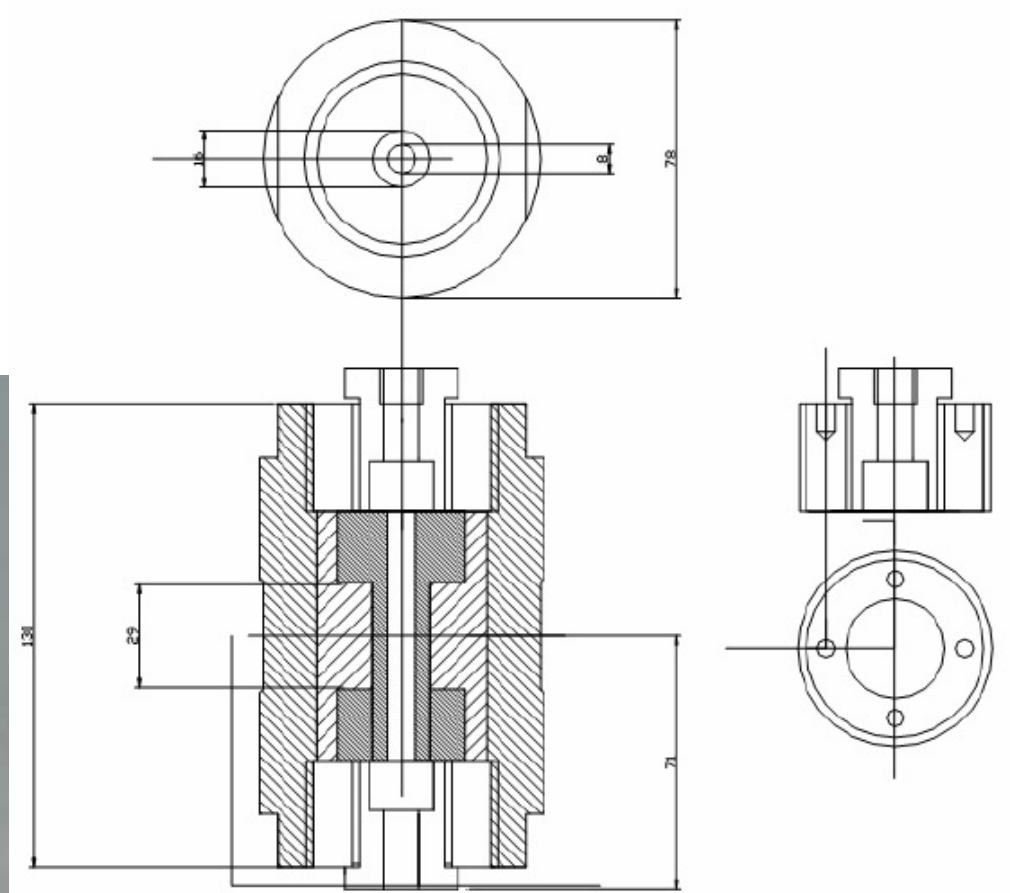
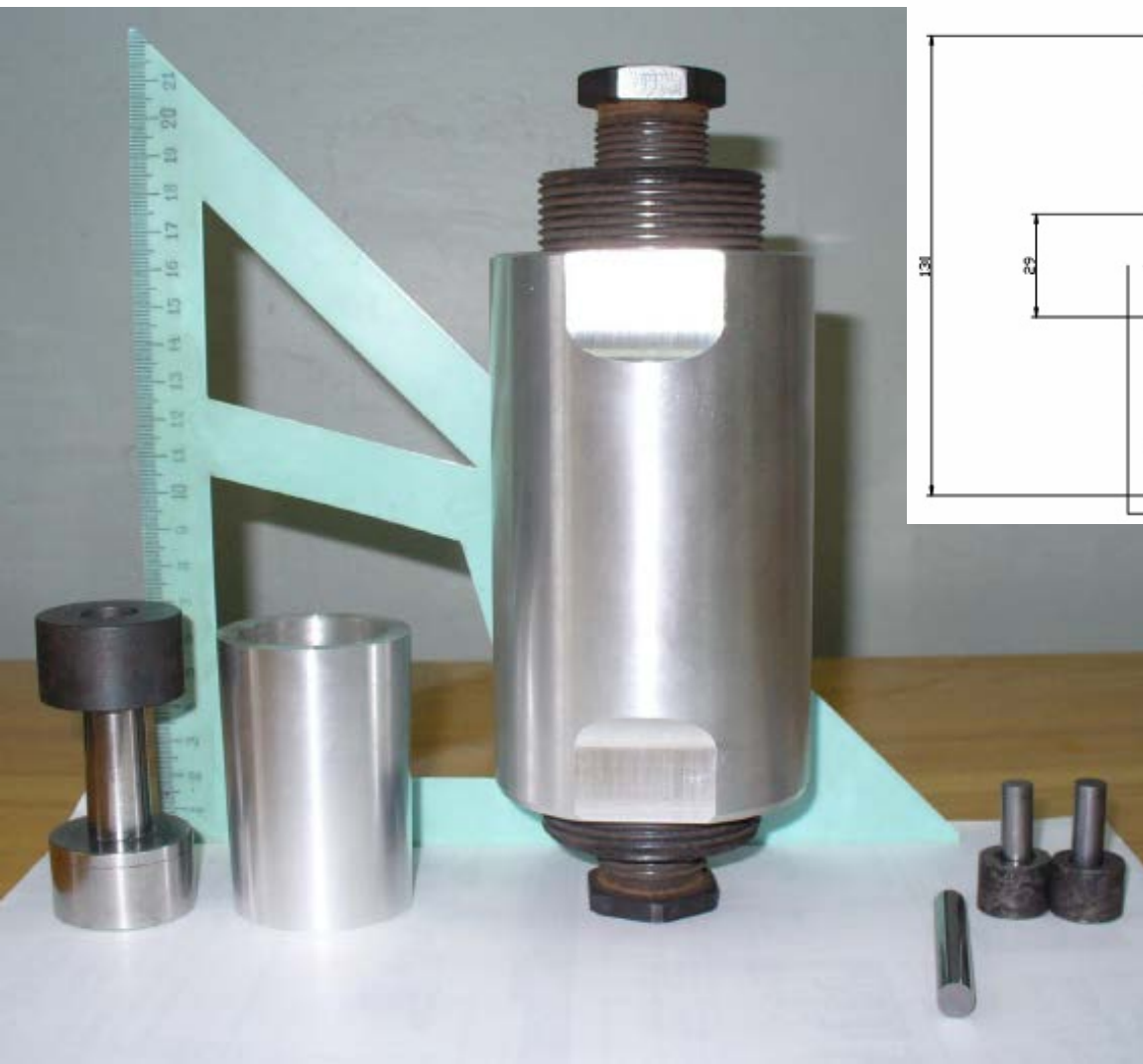
1. Al alloy - B95T
2. Insert part Al alloy- B96T
3. Inner part steel- 45XMHΦA
4. Nuts from steel-45XMHΦA.

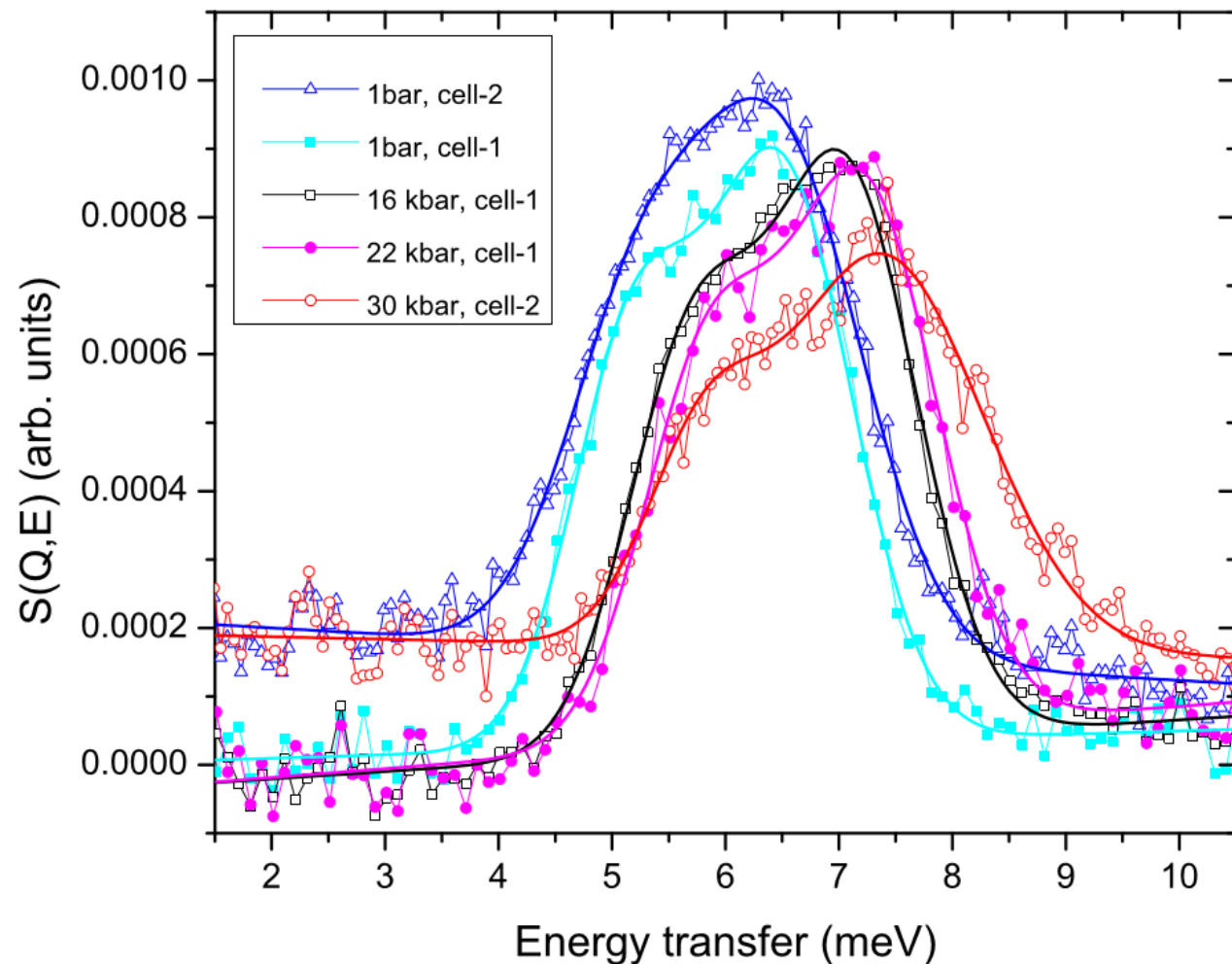
Hydrostatic Pressure Cell (HPC15-Al) : $P \leq 1.5 \text{ GPa}$

piston-cylinder clamp cell made of hardened aluminum



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





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.

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)

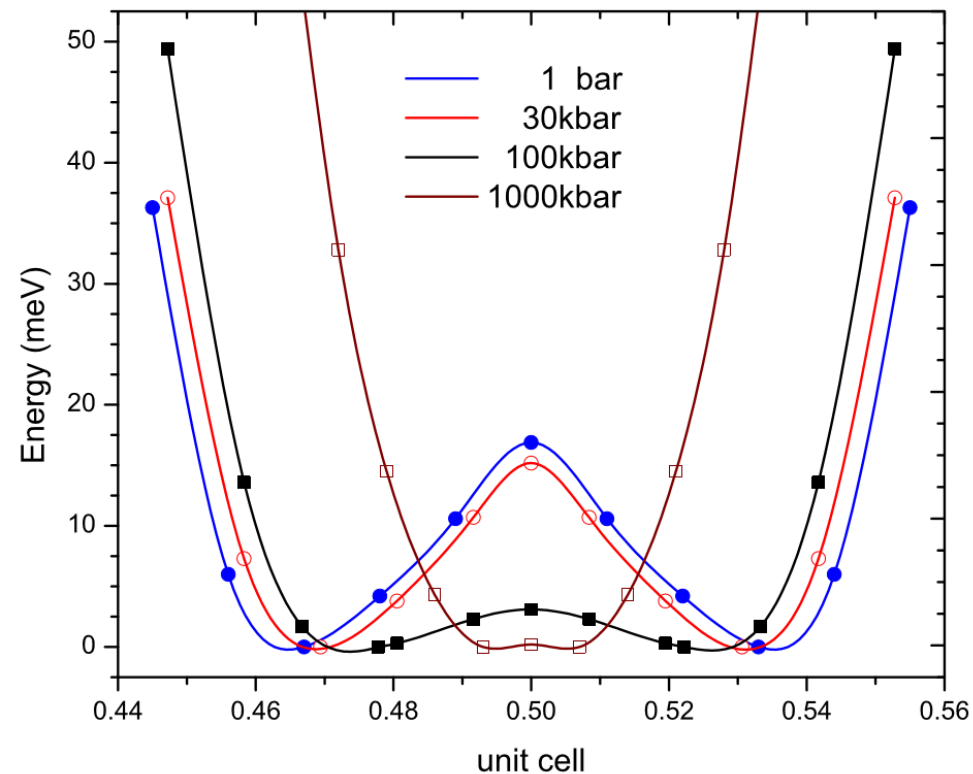


Рис.19.- Double-well potential for a hydrogen atom in α -Mn along the dumbbell calculated for different pressures; the distance is in the unit cell parameter units

Изучено влияние давления на режим туннелирования и колебательные спектры водорода в альфа-MnH0.07 методом неупругого рассеяния нейтронов. Применение гидростатического давления до 30 кбар показало сдвиг как оптической моды водорода так и пика туннелирования для более высоких энергий. Первопринципных расчеты показывают, что потенциал для водорода в альфа-Mn становится в целом более крутой с ростом давления. В то же время, высота барьера и его протяженность в направлении уменьшения туннельной и расчеты предсказывают, значительные изменения динамики водорода в α -Mn при 100 кбар, когда оцененное туннельное расщепление основного состояния водорода превышает высоту барьера [1].

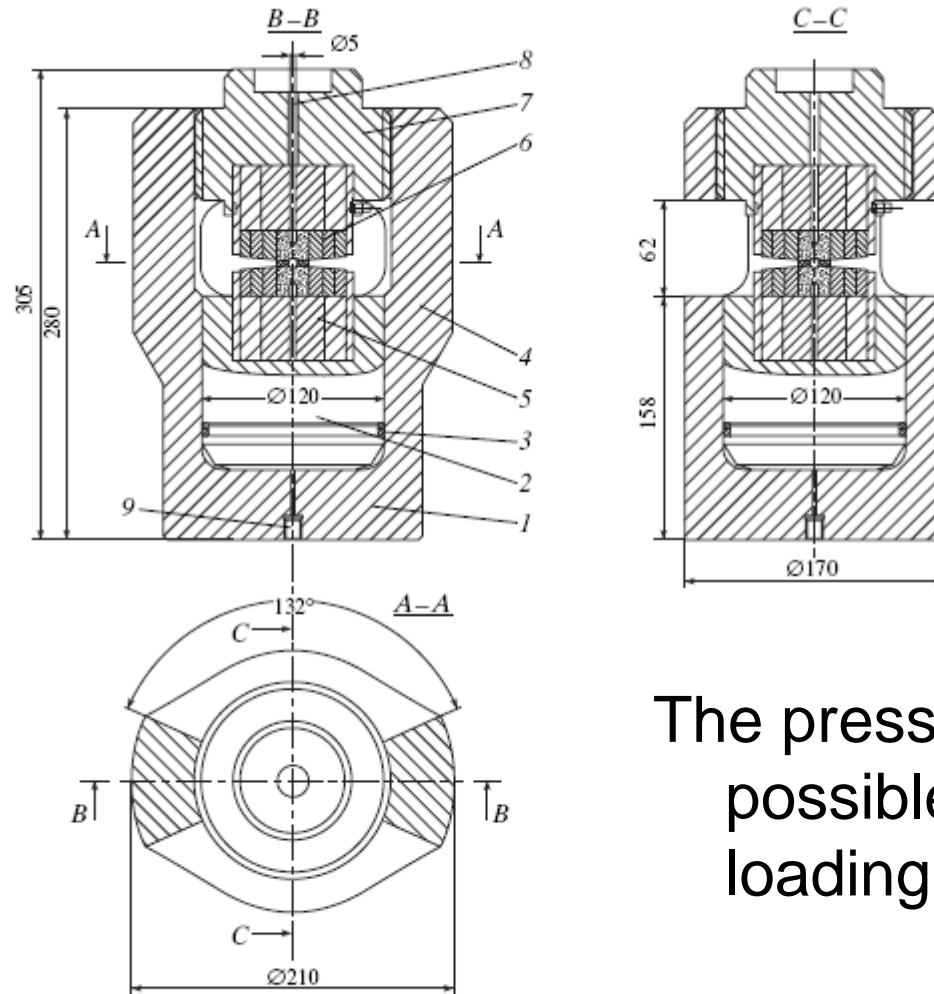
A Compact Hydraulic Press to Use with High-Pressure Devices for Neutron Scattering Studies

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¹ Vereshchagin Institute of High-pressure Physics, Russian Academy of Science, Troitsk, Moscow oblast, 142190 Russia
e-mail: sergei@hppi.troitsk.ru

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.

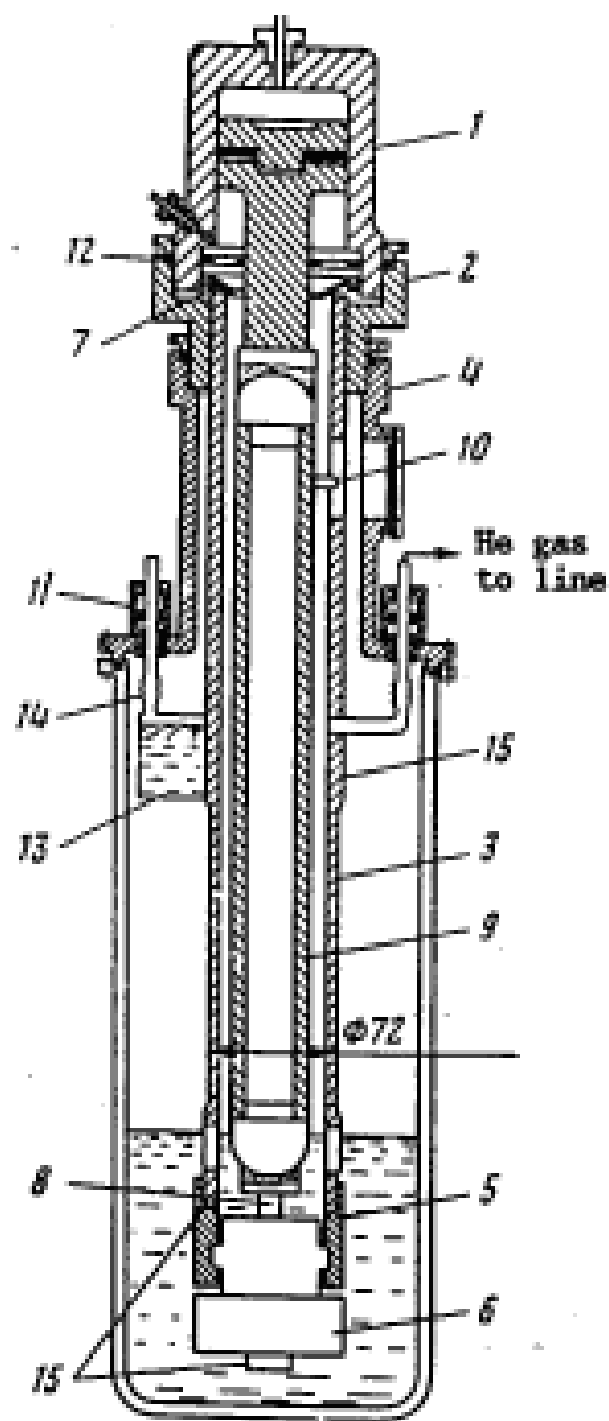
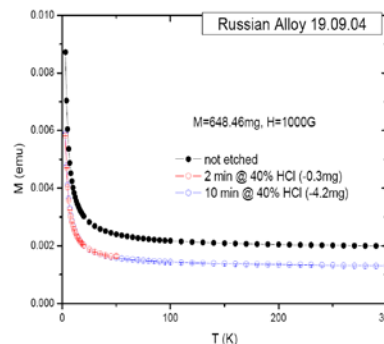
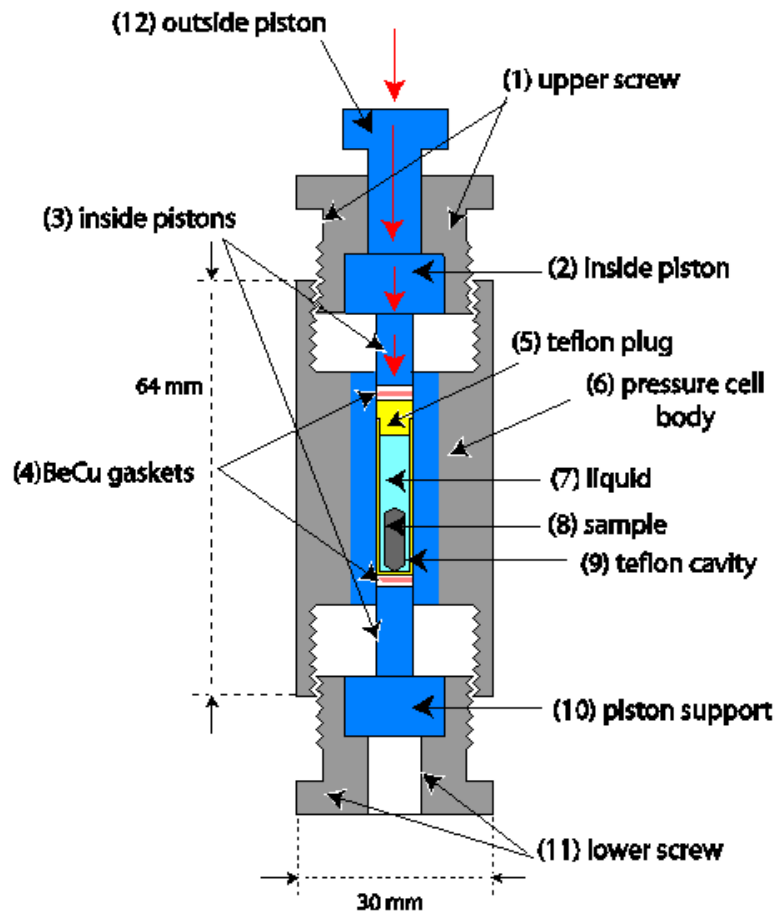


Fig. 1. Press with cryostat: 1) cylinder; 2) coupling; 3) outer tube; 4) part joining press to cryostat; 5) lock; 6) high-pressure chamber; 7) disk spring; 8) shaft of high-pressure chamber; 9) inner tube of press; 10) mark; 11) sleeves; 12) vacuum-rubber gas-kets; 13) vessel for liquid nitrogen; 14)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

Nonmagnetic high pressure cell for magnetic remanence measurements up to 1.5 GPa in a superconducting quantum interference device magnetometer",

Rev. Sci. Instrum. 79, 115102 (2008);

Ravil A. Sadykov, Natalia S. Bezaeva, Alexander I. Kharkovskiy, Pierre Rochette, Jerome Gattacceca, and Vladimir I. Trukhin.



Schema of the cell. Parts in grey are made of Ti-rich alloy and parts in blue are made of NiCrAl alloy (so-called Russian alloy)

Nonmagnetic HPC20-PL-MAGN-1-2006

