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NEUTRON TIME-OF-FLIGHT DIFFRACTOMETER FOR INVESTIGATIONS OF MICROSAMPLES UNDER HIGH EXTERNAL PRESSURE

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ABSTRACT

A new neutron time-of flight diffractometer for investigations of microsamples structure under high external pressure in diamond and sapphire anvils cells with the use of elastic neutron scattering is described. The diffractometer is operating at the IBR-2 pulsed reactor at JINR. The time-of-flight method and ring-shaped multicounter detectors technique are used to register the scattered neutrons. Parameters and methodical peculiarities of the device are given.

1. INTRODUCTION.

Progress in the study of substances under high pressure by direct methods is closely connected with the use of diamond anvils, providing pressures up to 400 GPa, and synchrotron radiation which allows the study of very small amounts of substance due to high illumination. At the same time a whole set of important problems, such as magnetic transitions, structural phase transitions in systems containing atoms with small or similar numbers demands for the use of neutron methods. But the range of neutron methods application was usually limited to the value of several GPa. This was connected with the use of relatively large sample volumes (~1 cm^3) and cylinder-piston type pressure cells. The use of diamond anvils for this purpose seemed to be impossible because of the small (~10^{-3} mm^3) amounts of substance and the relative weakness of neutron source fluxes. During the last ten years, however, a new method of neutron investigations at high pressures was developed.

Keywords: time-of-flight, high pressure, sapphire, anvil

It was based on the successful composition of diamond and sapphire anvil techniques and high-illumination, low-background neutron diffractometry [1]. This allowed the range of available pressures to be increased up to several tens GPa.
As inelastic neutron scattering experiments demand larger amounts of substance than diffraction ones, they were carried out only in of piston-cylinder type systems in a limited range of pressures. The first work on inelastic coherent neutron scattering investigations with the use of sapphire anvils was carried out just recently [2], and the possibilities of using this technique for inelastic incoherent neutron scattering has not been studied yet.
A new time-of-flight neutron diffractometer (DN-12) is described in this work. It is destined for elastic neutron scattering studies of polycrystal samples under high pressures in pressure cells based on sapphire and diamond anvils. The diffractometer is installed at a high-flux pulsed neutron source - the IBR-2 reactor of JINR. The parameters and methodical peculiarities of the device are discussed in this paper.

2. CONSTRUCTION OF THE DIFFRACTOMETER

A view of the DN-12 diffractometer is shown in fig.1. It is placed in the 12th channel of the IBR-2 reactor and consists of the following main systems: neutron beam chopper phased with the power pulse of the reactor, beam collimating system, multicounter circular detectors system, information management, registration and processing system.

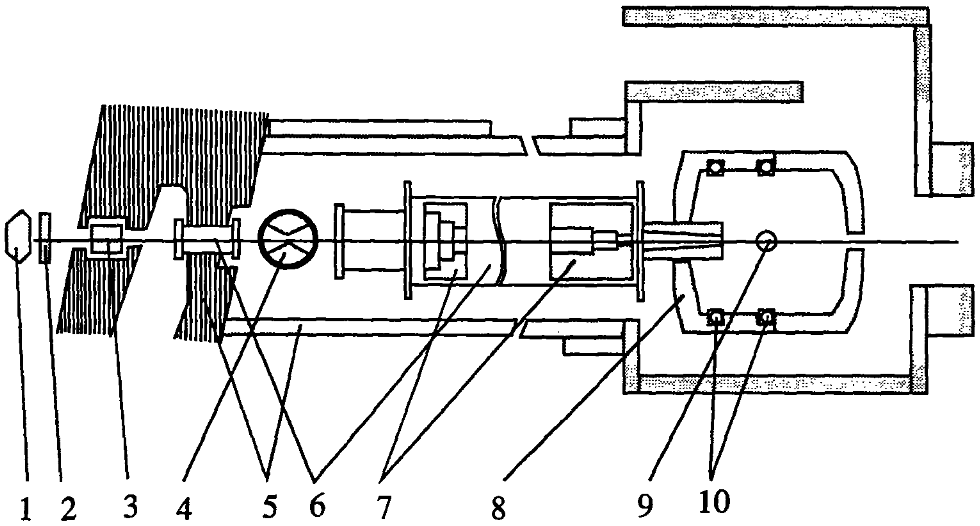

*fig. 1. Lay-out of the DN-12 diffractometer at the IBR-2 pulsed reactor.
1 - active core, 2 - moderator, 3 - beam damper, 4 - chopper,
5 - fast neutron shield, 6 - vacuum neutron guide, 7 - beam collimators,
8 - shielding, 9 - sample position, 10 - ring-shaped detectors.*

The DN-12 diffractometer is specifically intended for works with high-pressure cells based on sapphire anvils. A well collimated neutron beam passes through the sapphire single crystals, between which a sample is placed (Fig.2). Scattered neutrons are registered by the detectors consisting of small counters positioned around the circles of a vertically standing rings. The rings as a whole can be moved relative to the sample along the beam direction providing scattering angles from 45° to 135°.

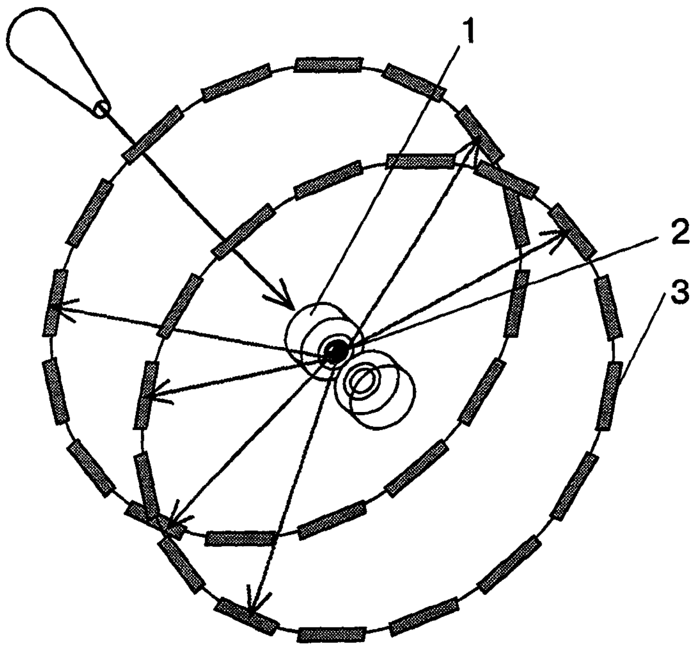

fig.2. Position of the high-pressure cell with the sample and the detector at the DN-12 diffractometer. The neutron beam passes through the sapphire single crystals 1. The neutrons scattered from the sample 2 are registered by the ring-shaped detectors 3 assembled of separate counters.

3. DN-12 PARAMETERS

TABLE 1. The main characteristics of DN-12.

Parameters	1994	1997
1. Thermal neutron flux at the sample position ($n/cm^2/s$)	$1\cdot10^6$	$2\cdot10^6$
2.Distances: moderator - sample sample - detector	31.8 m 0.4 m	27.0 m 0.4 m
3.Range of: wavelength	1 ÷ 4 A	0.8 ÷ 10 A
scattering angle	45° ÷135°	45° ÷ 135°
d_{hkl}	0.8 ÷ 5.2 A	0.6 ÷ 13 A
4.Resolution (Δd/d, d=2A): for 2θ=90°	0.022	0.022
for 2θ=135°	0.012	0.012
5. Solid angle of the detector system	0.125 sr	1.0 sr
6.Pressure range: with sapphire anvils	5 GPa	5 GPa
with diamond anvils	20 GPa	20 GPa

4. EXPERIMENTS ON DN-12

TABLE 2. Experiments performed with DN-12 during November 1993 to March 1994. Pressure is given in GPa, sample volume in mm^3.

Sample	Pressure	Volume	Anvils	Main result
DyD_3	10.0	0.01	diamond	equation of state
Hg-1212, Hg-1201	3.6 3.2	1	sapphire	structural changes
ND_4Cl	2.5	2	sapphire	structural changes
Fe_2O_3	4.7	1	sapphire	new magnetic phase above 3 GPa

4.1 The lowest limit of the sample volume.

Several experiments have been carried out in order to determine the minimal quantity of substance from which it would be possible to obtain a diffraction pattern at our facility within a reasonable amount of time (not more than 50 - 100 hours). For this purpose, the $^{164}DyD_3$ sample with a maximal coherent scattering cross-section was used (σ_{coh}≈250 b). This sample was placed in the diamond anvil cell. The sample volume was 0.027 mm^3, the pressure was about 10 GPa, and it was possible to increase the pressure up to 20 GPa. It took 24 hours to obtain a clear diffraction pattern.

4.2 Structural experiments

We have already used our diffractometer to carry out several structural experiments.

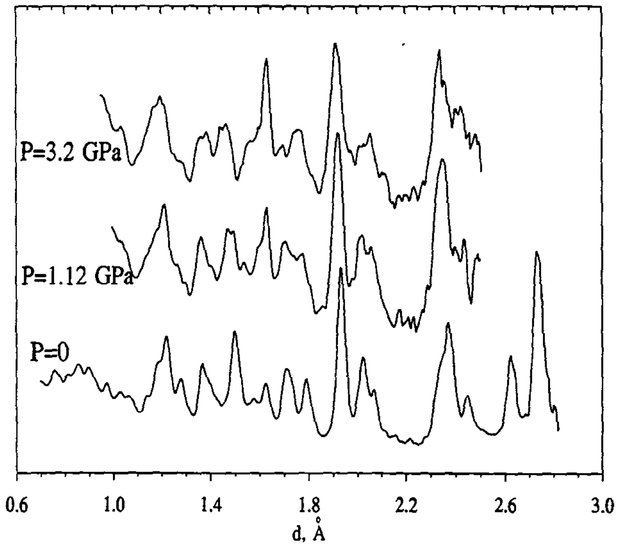

fig. 3. Typical view of raw neutron diffraction patterns obtained at the DN-12 diffractometer - patterns of hg-1201 structure under high external pressure

For example, the pressure dependences of lattice parameters and interatomic distances of some representatives of the recently discovered series of mercury-based superconductors were investigated [3]. We performed the studies of $HgBa_2CaCu_2O_{6.3}$ (Hg-1212) and $HgBa_2Cu_1O_{4.2}$ (Hg-1201) (see raw experimental spectra on the fig.3). The results including lattice parameters and interatomic distances (within the accuracy of 0.005 A) appeared to be in good agreement with the data obtained at another devices [4].

5. DEVELOPMENT OF THE DN-12

As the DN-12 is a new device, it is constantly developed and modified. The fig.4 shows the probable plan of the DN-12 diffractometer layout as it will be in a few months probably.

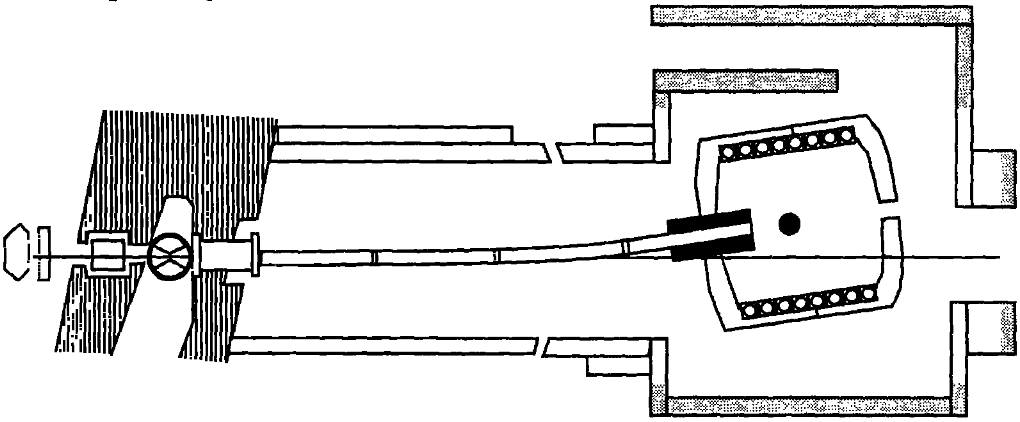

fig.4. Probable lay-out of the DN-12 diffractometer at the IBR-2 pulsed reactor after improvement.

It is planned to develop the DN-12 in the following directions:

- moving of the neutron beam chopper to another position (The distance will be 4.5 m between moderator and chopper instead of 8.9 m at the moment);
- substitution of the vacuum neutron guide tube by the curved mirror neutron guide tube;
- increasing of the number of the detector rings to 8 instead of 2.
- improvement of detector system, including the creation of data acquisition system in the VME standard;
- creation of the special equipment for loading the pressure cell with the optical spectrometer measurement of pressure;
- widening of experimental possibilities (using of new materials for the anvils, getting low and high temperatures at the samples).

CONCLUSION

The experiments carried out at the DN-12 have shown its compete-ability with the similar devices in such centres as RAL, RRC-KI, ANL, LANL. Putting of new detector system into operation, building of mirror neutron guide tube and moving of the beam chopper will allow to improve the device characteristics significantly, will increase the efficiency of its using and will make it more lovely for the users.

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