

## THE MATERIAL RESEARCH LABORATORY IN DEVELOPMENT OF THE NEW METHODS AND INSTRUMENTS WITH ITS TESTING IN CONDITIONS OF REAL SCIENTIFIC EXPERIMENTS

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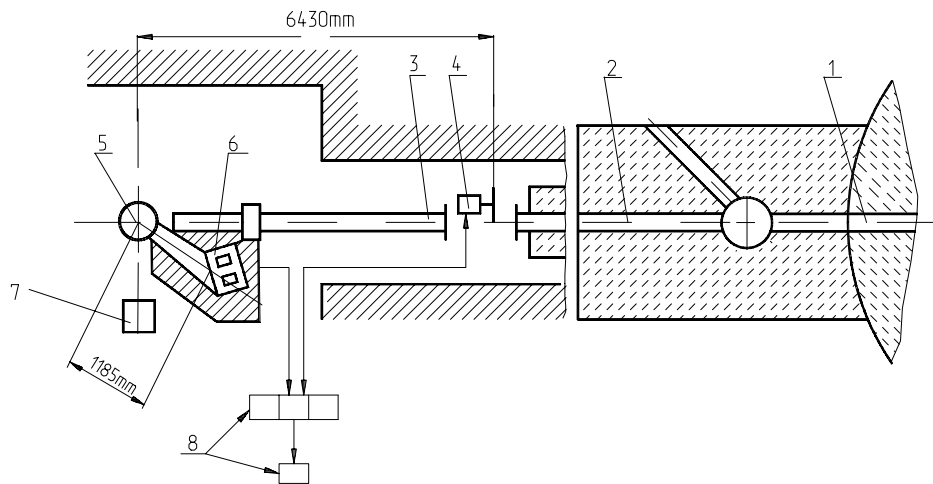
The neutrons are enough exotic and very expensive tool with evident shortage in intensity beam and their available number. One of main goals of any experimentalist is to improve situation in both mentioned directions, also extreme attention should be devoted to optimization of all the components of used facilities.

The neutron guide systems are widely applied for creation of experimental facilities at all the modern neutron centres. One of the basic reasons of application of neutron guide systems is the opportunity considerably to increase the number of efficiently used instruments at such expensive installation as a neutron source. The high-flux reactor of the Institute Laue-Langevin (ILL, Grenoble, France) is a demonstrative example of "mass" use of neutron guides. This neutron centre is also demonstrative in other occasion: the neutron guides are not ended in themselves, but they are used as components and optimized units of physical instruments. Just the necessity of optimization of neutron optical units of diverse physical experimental facilities has given serious stimulus to perfect neutron guide systems, for example, for designing and creation of polarizing and non-polarizing supermirrors.

One of technical shortages of neutron guide systems confining their application, was insufficient luminosity conditioned by the small input/output apertures. The input/output apertures were determined by a small value of angles of the total reflection on surfaces of the neutron guide channel, for example, for natural nickel coating  $\theta_{cr} \approx \lambda \cdot 1.7 \cdot 10^{-3}$  radian. Other deficiency of neutron guides is rather large losses depending on their selected geometry, reflective surface and neutron guide channel assembly defects.

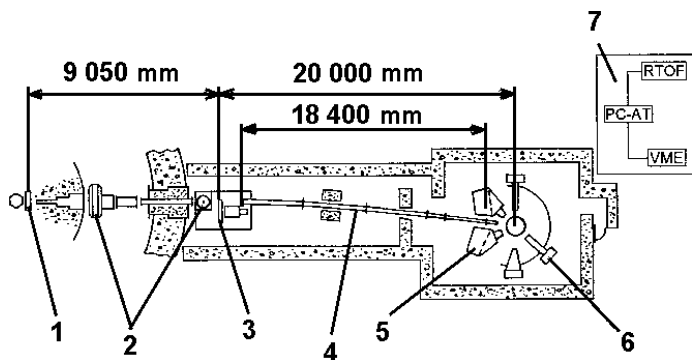
As consequence from mentioned above, in time of creation of experimental complexes, a neutron source, neutron guide, instrument (as a rule, high resolution instruments), the problem exists to estimate correctly origin of losses, methods of their minimization depending on assignment and parameters of the equipment. The collective of laboratory has coped successfully with task, basically thanks to works of Kudryashev V.A. and Kezerashvili V.L. (in that time we were named as the physical equipment development department). The original program of calculation was created, which allowed correctly to estimate neutron guides luminosity losses, their spectral distribution, "to visualize" the losses of neutrons during propagation within the neutron guide [1, 2]. Just with the help of this calculation method the constructional solutions were correctly selected in time of creation of neutron guide physical model of the PIK reactor. These guides surpassed the similar "thermal" neutron guides of ILL in transmission. The experience obtained in the process of production of neutron guide physical model was used to calculate and to estimate the parameters of PIK reactor neutron guide system [2, 3]. Then these neutron guides were produced ( $> 200$  m) at Petersburg Optical-Mechanical Association (LOMO) and were warehoused in the expectation of the PIK reactor. On the basis of neutron guide physical model in cooperation Technical Research Centre of Finland (VTT) the original time-of-flight Fourier-diffractometer was created, which came the base of some world best diffractometers of high-resolution today. When construction the diffractometer, the Fourier-method was modified and advanced in principle (the application of time focusing, electronic focusing and some variants of detector systems are introduced, designed, reworked and updated), the use of neutron guide elements in the Fourier-diffractometer was optimized [5 – 9]. Fig. 1.

All these developments have received rather wide application in Russia (JINR, Dubna) and abroad (Germany, Egypt) [10-12]. The accumulated experience has given us an opportunity to be engaged in increase of neutron guide system luminosity - by creation of focusing neutron guides and supermirrors together with Petersburg Optical-Mechanical Association (LOMO) and Hungarian company "MIRROTRON". So, in 1992 the twenty meter neutron guide with a vertical and horizontal focusing for the Fourier diffractometer at IBR-2 reactor, JINR, was designed and created. It was installed and adjusted by laboratory employees. Also by them in cooperation with Joint Institute for Nuclear Research (JINR), Dubna, and Technical Research Centre of Finland (VTT) the Fourier-diffractometer was installed and debugged as a whole. The record intensity for pulsed source of  $\approx 2 \cdot 10^7 \text{ n} \cdot \text{cm}^{-2} \cdot \text{s}^{-1}$  is obtained at sample position. Now it is one of the world best high resolution diffractometers which was created by use of the first in the world the full scale focusing neu-

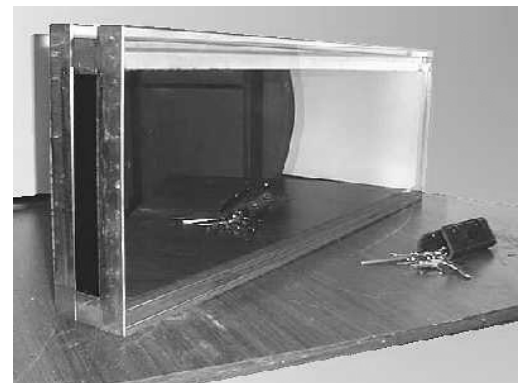


**Fig. 1.** Layout of "Mini-SFINKS" time-of-flight Fourier-diffractometer.  
 1 - in-pile collimator, 4 - Fourier chopper, 6 - back-scattering detector,  
 2 - curved neutronguide, 5 - sample position, 7 - 90°-detector  
 3 - mirror collimator,

tron guide [11, 12], Fig. 2 – 4. The optical section of the focusing neutron guide is presented in Fig. 3, and the scheme of the new designed detector with electronic time focusing is shown in Fig. 4.



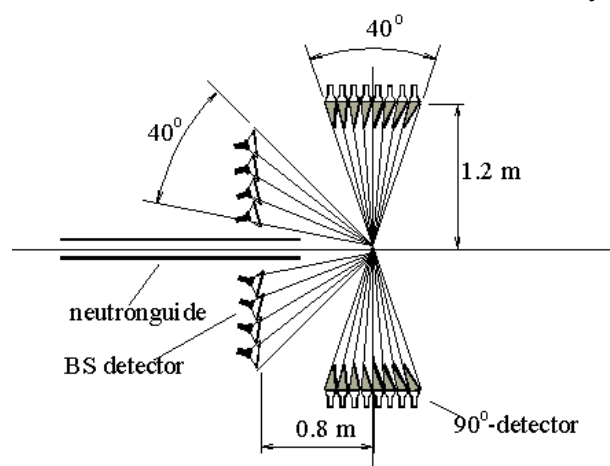
**Fig. 2.** The scheme of neutron reverse time-of-flight Fourier diffractometer installed at the IBR-2 high flux pulsed reactor, channel No.5, with the twice focusing neutron guide:  
 1 - Moderator, 5 - Main detectors,  
 2 - Background chopper, 6 - Low resolution detectors,  
 3 - Fourier chopper, 7 - VME control and operative  
 4 - Neutron guide tube, visualization/analysis and Data  
 acquisition,  
 $k=2.4$  is the gain factor in comparison with a standard neutron guide



**Fig. 3.** Optical section of a focusing neutron guide which set on the channel № 5 of IBR-2 reactor (JINR, Dubna)

The next implementation of the Fourier-method and neutron guide technique was the creation of the Fourier-diffractometer following the order of International Atomic Energy Agency (IAEA) for Nuclear Centre in Egypt. For this diffractometer by our laboratory in cooperation with Technical Research Centre of Finland (VTT) the Fourier-diffractometer equipment was designed, created and installed under "turn-key" in

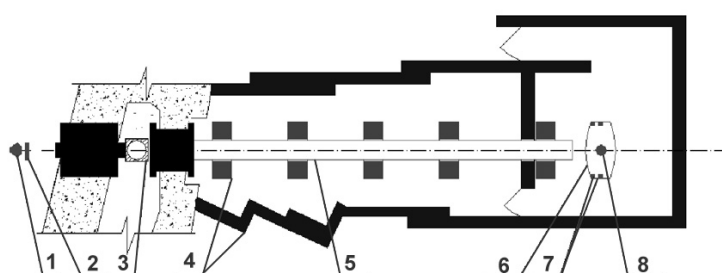
1995, including 25 meter neutron guide [13]. At sample position of this diffractometer the beam intensity of  $2 \cdot 10^6 \text{ n} \cdot \text{cm}^{-2} \cdot \text{s}^{-1}$  was obtained, that allowed to carry out with success high quality structural studies at reactor with a flux in active core of  $10^{13} \text{ n} \cdot \text{cm}^{-2} \cdot \text{s}^{-1}$ . The neutron parameters, obtained at this neutron guide, have exceeded their computed values by 1.4 times.



**Fig. 4.** The scheme of the new width-aperture detector with an electronic time focusing which is planned for use at both Fourier-diffractometers at 5 and 11 beams [8, 9]

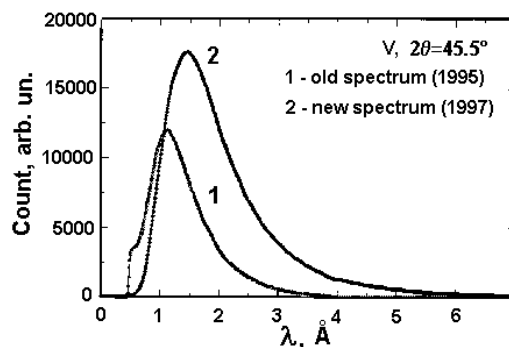
In 1997 at the Fast Pulsed IBR-2 Reactor (JINR, Dubna) for a diffractometer of "Superman" in cooperation with the Hungarian company "MIRROTRON" the unique vertical focusing super mirror neutron guide was created, mounted and justified. The installation of such neutron guide has allowed to increase luminosity in a long-wave part of spectrum by tens of times [14] Figs. 5, 6.

Once again it would be noted fruitfulness of cooperation with Petersburg Optical-Mechanical Association (LOMO), "MIRROTRON", State Optical Institute, "Mirel/Elis" and "SiNaTeh" in development of technology and production of super deeply polished glass and super mirrors. As a result, today we have an opportunity of production of super mirrors with critical angles  $\theta_{cr} \cong 2 \theta_{cr}^{\text{Ni(nat)}}$  and  $3 \theta_{cr}^{\text{Ni(nat)}}$  on mechanically



**Fig. 5.** The scheme of installation of the spectrometer DN-12 at the IBR-2 reactor (JINR, Dubna) and placement of its main units:

- |                             |                         |
|-----------------------------|-------------------------|
| 1 - reactor core,           | 5 - curved super mirror |
| 2 - moderator,              | neutron guide,          |
| 3 - chopper of neutron      | 6 - protection,         |
| beam,                       | 7 - detector system,    |
| 4 - protection against fast | 8 - sample              |
| neutrons,                   |                         |



**Fig. 6.** The spectral distribution of the neutron beam efficiency at vanadium sample before (1) and after (2) installation of the super mirror neutron guide

polished substrates with very high quality of surface: very low roughness parameter, and the macro/micro undulation ones. In cooperation with State Optical Institute (GOI) the effective programs of estimate of super mirrors coatings was created, that has facilitated substantially the development of the technology of creation of supermirror coating.

Again it is necessary to point out, that as a result of this cooperation the industrial polishing know-how of glass surfaces with the roughness at level of  $(4 - 5) \text{ \AA}$  was adopted and debugged, and this know-how is the major factor of production of high quality super mirror coatings. In cooperation with State Optical Institute four unique substrates were produced for the mirror analyzer of neutron beam polarization at IBR-2 reactor reflectometer. The uniqueness of these substrates is the achievement of roughness of  $(2 - 3) \text{ \AA}$  and mirrors sizes of  $(800 \times 100 \times 20) \text{ mm}^3$ . It is very high standard achievement.

Nevertheless our last experience has shown that the optimal solution in case of a wide range production of supermirrors was the use of specially selected "Borofloat" glass.

The following research programs were carried out with help of created tools and in the next order, with the experience of these studies being used for modernization of devices and their components:

1. Rare earth formates  $\text{Re}(\text{DCOO})_3$ ,  $\text{Re}=\text{Y, Ce, Sm, La, Tb, Tm}$  [15 – 18].
2. Hexaborides  $\text{Re}^{11}\text{B}_6$ ,  $\text{Re}=\text{Y, Ce, Sm, La, Nd}$  [19 – 26].
3. High- $T_c$  superconductors [27 – 40].
4. Compounds with anomalies in conducting properties [41 – 49].
5. Pharmacological compound and its components [50 – 51].
6. Modernization of Fourier method [5 – 10, 52, 53].

From the last practical results it is necessary to note the design, creation and adjustment of two vertical focusing neutron guides of the total length of  $\approx 40$  m in JINR (1999) for "Isomer" nuclear-physical facility and specialized Fourier-diffractometer, Fig. 7. Other indispensable diffractometer units (Fourier-chopper, detector) were constructed.

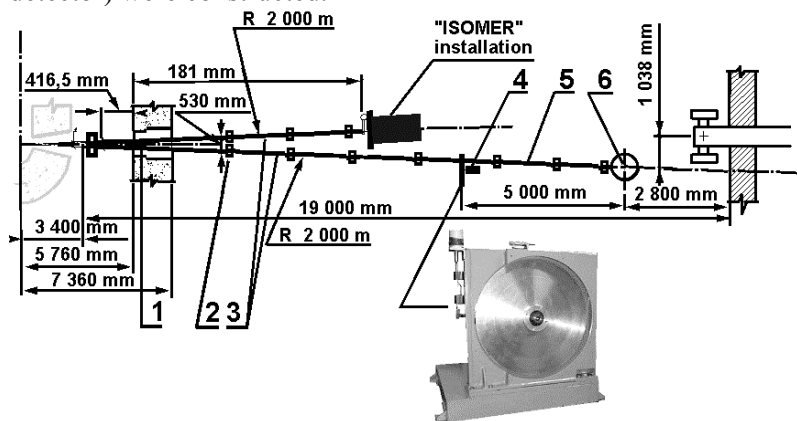


Fig. 7. The scheme of the ISOMER setup at the channel № 11 of the IBR-2 reactor (JINR, Dubna):  
 1 - reactor core,  
 2,3 - collimators,  
 4 - mirror neutron guide,  
 5 - chopper,  
 6 - moderator,  
 7 - neutron counters,  
 8 - Cd cylinder,  
 9 - sample

The creation of neutron guide system (more than 70 m of neutron guides, including 20 m super mirror neutron guide with a horizontal focusing) for the Hungarian reactor at the Budapest neutron centre is in a stage of successful completion. All mirror elements of this neutron guide have roughness parameter at a level of  $4\text{\AA}$ . These programs are realized in close cooperation with the «MIRROTRON» Hungarian company.

A neutron reflectometer (now one of the most occupied instruments of the Hungarian reactor), which was designed, produced and installed under "turn-key" by employees of our laboratory and Automation of Physics Experiments Division, promotes successful cooperation in creation of mirror neutron guide elements. The scheme of the instrument is illustrated in Fig. 8.

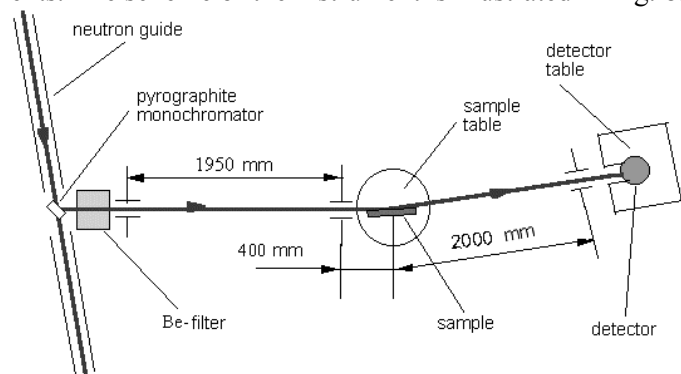
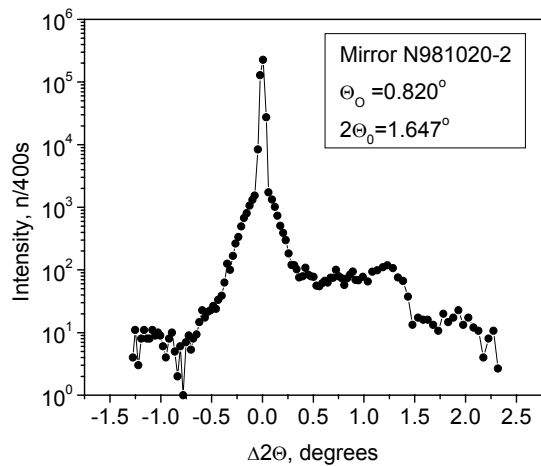


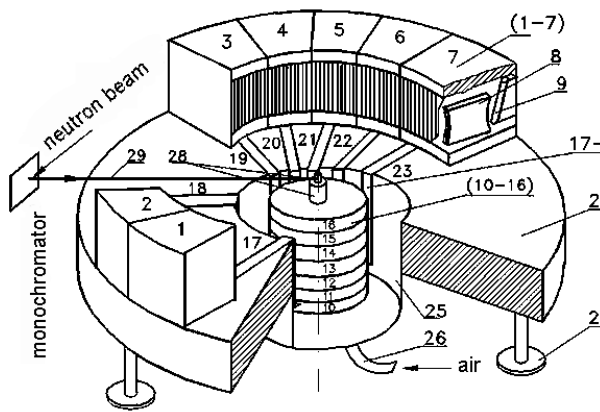
Fig. 8. The scheme of the reflectometer (it is started 14.11.1997) at the reactor of the Budapest neutron centre

The output beam for a reflectometer is realized with the help of pyrographite monochromator with the size of 40 mm in altitude which was set in a breaking of a leading neutron guide with section of  $100 \times 25 \text{ mm}^2$ . The leading neutron guide is included in system of three neutron guides which are set at cold neutrons source. At cross section of beam  $\approx 1 \times 40 \text{ mm}^2$  and angle divergence  $\approx 1.7$  angle minute the initial intensity is equal to  $1500 \text{ n}\cdot\text{s}^{-1}$  at wave length of  $4.3\text{\AA}$ . As an illustration of opportunities of the reflectometer of the Budapest neutron centre the angle distribution of neutron scattering at total reflection from a supermirror Ni-Ti is shown in Fig. 9.

The accumulated experience of cooperation with our Hungarian colleagues gives us an opportunity to participate in various international tenders, supposing creation of neutron guide systems and experimental facilities for new reactors built outside Russia.

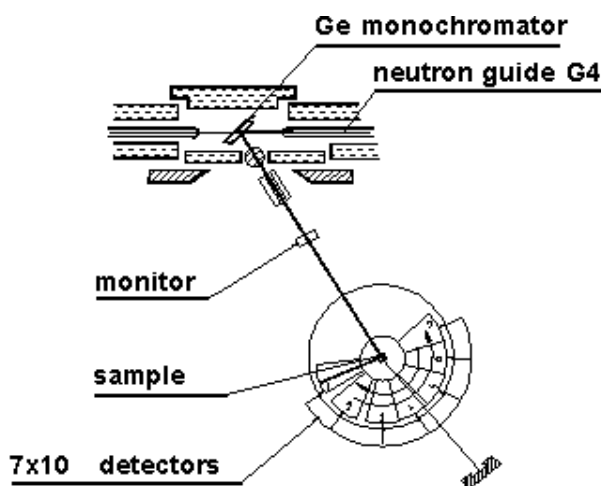


**Fig. 9.** The angle distribution of neutron scattering at total reflection from a supermirror Ni-Ti with a critical angle  $2\Theta$  with Ni



**Fig. 10.** The scheme of the 70-detector section neutron high resolution powder diffractometer.

- 1-7 – detector section with mechanical drives and air cushions;
- 8 – Soller collimator;
- 9 – neutron counter;
- 10-16 – the system of the measurement of the angle positions of the sections based on 19-grade absolute angle code decoder;
- 17-23 – brackets connected sections and the systems of the measurement of the angle positions of the sections;
- 24 – transport plate;
- 25 – assembly tank for arrangement of the systems of the measurements of the angle positions of the sections;
- 26 – entrance of the compressed air;
- 27 – diffractometer supports;
- 28 – sample with sample support or temperature equipment;
- 29 – direction of the incident monochromatic beam



To successful instrument developments of our laboratory it is necessary to attribute the 70-detectors high resolution diffractometer G4.2, which is included in neutron guide system of the ORPHEE reactor hall of Laboratoire Leon Brillouin (LLB, Saclay, France) at the G4.2 site [54], Fig. 10 and 11. For this instrument in our laboratory the focusing Ge monochromator elements were also elaborated. Now the modernization of this diffractometer with the help of a new sample unit is terminated, that, undoubtedly, will extend its experimental opportunities. For diffractometers similar to Russian-French one [54], the model of a focusing monochromator, Fig. 12, which at the present is being prepared to beam trials, is created.

**Fig. 11.** Schematic view of the diffractometer arrangement

**Fig. 12.** Model of focusing monochromator system

Summarizing all the mentioned above, it would be desirable to point out the success of activity on the development of the neutron methods, neutron guides and physical equipment, by demonstration of one statistical information: since 1987 in cooperation with other organizations the Material Research Laboratory has designed, created and installed  $\approx 170$  m of various neutron guides together with instruments, located on them. At the created instruments more than 100 experiments were carried out, the results of which are published in scientific magazines with a high international rating.

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