

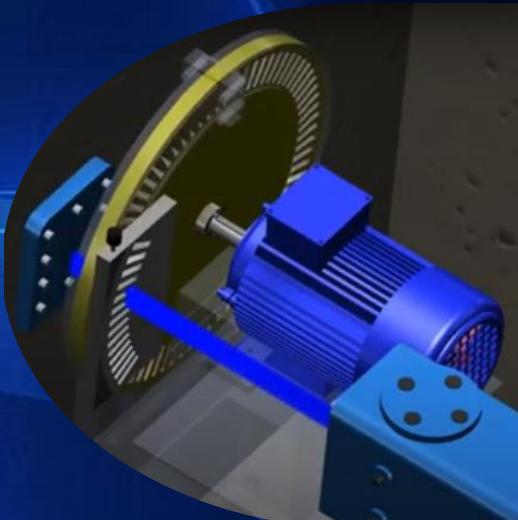
Joint Institute for Nuclear Research  
Frank Laboratory of Neutron Physics

Department of Neutron Investigations  
of Condensed Matter

# Proposals

for Development of  
a Suite of Instruments  
for Condensed Matter Research

at the IBR-2  
Reactor



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for 2026-2030**

**DUBNA 2026**

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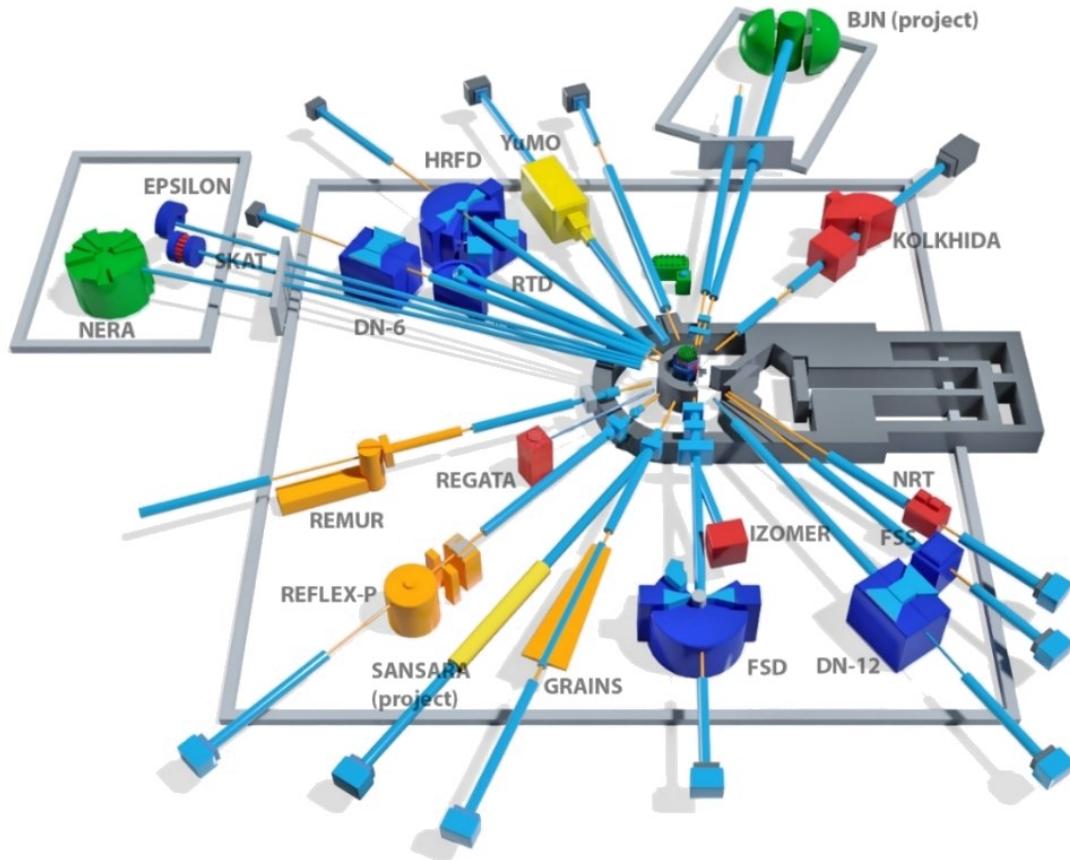
## INTRODUCTION

The IBR-2 high-flux pulsed reactor was commissioned in 1984. At the end of 2006, the reactor was shut down for scheduled maintenance, including the replacement of the core, reactor vessel and movable shields with neutron moderators, resulting in improved performance characteristics and enhanced reliability. These activities were carried out during the period of 2007-2010. Following the successful physical (December 2010) and power (July-October, 2011) startups of the reactor, operation for physics experiments at a nominal power of 2 MW was resumed in 2012 and continued until the autumn of 2021. For the subsequent period, until February 2025, the reactor was in a technical shutdown mode for the replacement of heat exchangers and to obtain a new operating license. Regular operation of the reactor for physics experiments was resumed in February 2025. The implementation of work to maintain and extend the service life of the reactor equipment and its systems has made it possible to create a long-term perspective for conducting neutron research using extracted neutron beams. The IBR-2 technical parameters are presented in the Table. In addition, the reactor is equipped with two cryogenic moderators, which provide an order of magnitude gain in cold neutrons for beamlines 4-11.

### Technical parameters of the IBR-2 reactor before and after modernization

IBR-2 parameter	Before modernization	After modernization
<b>Pulsed thermal neutron flux, <math>n/cm^2/s</math></b>	$5 \cdot 10^{15}$	$5 \cdot 10^{15}$
<b>Average power, MW</b>	2	2
<b>Power in pulse, MW</b>	1850	1850
<b>Fuel</b>	$PuO_2$	$PuO_2$
<b>Number of fuel assemblies</b>	78	69
<b>Maximum burnup, %</b>	6.5	9
<b>Pulse repetition rate, Hz</b>	5, 25	5, 10
<b>Neutron pulse half-width:</b>		
<b>Fast neutrons, <math>\mu s</math></b>	215	240
<b>Thermal neutrons, <math>\mu s</math></b>	320	340

The IBR-2 reactor is equipped with a unique suite of neutron spectrometers, which makes it possible to carry out a wide range of interdisciplinary investigations in the field of condensed matter physics, materials science, chemistry, biology, geophysics, pharmacology, medicine, nuclear physics, ecology, and others. At present, at the IBR-2 reactor, 14 instruments for condensed matter research are in service, including 8 diffractometers, 3 reflectometers, 1 small-angle neutron scattering spectrometer, 1 inelastic neutron scattering spectrometers, and 1 spectrometer for neutron radiography and tomography. Work on a new small-angle scattering and imaging spectrometer is in its final stages. A project to build a new high-luminosity inelastic neutron scattering spectrometer in inverse geometry (BJN) has also been launched.



*Layout of instruments in the IBR-2 experimental hall*

The prevalence of diffractometers is, to some extent, due to historical reasons, and a number of objective factors, such as the development of unique new techniques like Fourier diffractometry, which makes it possible to perform experiments with a very high resolution (down to  $\Delta d/d \sim 0.1\text{-}0.2\%$ ), diffraction studies under high-pressure conditions, and a broad potential for application of diffraction techniques in interdisciplinary scientific investigations—from condensed matter physics to biophysics, geophysics, and medicine.

The IBR-2 spectrometers operate in accordance with the User Program, with proposal calls being issued twice a year. Proposals are peer-reviewed and rated by panels of experts to ensure the quality and scientific merit of research projects. The beam time for experiments is allocated on the basis of the reviews by Expert Committees. A total of about 200 proposals are submitted annually from many countries, with the majority of them being from external organizations.

Among the recently constructed instruments of special note are the DN-6 diffractometer for investigations of micro-samples, which allows experiments with a record-small sample volume of the order of  $0.01\text{ mm}^3$  under extreme conditions (pressures of up to 40 GPa and temperatures in the range of 4 - 300 K); the *GRAINS* multifunctional reflectometer with a *horizontal sample plane*, which *opens up new possibilities for studying liquid and soft interfaces*; the spectrometer of neutron radiography and tomography, which allows for non-destructive testing and studies of the internal structure of materials and products with a spatial resolution of less than 200  $\mu\text{m}$ . On beamline 13, experiments are carried out using the basic configuration of the FSS correlation diffractometer, relocated from GKSS (Germany). To improve the parameters of the diffractometer, the mirror curved neutron guide and the Fourier chopper were replaced.

It should be noted, however, that the current situation with small-angle scattering instruments at IBR-2 is particularly acute. About 30% of the total number of submitted proposals is for one existing

SANS instrument, and the available beam time is significantly less than requested. The construction of a second small-angle instrument and a setup with a spin-echo small-angle scattering mode on the basis of the REFLEX reflectometer will extend the scope of application of this method. Furthermore, the planned development of the new high-luminosity inelastic scattering spectrometer BZN (to replace the old DIN-2PI spectrometer) will make it possible to significantly expand the possibilities for studying the atomic and magnetic dynamics of materials and take them to a qualitatively new level.

The availability of a neutron source with a long service life and world-class parameters, equipped with cold moderators, the intensive development of neutron scattering methods and competing synchrotron radiation scattering techniques, as well as the growing need for the use of neutron scattering techniques in interdisciplinary research, call for further development of the suite of IBR-2 spectrometers with due consideration of all these factors, aimed at expanding experimental possibilities and improving technical parameters.

Furthermore, with the IBR-2 reactor reaching the end of its service life within the next 15-20 years, work is currently underway on the project to develop and build a new pulsed neutron source, which is expected to be commissioned in 2035-2040. The existing IBR-2 instruments could serve as prototypes for the development of advanced neutron spectrometers for the new source.

This booklet contains proposals for the development and modernization of the existing spectrometers and for the design and construction of new instruments in the medium-term perspective up to 2030. The proposals are structured in order of priority. The top-priority ones are expected to be implemented within the budget of the Seven-Year Plan for the Development of JINR for 2024-2030, and their entire list is supposed to be realized if additional funding can be secured. The implementation of the planned work on the development of the complex of IBR-2 spectrometers will significantly expand the scope of condensed matter investigations, substantially improve technical parameters, and bring the experimental possibilities of a number of instruments to a qualitatively new level. In particular, the development and construction of the new BZN spectrometer is expected to increase the intensity of neutrons detected in experiments on condensed matter dynamics by more than two orders of magnitude. The modernization of the detector system of the HRFD high-resolution Fourier diffractometer will make it possible to increase the luminosity in precision structural studies of functional materials by up to 3-5 times. The reconstruction of the Epsilon diffractometer for texture studies will enable the development of a new research area—the study of the texture of cultural heritage objects—which will qualitatively complement investigations conducted using neutron radiography and tomography. Completion of work on the new small-angle scattering and imaging spectrometer will significantly expand experimental possibilities for studying biological and soft nanosystems, as well as develop new methods of energy-selective tomography. The modernization of the SKAT diffractometer will allow a 2-fold increase in the number of conducted experiments. Significant qualitative improvements are expected at other beamline instruments as well.

## 1. LIST OF PROPOSALS

### № INSTRUMENT PROPOSAL TITLE

#### DEVELOPMENT OF OPERATING INSTRUMENTS

1. HRFD	High-Resolution Fourier Diffractometer
2. FSD	Fourier Stress Diffractometer
3. FSS	Fourier Diffractometer
4. RTD	Neutron Diffractometer (Real-Time Diffraction)
5. DN-6	Neutron Diffractometer for Ultrahigh-Pressure Research
6. DN-12	Neutron Diffractometer for Investigations of Microsamples at High Pressures
7. EPSILON	Neutron Diffractometer for Measuring Crystallographic Texture and Internal Strains
8. SKAT	Texture Diffractometer
9. YuMO	Small-Angle Neutron Scattering Instrument
10. GRAINS	Neutron Reflectometer with Horizontal Sample Plane
11. REFLEX	Reflectometer with Polarized Neutrons
12. REMUR	Reflectometer with Polarized Neutrons
13. NRT	Neutron Radiography and Tomography Station
14. NERA	Inverse Geometry Spectrometer for Simultaneous Investigations of Atomic Structure and Dynamics of Condensed Matter

#### NEW INSTRUMENTS

15. BJJN	Inverse-Geometry Inelastic Neutron Scattering Spectrometer
16. SANSARA	Small-Angle Neutron Scattering Instrument
17. NRT_COLD	Neutron Radiography and Tomography Station with Cold Neutrons

## 2. PROPOSALS FOR DEVELOPMENT OF OPERATING INSTRUMENTS

### HRFD – High-Resolution Fourier Diffractometer

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<b>Collaborating organizations:</b>	M. V. Lomonosov Moscow State University, National University of Science and Technology, St. Petersburg State University, Saratov State University, Mongolian Academy of Sciences, Institutes of the Russian Academy of Sciences, Belarusian State University, Tsin Hua National University, China Institute of Atomic Energy

#### 1. Abstract

The High-Resolution Fourier Diffractometer (HRFD) constructed within the framework of the collaboration between FLNP JINR (Dubna), PNPI (Gatchina) and VTT (Espoo, Finland) has been in continuous operation at IBR-2 since 1995. Its initial design, principle of operation and rated parameters are described in detail in [1]. Examples of numerous studies carried out with HRFD as well as some ideas for possible development of the diffractometer are presented in [2, 3]. Over the past period, some of the main units of HRFD have already been replaced, including Fourier chopper, mirror neutron guide, and data acquisition electronics. In 2025, the old detector system was replaced with a new one, which includes a large backscattering detector and a 2D position-sensitive detector on a movable platform.

The extensive experience gained in operating HRFD allowed us to reveal some technical problems in its operation and find ways to rectify them. To further develop the diffractometer, it is necessary to put into service the main detector. First, the background from rescattered neutrons needs to be reduced by adding and improving the external shielding. Second, it is necessary to upgrade the data acquisition system, and replace the MPD240 modules with CAEN Digitizer discriminators, which will allow for more accurate filtering of signals from the new detector and prevent data loss caused by dead time effect. Further work plan includes replacement of the Fourier chopper and sample environment devices.

#### 2. Scientific program, relevance and comparison with the world level

Over the past years, wide experience has been gained in operating HRFD and a scope of problems that can be studied most effectively using the diffractometer has been identified. The HRFD is mainly intended for precision structural analysis of polycrystalline substances with an average unit cell volume of up to  $\sim 500 \text{ \AA}^3$ . Among the most remarkable studies performed with HRFD are investigations of mercury-based high-temperature superconductors with different amounts of oxygen or fluorine [4, 5], origin of a giant isotope effect in manganites [6, 7], magnetic effects in ruthenates [8, 9], structural anomalies in cobaltites [10, 11]. The HRFD is also used to perform analysis of single crystals when its unique  $d_{\text{hkl}}$  resolution is required, e.g., to study phase separation in  $\text{La}_2\text{CuO}_{4+\delta}$  crystals due to low-temperature diffusion of hyperstoichiometric oxygen [12].

The high resolution of HRFD enables us to reliably determine microstrains and characteristic

sizes of coherent blocks of polycrystals under study using the  $d_{hkl}$  dependence of diffraction peak width (Williamson-Hall analysis). The practice of studying microstructural effects using HRFD has shown that its resolution allows microstrains in crystallites to be determined at a level of  $\varepsilon \approx 0.0008$  and higher, and the average sizes of coherently scattering domains – at a level of  $L_{coh} \approx 2500 \text{ \AA}$  and smaller [13].

Starting from 2012, HRFD has been used for experiments to study structural processes in electrodes of Li-ion batteries during charge-discharge processes [14]. The possibility to switch between two different modes of operation (high-intensity and high-resolution) without changing the experiment geometry has significantly added to the success of these investigations. The high-intensity mode was used for acquiring data in real time with reasonable statistics collection time (1-10 min). The high-resolution mode was applied to obtain data on batteries in a steady state, which allowed us to reliably identify emerging structural phases. Since 2016, this approach has been successfully applied to study phase transitions in functional iron-based alloys with giant magnetostriiction [15-20]: upon real-time heating or cooling, phase transitions were studied in the high-intensity mode, while the high-resolution mode was used to determine the phase composition of samples in a steady state at a given temperature and to study the features of their microstructural state.

The greater part of the experiments conducted with HRFD are carried out in cooperation with Russian and foreign scientific organizations for condensed matter, crystallography and materials science research. Thus, it can be stated that HRFD is a successfully operating spectrometer with a well-established research program and a wide community of users.

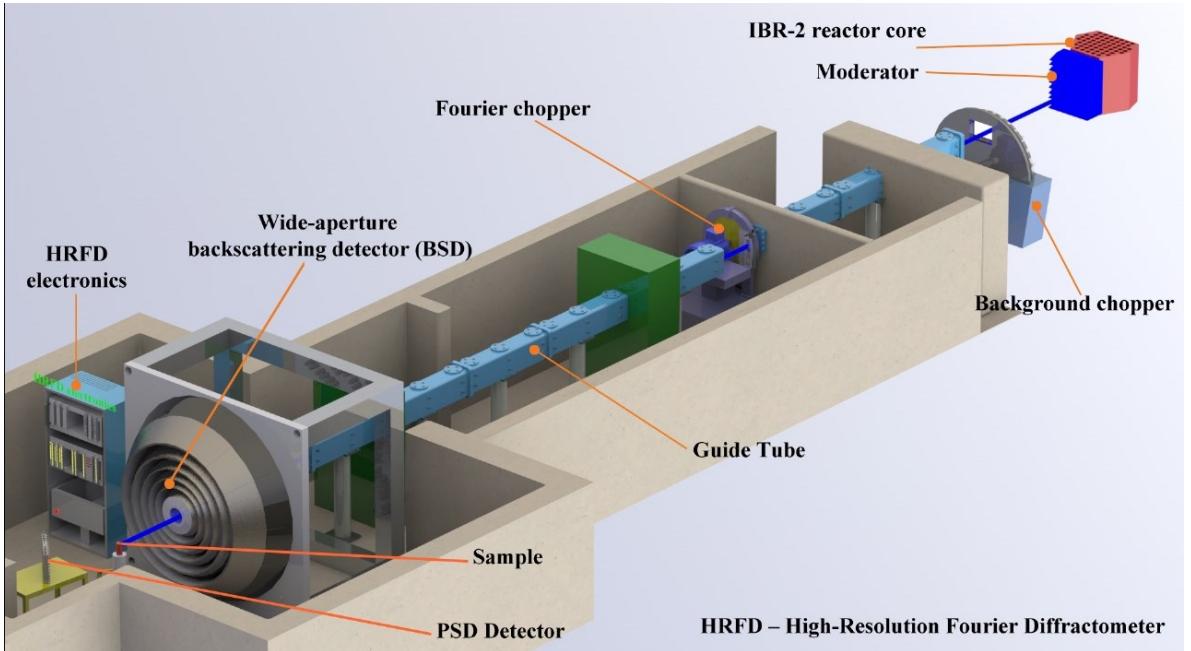
### **3. Scientific and methodological groundwork laid in FLNP JINR**

FLNP JINR is currently the only research center in the world with operating neutron Fourier diffractometers. The main specialists and engineers specializing in this technique work in the Frank Laboratory of Neutron Physics. The HRFD diffractometer has been successfully operated since 1994, undergoing periodic upgrades and replacements of units. Interim results of its operation are summarized in [2, 3].

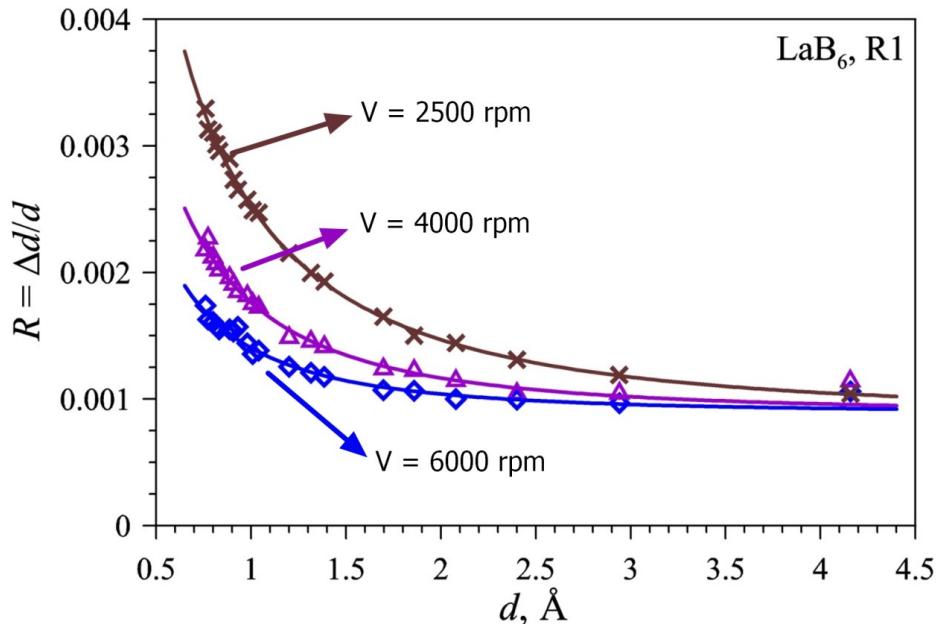
### **4. Current status of the instrument and proposals for its modernization**

The HRFD diffractometer is operated with the application of the correlation method of data collection using a fast Fourier chopper and reverse time-of-flight electronics. The layout of the diffractometer is shown in Fig. 1.

At present, the design of HRFD makes it possible to obtain high-resolution diffraction patterns using a backscattering detector ( $2\theta = 133\text{--}175^\circ$ ) in the  $d_{hkl}$  range of  $0.5\text{--}5 \text{ \AA}$ . Using a position-sensitive detector (2D PSD) placed on a movable platform, it is possible to observe diffraction spectra up to  $d_{max} = 20 \text{ \AA}$ .



**Fig. 1.** Schematic of HRFD. The fast Fourier chopper is positioned immediately behind the wall of the ring corridor. The neutron beam at the sample position is formed by a curved focusing neutron guide. The main backscattering detector and the position-sensitive detector, operating in the low-resolution mode are placed around the sample position. Signals from the detectors are recorded in list-mode to disk memory, and then converted into histograms using standard or reverse (correlation) time-of-flight methods.



**Fig. 2.** The HRFD resolution function measured with a standard  $\text{LaB}_6$  sample at different chopper rotational speeds: 6000, 4000, and 2500 rpm. The figure shows the experimental points and the calculated curve.

The main methodological feature of HRFD is exceptionally high resolution ( $\Delta d/d \approx 0.001$ ) at a rather short flight path from the chopper to the sample position ( $L = 20 \text{ m}$ ). The HRFD resolution is determined by the maximum rotational speed of the fast Fourier chopper, poorly depends on interplanar spacing and enhances with increasing  $d_{\text{hkl}}$  (Fig. 2).

### ***Main parameters of HRFD***

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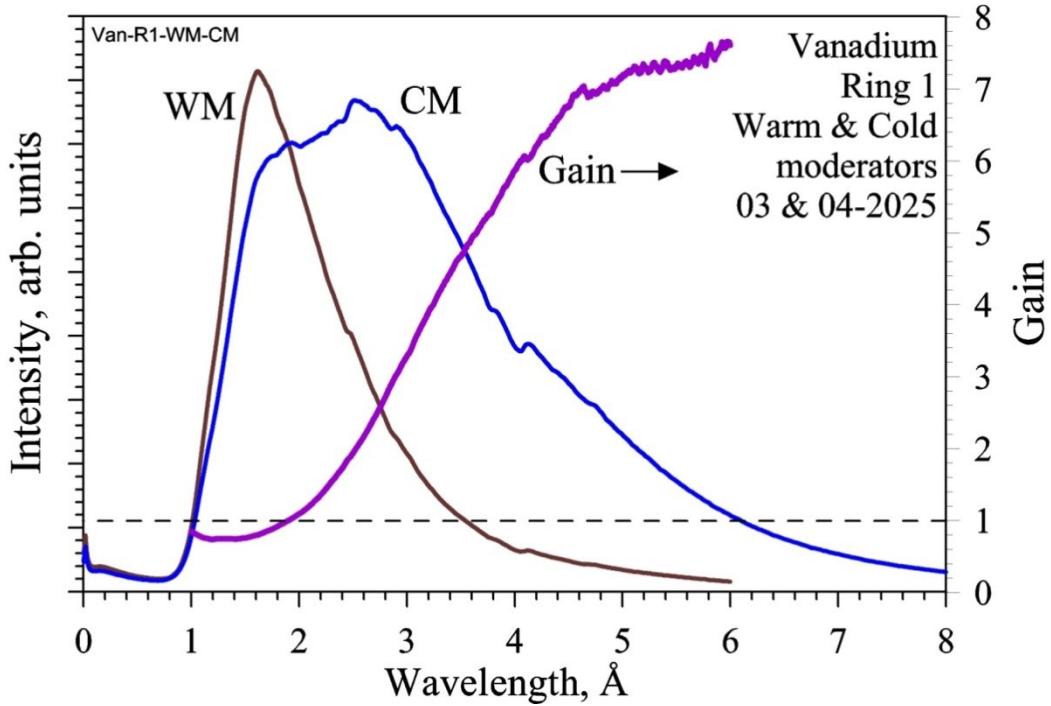
<b>Curved neutron guide</b>	mirror, Ni coating, m=1.75
- length, m	20
- radius of curvature, m	2120
<b>Moderator-to-sample distance, m</b>	29
<b>Chopper-to-sample distance, m</b>	20
<b>Fourier chopper (disk)</b>	high-strength Al alloy
- outer diameter, mm	540
- slit width, mm	0.7
- slit number	1024
- maximum rotational speed, rpm.	6000
- maximum beam modulation frequency, kHz	102.4
<b>Pulse width for thermal neutrons:</b>	
- low-resolution mode, $\mu$ s	340
- high-resolution mode, $\mu$ s	10
<b>Neutron flux at sample position:</b>	
- without Fourier chopper, $n/cm^2 \cdot s^{-1}$	$10^7$
- with Fourier chopper, $n/cm^2 \cdot s^{-1}$	$2 \cdot 10^6$
<b>Detectors:</b>	
- $2\theta = 133 - 175^\circ$ (backscattering)	ZnS, with combined electron-geometric focusing
- $2\theta = 5 - 90^\circ$ (movable PSD)	2D PSD, $^3\text{He}$
<b>Detector resolution <math>\Delta d/d</math> (<math>d = 2 \text{ \AA}</math>):</b>	
- $2\theta = 133 - 175^\circ$ (backscattering)	$1 \cdot 10^{-3}$
- $2\theta = 5 - 90^\circ$ (movable PSD)	$2 \cdot 10^{-2}$
<b><math>d_{hkl}</math>-range, <math>\text{\AA}</math></b>	
- $2\theta = 133 - 175^\circ$ (backscattering)	0.5 - 5
- $2\theta = 5 - 90^\circ$ (movable PSD)	1 - 20

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The modernization of HRFD should be aimed at enhancing the luminosity of the diffractometer, reducing the background level, developing sample environment equipment, and improving Fourier analysis parameters. According to the estimates, this will make it possible to increase the number of conducted experiments, noticeably enhance the precision of the obtained structural information, and significantly expand the possibilities of the diffractometer for carrying out experiments in a wide range of temperatures and other external conditions.

#### ***4.1. Operation of HRFD at a cold neutron source***

In 2020, the IBR-2 reactor was equipped with a CM-201 cold moderator, which supplies neutrons to beamlines 4, 5, and 6. The first spectra from the moderator operating in cryogenic mode were obtained in November 2020 and demonstrated the advantage of the cold moderator over the standard water moderator (Fig. 3). The increased number of long-wavelength neutrons makes it possible to record diffraction peaks at high  $d_{hkl}$  values on HRFD, which is often an extremely important factor when studying magnetic structures. The new backscattering detector for detecting neutrons from the cold moderator enables reliable recording of diffraction peaks at interplanar spacings of 5  $\text{\AA}$  or more in high-resolution mode.



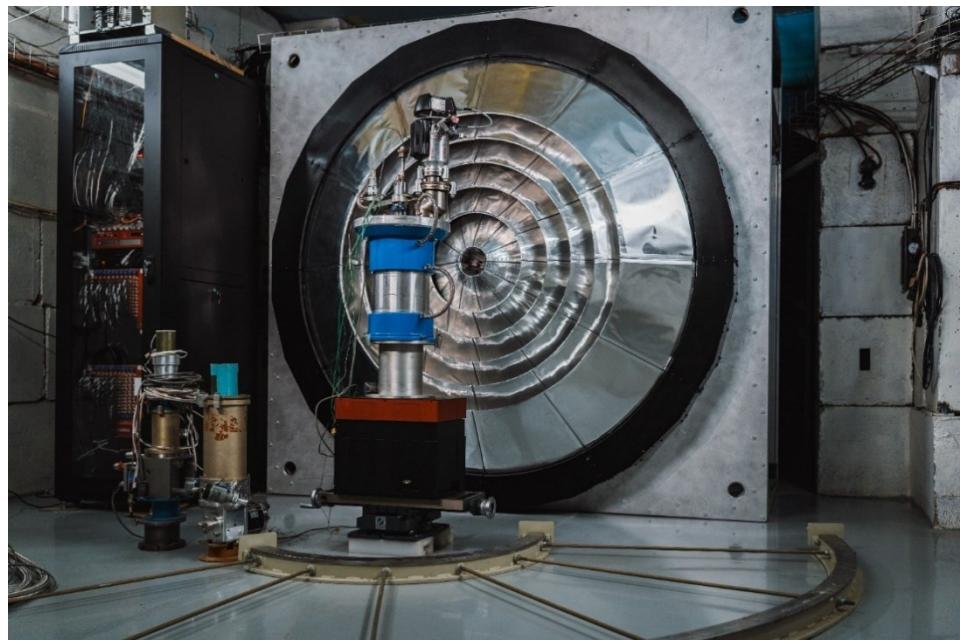
*Fig. 3. Comparison of neutron fluxes for warm and cold moderators.*

#### 4.2. Enhancement of diffractometer luminosity and reduction of background level

During the shutdown of the IBR-2 reactor in 2021–2025, the detector system was upgraded. The main backscattering detectors with a total solid angle of 0.16 sr were replaced with a large-area backscattering detector with a total solid angle of 2 sr. The detector was manufactured in the SC Department using ZnS(Ag)-based scintillators and combined electron-geometric focusing (Fig. 4). These scintillators have significantly lower sensitivity to  $\gamma$ -radiation than the previously used Li-glass, which made it possible to reduce the background and improve the signal-to-noise ratio. Each segment of the detector has a specific shape corresponding to the time focusing. The first experiments demonstrated the high quality of manufacture and alignment of the detector. The corresponding calculated constants matched the experimental ones, which allows the spectra to be summed within one ring without additional corrections. The resolution of the total spectrum from the new detector remains at the same level as the old one,  $\sim 0.0015$ , but on the inner rings, a resolution better than 0.001 can be achieved (Fig. 2). The efficiency of the new detector exceeds that of the old detector system by more than a factor of five.

The increased load on the data acquisition system from the new detector has caused its unstable operation, and therefore it is planned to upgrade it in the near future. Testing of two data acquisition systems (MPD32 and Digitizer) is scheduled for the IBR-2 cycles in the autumn of 2025. The latter system has a more sophisticated filtering of incoming signals with a lot of settings, which will probably make it possible to reduce the background effect. The first experiments showed an increased background level, especially on the outer uncovered ring. To improve the quality of diffraction patterns and reduce background effect, it is necessary to build additional shielding to protect the outer rings from rescattered neutrons. The development of the detector shielding with subsequent manufacturing and installation is planned for the near future.

The planned work should eliminate the identified deficiencies in the operation of the HRFD diffractometer, improve its efficiency and allow it to be included in the User Program for 2026.



*Fig. 4. New backscattering detector at the HRFD diffractometer.*

#### **4.3. Sample environment system**

At present, HRFD is equipped with a helium refrigerator ( $T \geq 4.0$  K) and a high-temperature vacuum furnace ( $T \leq 1200^\circ\text{C}$ ). Recently, there has been a significant increase in the number of beamtime proposals for experiments that require studying extreme processes of external influence on samples, such as extremely rapid cooling. In this case, samples are exposed to extreme conditions outside the experimental setup and delivered frozen. Such experiments, conducted continuously over a wide temperature range, require a shaft cryostat with a temperature of  $T_{\min} \leq 5$  K. Only 20% of requests for experiments without external conditions are made. The share of applications for experiments without the application of external conditions is only 20%.

#### **4.4. Modernization of Fourier chopper**

The available Fourier chopper was manufactured in Hungary in 2016. The chopper disk is made of Al alloy and has 1024 slits filled with  $\text{Gd}_2\text{O}_3$ , and is designed for a maximum rotational speed of  $V_{\max} = 6000$  rpm. A magnetic rotational speed sensor is fixed on the motor shaft. A stator with a similar set of slits non-transparent to thermal neutrons is located next to the disk. Due to the poor stability of magnetic sensors, auxiliary sensors, and a digital control device under radiation, they need to be replaced frequently, which negatively affects the operational stability of HRFD. One more drawback of the available Fourier chopper is an indirect method of acquiring information about the position of the disk relative to the stator. This problem can be overcome by using a design developed for the FSD diffractometer and realized by Airbus, in which the disk and the stator of the chopper have real slits, which allows one to control the parameters of rotation and signals of the "open-closed" type using conventional optical sensors. This chopper is currently operating successfully at the FSD diffractometer and has proven itself to be an excellent choice. In the future, it is planned to manufacture a similar chopper for HRFD.

Since the projected reactor at FLNP JINR will have generally similar characteristics to those of the IBR-2 reactor, the upgraded HRFD diffractometer with minor changes in the configuration of the neutron guide and the location of the chopper can be used at the new neutron source.

## 5. Expected scientific results, comparison with the world level

After the new detector and data acquisition system are put into operation, the luminosity of the HRFD diffractometer will be significantly enhanced. Given the current level of resolution, the HRFD will become one of the best high-resolution diffractometers in the world, comparable, for example, to the POWGEN diffractometer (solid-state detector area – 12 m<sup>2</sup>. SNS, USA). So, the experiment time to get sufficient statistics at a medium resolution ( $\Delta d/d = 0.01$ ) will be decreased by more than a factor of 10, and at a high resolution by a factor of 5 ( $\Delta d/d = 0.001$ ). The modernization of the diffractometer will make it possible to efficiently conduct high-resolution experiments with a small amount of samples (less than 1 g), which is currently almost impossible; to achieve unique characteristics in the accuracy of determining the parameters of the crystal structure of substances, and to study processes with a time resolution of a fraction of a second. An increase in the covered range of interplanar spacings due to the operation of HRFD with a cold moderator will allow us to effectively study complex magnetic structures, as well as organic crystals. The use of a cryofurnace will make it possible to efficiently study phase transitions not only over a wide temperature range, but also in the region of room temperatures. The application of an electromagnet with sample temperature control will significantly expand the range of problems in crystal physics, which can be studied using high-resolution neutron diffraction. Information on the structure, magnetic structure, and microstructure at different temperatures and/or magnetic fields is of key importance for studying various phenomena and effects in crystals.

## 6. Requested resources, costs and time frames of instrument modernization

The units to be upgraded are the backscattering detector, cryofurnace, cryomagnet, precision motorized platform and Fourier chopper. Their costs are given in **Table 1 (Section 4)**. Work on the project is to be carried out by the specialists of the HRFD/FSD Group of the Diffraction Sector with the assistance of the personnel of the SC Department.

## References

1. V.L. Aksenov, A.M. Balagurov, V.G. Simkin, A.P. Bulkin, V.A. Kudrjashev, V.A. Trounov, O. Antson, P. Hiismaki, A. Tiitta “Performance of the high-resolution Fourier diffractometer at the IBR-2 pulsed reactor” *J. of Neutron Research, J. of Neutron Research*, **5** (1997) 181-200.
2. Anatoly Balagurov, Dmitry Balagurov, Ivan Bobrikov, Andrey Bogdzel, Vladimir Drozdov, Andrey Kirilov, Vladimir Kruglov, Sergey Kulikov, Svetlana Murashkevich, Valentin Prikhodko, Vasily Shvetsov, Valery Simkin, Alexander Sirotin, Nikolay Zernin, Valery Zhuravlev, High-resolution neutron Fourier diffractometer at the IBR-2 pulsed reactor: a new concept, *Nuclear Inst. and Methods in Physics Research B* **436** (2018) 263–271.
3. A.M. Balagurov, I.A. Bobrikov, G.D. Bokuchava, V.V. Zhuravlev, V.G. Simkin, Correlation Fourier diffractometry: 20 Years of experience at the IBR-2 reactor. *Phys. Part. Nucl.* **46** (2015) 249-276.
4. Aksenov V.L., Balagurov A.M., V.V.Sikolenko, V.G.Simkin, V.A.Aleshin, Antipov E.V., A.A.Gippius, D.A.Mikhajlova, S.N.Putilin, F.Bouree “Precision neutron diffraction study of the high-T<sub>c</sub> superconductor HgBa<sub>2</sub>CuO<sub>4+δ</sub>” *Phys. Rev. B* **55** (1997) 3966-3973.
5. Abakumov A.M., Aksenov V.L., Antipov E.V., Balagurov A.M. et al., “Effect of fluorination on the structure and superconducting properties of the Hg-1201 phase” *Phys. Rev. Lett.* **80** (1998) 385-388.
6. A.M.Balagurov, V.Yu.Pomjakushin, D.V.Sheptyakov, V.L.Aksenov, N.A.Babushkina, L.M.Belova, A.H.Taldenkov, A.V.Inyushkin, P.Fischer, M.Gutmann, L.Keller, O.Yu.Gorbenko, A.R.Kaul “Effect of oxygen isotope substitution on magnetic structure of (La<sub>0.25</sub>Pr<sub>0.75</sub>)<sub>0.7</sub>Ca<sub>0.3</sub>MnO<sub>3</sub>” *Phys. Rev. B* **60** (1999) 383-387.
7. Balagurov A.M., Pomjakushin V.Yu., Sheptyakov D.V., Aksenov V.L., Fischer P., Keller L., Gorbenko O.Yu., Kaul A.R., Babushkina N.A. “Long-scale phase separation versus homogeneous magnetic state in (La<sub>1-y</sub>Pr<sub>y</sub>)<sub>0.7</sub>Ca<sub>0.3</sub>MnO<sub>3</sub>: A neutron diffraction study” *Phys. Rev. B* **64** (2001) 024420 (10 pp).

8. A.M. Balagurov, S.N. Bushmeleva, V.Yu. Pomjakushin, D.V. Sheptyakov, V.A. Amelichev, O.Yu. Gorbenko, A.R. Kaul, E.A. Gan'shina, N.B. Perkins "Magnetic structure of NdMnO<sub>3</sub> consistently doped with Sr and Ru" Phys. Rev. B, v.70 (1) 014427-1-8, 2004.
9. S.N. Bushmeleva, V.Yu. Pomjakushin, E.V. Pomjakushina, D.V. Sheptyakov, A.M. Balagurov "Neutron diffraction evidence for band ferromagnetism in SrRuO<sub>3</sub>" JMMM **305** (2006) 491-496.
10. Balagurov A.M., Bobrikov I.A., Karpinsky D.V., Troyanchuk I.O., Pomjakushin V.Y., Sheptyakov D.V., Successive structural phase transitions in Pr<sub>0.5</sub>Sr<sub>0.5</sub>CoO<sub>3</sub> in the range 10-1120 K, JETP Letters 88 (2008) 531-536.
11. Balagurov A.M., Bobrikov I.A., Pomjakushin V.Y., Pomjakushina E.V., Sheptyakov D.V., Troyanchuk I.O., Low-temperature structural anomalies in Pr<sub>0.5</sub>Sr<sub>0.5</sub>CoO<sub>3</sub>, JETP Lett. 93 (2011) 263-268.
12. Balagurov A.M., Pomjakushin V.Yu., Simkin V.G., Zakharov A.A. "Neutron diffraction study of phase separation in La<sub>2</sub>CuO<sub>4+y</sub> single crystals" Physica C, 272 (1996) 277-284.
13. A.M. Balagurov, I.A. Bobrikov, J. Grabis, D. Jakovlevs, A. Kuzmin, M. Maiorov, N. Mironova-Ulmane "Neutron scattering study of structural and magnetic size effects in NiO" Materials Science and Engineering, 49 (2013) 012021 (4 pp).
14. I.A. Bobrikov, A.M. Balagurov, Chih-Wei Hu, Chih-Hao Lee, Sangaa Deleg, D.A. Balagurov "Structural evolution in LiFePO<sub>4</sub>-based battery materials: *in-situ* and *ex-situ* time-of-flight neutron diffraction study", Journal of Power Sources 258 (2014) 356-364.
15. A.M. Balagurov, I.A. Bobrikov, S.V. Sumnikov, I.S. Golovin "Dispersed clusters in (Fe, Cr)<sub>3</sub>Al alloys: Neutron time-of-flight diffraction study" Phys. Rev. Materials. **3** (2019) 013608 (1-9).
16. A.M. Balagurov, N.Yu. Samoylova, I.A. Bobrikov, S.V. Sumnikov, I.S. Golovin "The first- and second- order isothermal phase transitions in Fe<sub>3</sub>Ga-type compounds" Acta Cryst. B75 (6) (2019) pp.1024-1033.
17. Balagurov, A.M., Samoylova, N.Y., Sumnikov, S.V., Palacheva, V.V., Golovin, I.S. "Structural and magnetic phase transitions in Fe<sub>3</sub>Ge: A neutron diffraction study" Physical Review Materials, 2023, 7(6), 063603.
18. Balagurov, A.M., Sumnikov, S.V., Cifre, J., Chubov, D.G., Golovin, I.S. "In situ study of order-disorder transitions and anelasticity in Fe-26Al alloy" Journal of Alloys and Compounds., 2023, 932, 167663.
19. A.M. Balagurov, I.A. Bobrikov, S.V. Sumnikov, I.S. Golovin, "Coherent cluster ordering in Fe-xAl and Fe-xGa alloys" Journal of Alloys and Compounds 895 (2022) 162540.
20. S.V. Sumnikov, I.A. Bobrikov, I.S. Golovin, A.M. Balagurov "Bulk vs. surface structural phases in Fe-27Ga alloy" J. Alloys and Comp. 928 (2022) 167116 (1-8).

## FSD – Fourier Stress Diffractometer

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### 1. Abstract

The special-purpose Fourier diffractometer FSD is located on beamline 11a of the IBR-2 reactor in FLNP JINR and intended for measurements of residual stresses in bulk products and novel advanced materials. Regular experiments to measure residual stresses in various industrial products and to study elastic properties of advanced materials, as well as methodological investigations have been conducted on FSD since 2001.

To further upgrade the FSD diffractometer, extend its possibilities and improve its technical characteristics, it is necessary to complete the construction of the detector system and carry out the modernization of a number of its units.

### 2. Scientific program, relevance and comparison with the world level

The investigation of internal mechanical stresses in materials has both fundamental scientific and applied significance. The neutron diffraction technique for stress investigations came into use in the mid-1980s and since then it has become widely applied due to a number of significant advantages over conventional methods. The most important advantage is that neutrons can penetrate matter to a depth of 2-3 cm in steels and up to 10 cm in aluminium. The advantages of neutron diffraction are so great that in recent years, diffractometers for internal stress investigations have been built in almost all advanced neutron centers in the world.

The FSD belongs to a class of high-resolution diffractometers. The high-resolution neutron diffractometer is a complex and expensive instrument, that is why precision neutron diffraction experiments with a very high resolution (at a level of  $\Delta d/d \approx 0.002$  or higher) are conducted only in a few most advanced neutron laboratories in the world. At present, JINR is the only scientific center in Russia where regular world-class neutron diffraction investigations of residual stresses are conducted. The technical characteristics and description of FSD are given in [1, 2]. During the period of the FSD operation, the real potential of the diffractometer in solving various problems has been revealed and the main directions of research have been identified and formed. They are connected with the achieved resolution and luminosity as well as the accessible range of interplanar spacings  $d_{hkl}$ .

The greater parts of the studies are concerned with the determination of residual stresses in industrial products and structures. The most common source of stresses is various technological processes. These studies are of interest for manufacturers from the viewpoint of creating optimum properties of materials and optimization of technological production processes. The results of these

studies help to create optimum residual stress states in different cross sections of the component part and consequently, improve its performance characteristics and service life [3, 4].

Another important direction of research activity is the study of residual stresses and mechanical properties of advanced materials, such as composite or gradient materials, as well as various grades of steel. Within the framework of these tasks, the coexistence of several different phases in one material and their combined influence on the elastic properties and residual stresses in the material are studied. These investigations are important for the creation of materials with tailored physical, chemical and elastic properties. The results of these studies make it possible to design novel advanced materials with targeted properties and behavior. Typical examples are studies of residual stresses and elastic properties in steels, advanced composite and gradient materials [5, 6].

### **3. Scientific and methodological groundwork laid in FLNP JINR**

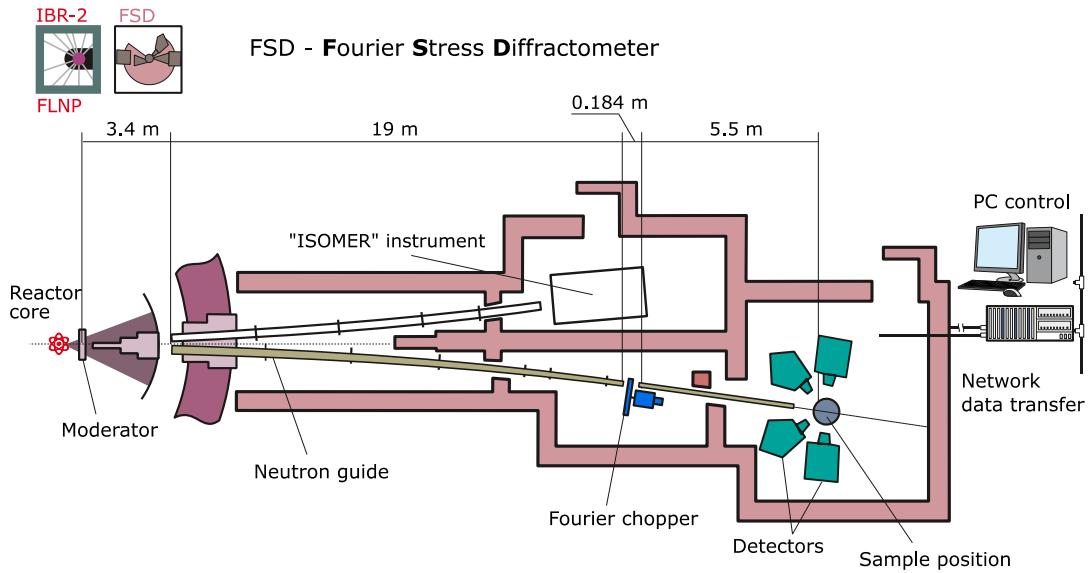
First studies on the determination of residual stresses in bulk products and novel materials started in FLNP JINR in 1993 on the basis of the neutron high-resolution Fourier diffractometer at beamline 5 of the IBR-2 reactor. For this purpose, a new method for analysis of internal mechanical stresses in materials using neutron correlation Fourier diffraction technique on a long-pulse neutron source (IBR-2) has been developed, the necessary equipment has been designed and constructed, test experiments to study stresses in specific materials have been conducted in order to trial the developed technique and determine the potential scope of studies.

A few years later the accumulated operating experience allowed us to start the implementation of a new project aimed at constructing a special-purpose Fourier diffractometer FSD at beamline 11a of the IBR-2 reactor for internal stress studies, which started to operate in 2001. During the period of its operation the FSD diffractometer was used to carry out numerous experiments characterizing the main directions of research in this area: studies of mechanical properties of materials under different loading modes, of various welded joints, structural components of various industrial products, novel advanced materials, gradient structures and composites. The results of these experiments have demonstrated a high efficiency of the Fourier diffractometer in addressing the challenges.

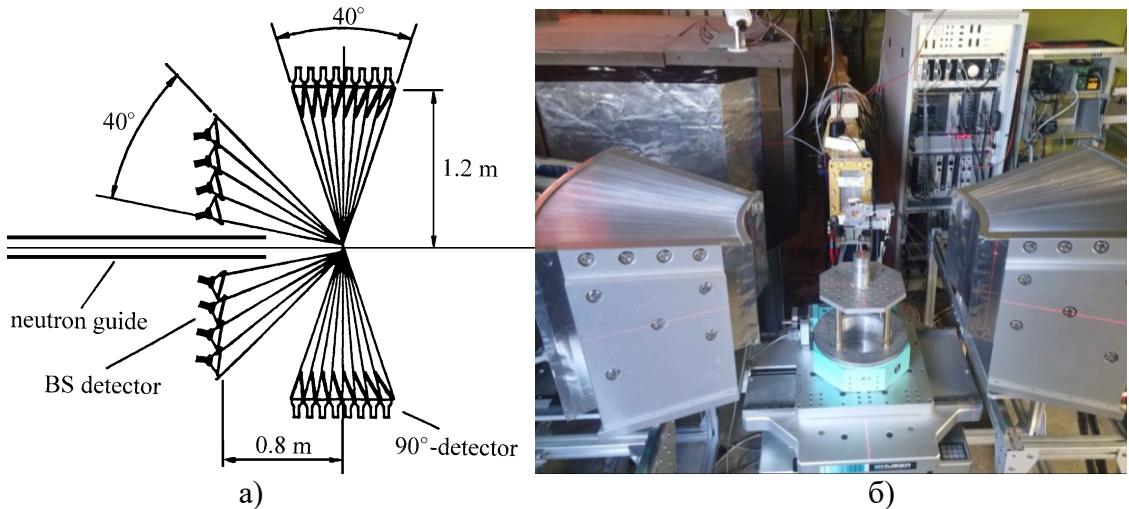
### **4. Current status of the instrument and proposals for its modernization**

#### ***4.1. Current status and main units of FSD***

The FSD diffractometer is located at beamline 11a of the IBR-2 reactor at FLNP JINR. The instrument was developed taking into account the world experience in conducting internal mechanical stress studies in bulk samples and products [7]. The experience of the Petersburg Nuclear Physics Institute, Gatchina (mini-SFINKS diffractometer [8]), GKSS, Geesthacht (FSS diffractometer [9]), and the Frank Laboratory of Neutron Physics, JINR, Dubna (HRFD diffractometer [10]) in the application of the correlation Fourier technique in neutron diffraction was taken into account. All three above-listed instruments are TOF diffractometers using a fast Fourier chopper for primary beam intensity modulation and the RTOF method [11] for data acquisition.



**Fig. 5.** Layout of the FSD diffractometer at the IBR-2 reactor.



**Fig. 6. a)** Schematic of the detector system of FSD. BS – backscattering detectors at  $2\theta=140^\circ$ ,  $90^\circ$ -detector – ASTRA detector system (left and right wings) at  $2\theta=\pm 90^\circ$ . **b)** Sample position at FSD. One can see a HUBER goniometer with a sample, radial collimators in front of  $\pm 90^\circ$ -detectors, BS detector, end part of the neutron guide with a diaphragm for the incident beam.

**The main functional units of the FSD diffractometer are:**

- 1) neutron source (IBR-2 reactor with grooved water moderator) producing thermal neutron pulses of  $\sim 340 \mu\text{s}$  long at a repetition rate of 5 Hz;
- 2) long mirror neutron guide separating the beam from fast neutrons and  $\gamma$ -rays;
- 3) fast Fourier chopper providing neutron beam intensity modulation;
- 4) straight mirror neutron guide shaping the thermal neutron beam on the sample;
- 5) detector system comprising detectors at scattering angles of  $\pm 90^\circ$  and a backscattering detector;
- 6) collimation devices (diaphragms and radial collimator) setting primary beam divergence and defining a gauge volume in the sample;
- 7) HUBER goniometer (up to 300 kg) which can accommodate bulk samples and additional equipment (testing machine, furnace, etc.);
- 8) SONIX+ software package, allowing local and remote control of experiments.

**Main parameters of FSD**

<b>Curved neutron guide</b>	mirror, Ni-coated
- length, m	19
- radius of curvature, m	2864.8
<b>Straight neutron guide</b>	mirror, Ni-coated
- length, m	5.01
<b>Moderator-to-sample distance, m</b>	28.14
<b>Chopper-to-sample distance, m</b>	5.55
<b>Fourier chopper (disk)</b>	High-strength Al-based alloy
- outer diameter, mm	540
- slit width, mm	0.7
- number of slits	1024
- maximum rotational speed, rpm	6000
- maximum beam modulation frequency, kHz	102.4
<b>Thermal neutron pulse width:</b>	
- in low-resolution mode, $\mu$ s	320
- in high-resolution mode, $\mu$ s	9.8
<b>Flux at sample position:</b>	
- without Fourier chopper, $\text{n/cm}^2 \cdot \text{s}^{-1}$	$1.8 \cdot 10^6$
- with Fourier chopper, $\text{n/cm}^2 \cdot \text{s}^{-1}$	$3.7 \cdot 10^5$
<b>Wavelength range, Å</b>	$0.9 \div 8$
<b>Detectors:</b>	
- $2\theta=140^\circ$ (backscattering)	$^6\text{Li}$ , with time focusing
- $2\theta=\pm 90^\circ$	ZnS, with combined electronic and geometric focusing
<b>Detector resolution <math>\Delta d/d</math> (<math>d=2</math> Å):</b>	
- $2\theta=140^\circ$ (backscattering)	$2.3 \cdot 10^{-3}$
- $2\theta=\pm 90^\circ$	$4.0 \cdot 10^{-3}$
<b><math>d_{hkl}</math> range, Å</b>	
- $2\theta=140^\circ$ (backscattering)	$0.5 - 5.4$
- $2\theta=\pm 90^\circ$	$0.6 - 6.7$

**Detector system**

Progress in the development of relatively low-cost correlation electronics based on digital signal processors made it possible to propose a new principle for the development of the FSD detector system, namely, a multi-element detector with combined electronic and geometric focusing [12]. At present, two new ASTRA detectors are installed at the FSD at scattering angles  $2\theta = \pm 90^\circ$ , each comprising seven independent (i.e. with independent outputs of electronic signals) elements [13]. These elements are made on the basis of ZnS(Ag) scintillator with wavelength-shifting optical fibers. The combined use of electronic and time focusing of the scattered neutron beam allows an increase in the solid angle up to  $\sim 0.276$  sr for each ASTRA detector. This sharply increases the detector solid angle while retaining high resolution for the interplanar spacing of  $\Delta d/d \approx 4 \cdot 10^{-3}$ . In addition, the FSD is equipped with one backscattering detector BS with time focusing, which consists of 16 scintillation  $^6\text{Li}$ -elements at a scattering angle  $2\theta = 140^\circ$  (solid angle  $\sim 0.054$  sr). The detector is designed for precision measurements and provides a high level of resolution for the interplanar spacing of  $\Delta d/d \approx 2.3 \cdot 10^{-3}$ .



**Fig. 7. New modules of ASTRA 90°-detectors on FSD.**

### **Beam-forming system**

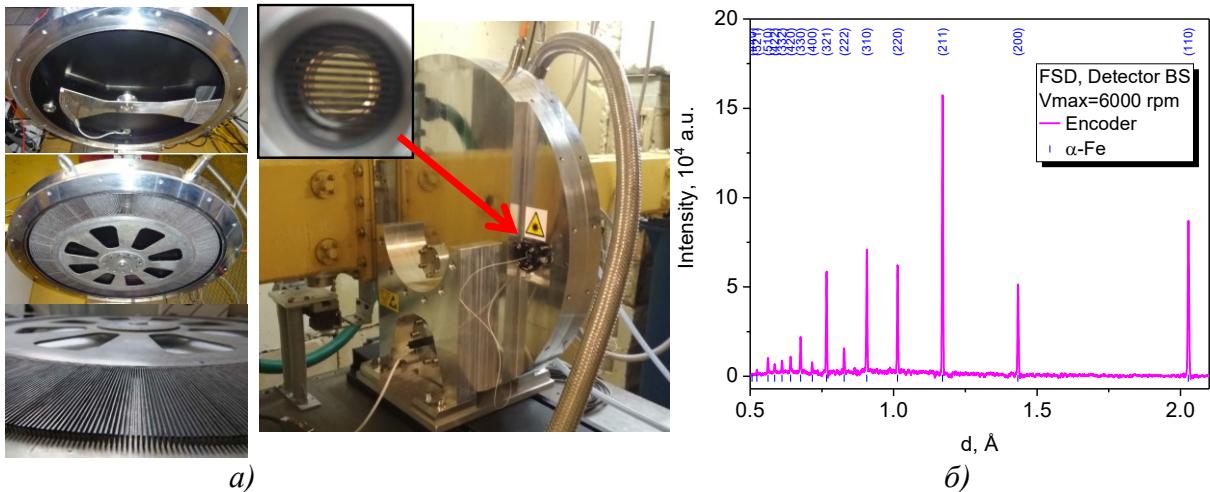
To conduct experiments to study internal stresses, it is necessary to form incident and scattered neutron beams, and thus to define a gauge volume in the sample bulk with characteristic sizes of several cubic millimeters. To form an incident neutron beam of the required dimensions on the sample, an automated diaphragm made of 3-mm thick boron carbide with an adjustable aperture of  $(0\div30)\times(0\div80)$  mm is used, which is installed at the exit of the mirror neutron guide. To form a scattered neutron beam, two unique wide-aperture radial collimators with a spatial resolution of about 1.8 mm are installed in front of the ASTRA 90°-detectors. These collimators feature double convergence (in both the horizontal and vertical planes) and record-breaking angular coverage in the horizontal scattering plane ( $\alpha = 40^\circ$ ), allowing them to completely cover the entire solid angle of the ASTRA detectors. The collimators are mounted on movable platforms with stepper motors and can be remotely moved in and out of their working positions.

### **Fourier chopper**

A number of factors influence the quality of Fourier correlation analysis. One of the most important is the degree to which the Fourier chopper follows the specified rotation frequency distribution law, which depends on the accuracy and stability of operation of the control system. Also important are the depth of modulation of the intensity of the neutron beam passing through the chopper and the stability of operation of the system generating pick-up signals.

As part of the development of the experimental base of the FSD diffractometer, a new Fourier chopper manufactured by Airbus Defence and Space (Germany) was manufactured and installed in 2020. The new chopper is a rotor-stator system in a vacuum housing. A distinctive feature of the chopper is a number of real radial slits cut in the material of the stator plate and rotor disk with an absorbing  $^{10}\text{B}$  coating. A laser beam passing through the chopper slits serves as a pickup signal source, allowing for the measurement of the actual transmission function, which precisely corresponds to the modulation of the neutron beam. An additional source of pickup signals is the HEIDENHAIN optical encoder. The design of the chopper provides more accurate PID control, no absorption or scattering from the material of the stator disk, less vibration and a low level of gamma-background. The chopper is mounted on a high-precision positioner, which allows the Fourier chopper to be remotely moved into and out of the neutron beam as needed. This makes it possible to flexibly formulate a program of experiments on the FSD, quickly switching between the TOF (high luminosity) and RTOF (high resolution) modes. The correlation RTOF spectra measured with both the optical encoder and the laser system show good signal-to-noise ratios and are quite similar, indicating that all systems are properly adjusted. The resolution level of the diffractometer at the maximum speed of rotation of the chopper  $V_{\max} = 6000$  rpm

corresponds to the calculated values:  $\Delta d/d \approx 2.3 \times 10^{-3}$  for the backscattering detector and  $\Delta d/d \approx 4 \times 10^{-3}$  for 90°-detectors at  $d = 2 \text{ \AA}$ .



**Fig. 8.** a) New Fourier chopper installed on the FSD diffractometer. b) High-resolution RTOF spectrum of a standard  $\alpha$ -Fe sample measured using the new Fourier chopper.

### Mirror neutron guide

The mirror neutron guide for FSD is made of high-quality 19-mm-thick K8 borosilicate glass with a Ni (natural isotopic composition) coating. The neutron guide consists of two sections: a curved section with a radius of curvature  $R = 2864.8 \text{ m}$  and a length of 19 m, and a straight section of 5.01 m. The characteristic wavelength of the neutron guide is  $\lambda_C = 1.554 \text{ \AA}$ . The mirror sections of the neutron guide are installed in a steel housing that provides the necessary level of vacuum and stability of the construction. The neutron guide is made conical in the vertical plane with the following cross sections: at the entrance of the curved section –  $10 \times 155 \text{ mm}$ , at the exit of the curved section and at the entrance of the straight section –  $10 \times 91.8 \text{ mm}$ , at the exit of the straight section –  $10 \times 75 \text{ mm}$ .

### Data acquisition system

Since 2025, the FSD diffractometer has been equipped with a new data acquisition system based on the CAEN DT5560SE digitizer, which allows recording all events in the experiment (reactor starts, detector signals, and Fourier chopper pickup signals) in a list mode. Compared to the previously used MPD-32 module, the CAEN digitizer has half the discretization time (8 ns) and a larger data format capacity (64 bits), which allows for a significant improvement in the data accumulation process. In addition, a new, improved algorithm for processing signals from a ZnS scintillation detector has been developed for the CAEN digitizer. The results of the conducted test experiments showed that the CAEN digitizer allows recording high-quality raw data and enables the accurate reconstruction of chopper sweep functions, as well as high-resolution neutron diffraction spectra.

### Sample environment system

The available sample environment equipment integrated into the experiment control system makes it possible to create various conditions at the sample. It includes:

- 1) 4-axis ( $x, y, z, \omega$ ) HUBER goniometer for positioning samples with an accuracy of  $\sim 0.005 \text{ mm}$  and better. The maximum load on the goniometer axis is 300 kg;
- 2) additional XY-motion platform;
- 3) HUBER goniometer rotation module with  $\Delta\varphi = \pm 15^\circ$ ;

- 4) LM-29 testing machine with a maximum load of up to  $\pm 29$  kN for uniaxial tension/compression of samples 30-100 mm long with a screwed cylindrical head in the neutron beam at temperatures ranging from room temperature to 800°C. The main advantage of this machine is almost backlash-free load transmission to the sample. The sample elongation is measured by extensometers from EpsilonTech;
- 5) MF2000 mirror furnace with halogen lamps (maximum temperature up to 1000°C) with temperature control by a Lakeshore controller.

#### ***4.2. Proposals for FSD modernization***

The development of FSD is aimed at enhancing the diffractometer luminosity, reducing the background, improving the Fourier analysis parameters and equipping the diffractometer with supplementary sample environment devices. Estimates show that the accomplishment of these tasks will make it possible to increase the number of conducted experiments several times, significantly improve the accuracy of the data obtained, and significantly expand the possibilities of the diffractometer in performing experiments in a wide range of temperatures and external loads.

##### ***1) Detector system***

For experiments requiring the maximum resolving power of the diffractometer, it is necessary to use backscattering detectors. The available backscattering detector has a small solid angle, limiting the range of possible experiments. Therefore, it is necessary to replace it with two new backscattering detectors based on  $^6\text{Li}$ -glasses with time focusing or based on ZnS(Ag) with combined electron-geometric focusing.

##### ***2) Neutron guide***

To increase the neutron flux on the sample, it is necessary to install a new mirror neutron guide with an advanced multilayer coating of mirrors ( $m = 2 \div 3$ ), which will provide a significantly higher reflection coefficient, less roughness and waviness of the surface. To significantly enhance the neutron flux, the optimal parameters of the neutron guide, including the curvature and the degree of vertical convergence (taper), and the size of the aperture of the outgoing beam will be chosen. It is also planned to solve a number of technical problems related to the extraction of beamlines 11a and 11b from the reactor core area.

#### ***4.3. Operation of the instrument with a cold moderator***

The FSD diffractometer is located on beamline 11a of the IBR-2 reactor, where the cold moderator is not supposed to be placed. The experiments are carried out using a thermal neutron beam.

#### ***4.4. Expected technical parameters after modernization***

The modernization is expected to significantly increase the luminosity of the diffractometer due to an increase in the solid angle of the detectors: up to  $\sim 0.551$  sr for the ASTRA detectors and up to  $\sim 0.2$  sr for the BS detectors. The installation of a new neutron guide will result in a 2-3-fold increase in the neutron flux at the sample position.

#### ***4.5. Relevance of the instrument development for the concept of a suite of spectrometers within the project of a new neutron source at JINR***

For long-pulse neutron sources, the only practical way to achieve high resolution is to use the reverse time-of-flight method in combination with a Fourier chopper (RTOF method) [14], which provides an optimal balance between resolution and luminosity. The experience of upgrading the FSD will be extremely useful for the development of spectrometers on the new neutron source of FLNP.

## 5. Expected scientific results, comparison with the world level

It is expected that after modernization, the FSD diffractometer will be comparable in resolution and luminosity to the best time-of-flight stress diffractometers such as ENGIN-X (ISIS, UK), TAKUMI (J-PARC, Japan), EMD (CSNS, China). An advanced high-resolution wide-aperture detector system will provide a significant gain in luminosity, considerably reduce the spectrum accumulation time, and increase the number of experiments performed in the framework of the User Program. In addition, it will become possible to study in detail the distribution (2D map) of residual stresses in thick industrial samples for a reasonable measurement time. The planned improvements in the resolution and peak shape will enhance the accuracy in determining residual stresses in advanced structural materials and allow reliable determination of the level of microstrains and characteristic crystallite sizes from the profiles of diffraction peaks.

## 6. Requested resources, costs and time frames of instrument modernization

The cost of the FSD components to be manufactured or renewed is given in **Table 2 (Section 4)**. Work on the project will be carried out by the employees of the HRFD/FSD group of Sector № 1 (Diffractometry), NICM Department, and the personnel of the SC Department.

## References

1. G.D. Bokuchava, V.L. Aksenov, A.M. Balagurov et al. *Neutron Fourier diffractometer FSD for internal stress analysis: first results*, Applied Physics A: Materials Science & Processing, 2002, Vol. 74 [Suppl1], pp. s86-s88.
2. G.D. Bokuchava, A.M. Balagurov, V.V. Sumin, I.V. Papushkin, *Neutron Fourier diffractometer FSD for residual stress studies in materials and industrial components*, Journal of Surface Investigation. X-ray, Synchrotron and Neutron Techniques, 2010, Vol. 4, No. 6, pp. 879-890.
3. G.D. Bokuchava, P. Petrov, I.V. Papushkin, *Application of Neutron Stress Diffractometry for Studies of Residual Stresses and Microstrains in Reactor Pressure Vessel Surveillance Specimens Reconstituted by Beam Welding Methods*, Journal of Surface Investigation. X-ray, Synchrotron and Neutron Techniques, 2016, Vol.10, Issue 6, pp. 1143-1153..
4. Gancho Genchev, Nikolay Doynov, Ralf Ossenbrink, Vesselin Michailov, Gizo Bokuchava, Peter Petrov, *Residual stresses formation in multi-pass weldment: A numerical and experimental study*, Journal of Constructional Steel Research, 2017, V. 138, pp. 633–641.
5. G.D. Bokuchava, I.V. Papushkin, V.I. Bobrovskii, N.V. Kataeva, *Evolution in the Dislocation Structure of Austenitic 16Cr-15Ni-3Mo-1Ti Steel Depending on the Degree of Cold Plastic Deformation*, Journal of Surface Investigation. X-ray, Synchrotron and Neutron Techniques, 2015, Vol. 9, Issue 1, pp. 44-52.
6. Przemysław Kot, Andrzej Baczański, Elżbieta Gadalińska, Sebastian Wroński, Marcin Wroński, Mirosław Wróbel, Gizo Bokuchava, Christian Scheffzük, Krzysztof Wierzbanowski, *Evolution of phase stresses in Al/SiCp composite during thermal cycling and compression test studied using diffraction and self-consistent models*, Journal of Materials Science and Technology, 2020, Vol. 36, pp. 176-189.
7. Gizo Bokuchava, *Neutron Fourier Stress Diffractometer FSD at the IBR-2 pulsed reactor*, Crystals, 8 (2018) 318.
8. O.K. Antson, A.P. Bulkin, P.E. Hiismäki, T.K. Korotkova, V.A. Kudryashev, H.S. Kukkonen, V.G. Muratov, H.O. Pöyry, A.F. Schebetov, A.T. Tiita, V.A. Trunov, V.A. Ul'yanov, *High-resolution Fourier TOF powder diffraction: I. Performance of the “Mini-SFINKS” facility*, Physica B: Condensed Matter, 156-157 (1989) 567-570.
9. J. Schröder, V.A. Kudryashev, J.M. Keuter, H.G. Priesmeyer, J. Larsen, A. Tiitta, *FSS - a novel RTOF-diffractometer optimized for residual stress investigations*, Journal of Neutron Research, 2 (1994) 129-141.
10. A.M. Balagurov, *Scientific Reviews: High-Resolution Fourier Diffraction at the IBR-2 Reactor*, Neutron News, 16-3 (2005) 8-12.
11. H. Pöyry, P. Hiismäki, A. Virjo, *Principles of reverse neutron time-of-flight spectrometry with Fourier chopper applications*. Nuclear Instruments and Methods in Physics Research, 126 (1975) 421-433.

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at the IBR-2 Reactor in 2026-2030

12. V.A. Kudryashev, V.A. Trounov, V.G. Mouratov, *Improvement of Fourier method and Fourier diffractometer for internal residual strain measurements*, Physica B: Condensed Matter, 234-236 (1997) 1138-1140.
13. E.S. Kuzmin, A.M. Balagurov, G.D. Bokuchava, V.V. Zhuk, V.A. Kudryashev, *Detector for the FSD Fourier diffractometer based on ZnS (Ag) /<sup>6</sup>LiF scintillation screen and wavelength shifting fiber readout*, Journal of Neutron Research, 10 (2002) 31-41.
14. P. Hiismäki, H. Pöyry, A. Tiitta, *Exploitation of the Fourier chopper in neutron diffractometry at pulsed sources*, Journal of Applied Crystallography, 21 (1988) 349-354.

## FSS — Fourier Diffractometer

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### 1. Abstract

The FSS neutron Fourier diffractometer (Fourier Strain Scanner) successfully operated in 1990-2010 at the FRG-1 steady-state reactor at the GKSS research center (Germany) [1]. The FRG-1 reactor was finally decommissioned in 2010. In view of this, in 2014, the FSS diffractometer was transported to FLNP JINR (Dubna) and placed at the IBR-2 pulsed reactor on beamline 13, which had not previously been used. Over the past few years, a large amount of work has been carried out to develop the beamline infrastructure and adapt FSS to work at a pulsed source [2]. This project outlines plans for further modernization of the FSS Fourier diffractometer.

### 2. Scientific program, relevance and comparison with the world level

The FSS Fourier diffractometer on beamline 13 of the IBR-2 reactor is designed to study internal stresses in structural materials and industrial products using high-resolution neutron diffraction ( $\Delta d/d \approx 5.5 \cdot 10^{-3}$ ). In addition to carrying out scientific research, another important area of scientific and methodological activities on FSS is the further development of the neutron correlation RTOF technique for the analysis of elastic neutron scattering from crystals, as well as the development and testing of new detectors, detector electronics and data acquisition electronics. At the FSS, it is possible to probe new ideas in correlation neutron spectrometry, which can later be applied at the European Spallation Source (ESS) and the new neutron source at FLNP JINR.

The FSS is similar in design to the existing IBR-2 Fourier diffractometers FSD [3] and HRFD [4]. Unlike these diffractometers, the FSS has a medium flight path length between a Fourier chopper and detector ( $L \approx 12.37$  m) and a comparatively low maximum rotational speed of the chopper ( $\sim 2000$  rpm). During the implementation of the project, the main focus will be on increasing the luminosity, improving the resolution, and solving existing technical problems.

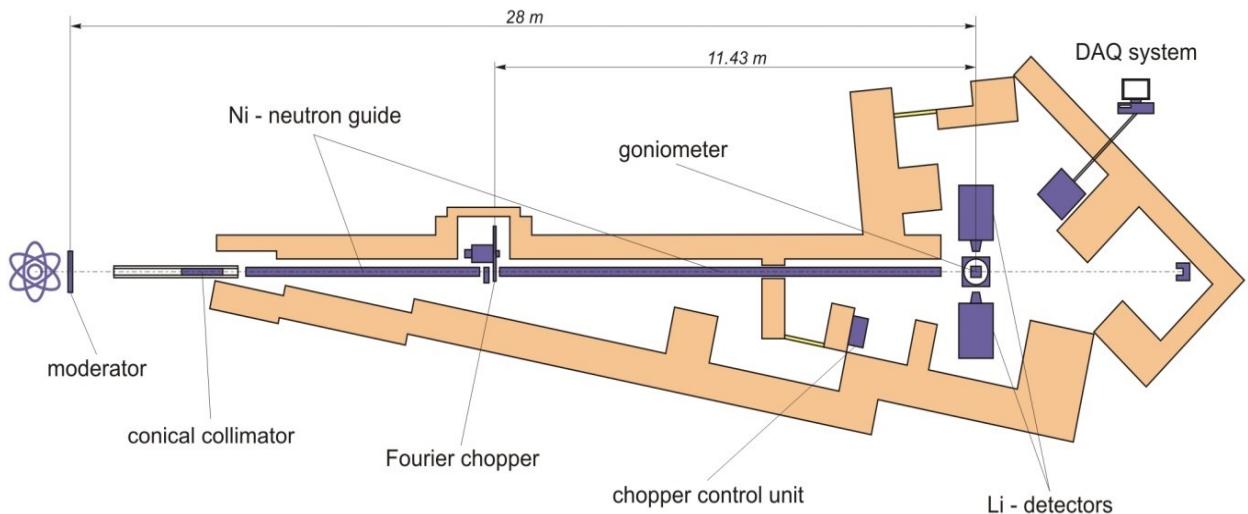
### 3. Scientific and methodological groundwork laid in FLNP JINR

Over the years of operation of the HRFD and FSD diffractometers at FLNP JINR, considerable experience has been gained in using the neutron correlation Fourier technique at a long-pulse neutron source. During this period, a great number of experiments have been conducted on both diffractometers in the fields of structural investigations, physical materials science and engineering sciences.

#### 4. Current status of the instrument and proposals for its modernization

##### 4.1. Current status and main units of FSS

The FSS diffractometer is located on beamline 13 of the IBR-2 reactor at FLNP JINR. In 2014-2019, the major stage of work on the construction of beamline 13, the development of its infrastructure, as well as the installation and adaptation of the main FSS units and performing a series of test experiments was completed. The main units of the diffractometer are steel conical collimator, a mirror neutron guide, a Fourier chopper, a sample assembly, two 90°-detectors, acquisition and control electronics (Fig. 9).



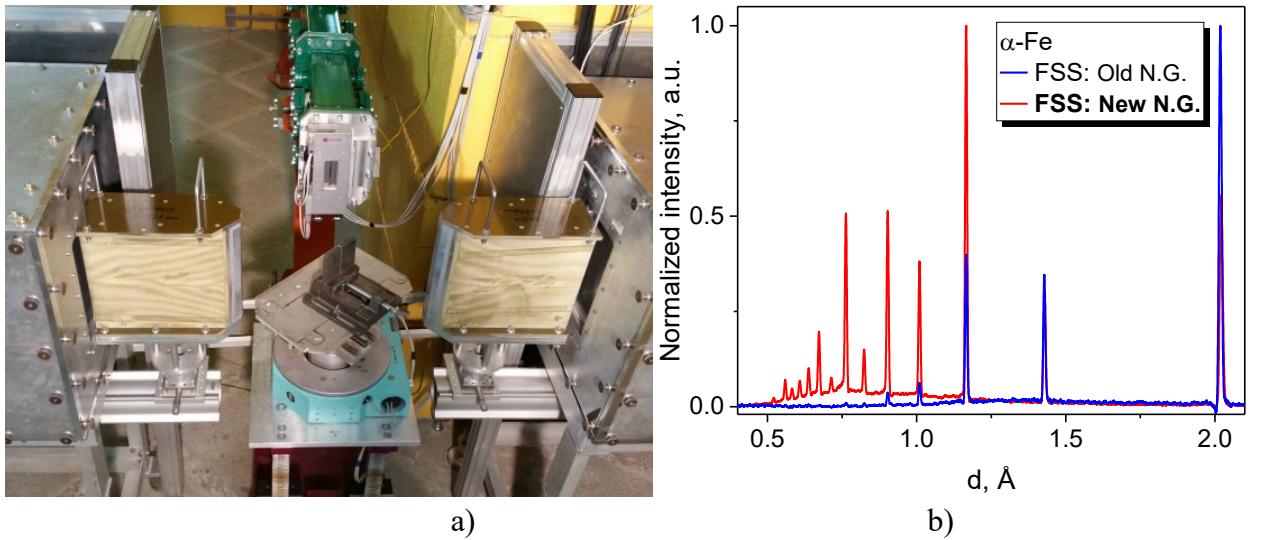
**Fig. 9.** Layout of the Fourier diffractometer on beamline 13 of the IBR-2 reactor.

A steel collimator is installed in front of the mirror neutron guide. It is designed to reduce the radiation load on all subsequent components of the instrument and has a conical shape inside both horizontally and vertically. To fit the geometry of the neutron guide, the inlet and outlet openings of the collimator have a rectangular cross section. The angular dimensions of the openings of the conical collimator are such as to ensure maximum illumination of the input cross-section of the neutron guide relative to the visible surface of the moderator.

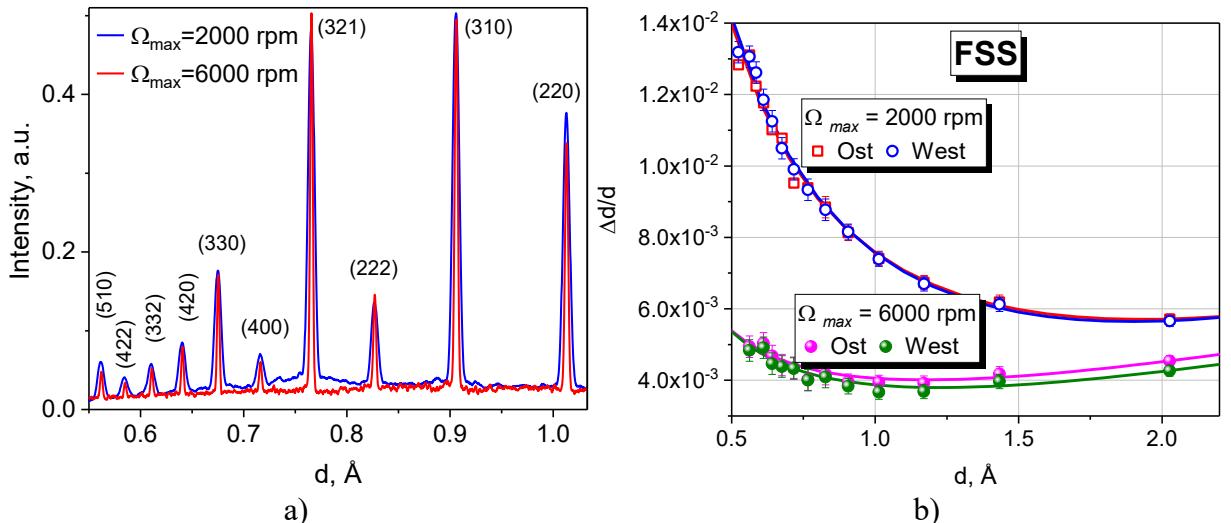
In 2019, to improve the characteristics of the diffractometer, a new curved mirror neutron guide was installed in a steel vacuum housing, which allowed a several-fold increase in the flux of short-wavelength neutrons. The neutron guide is located between the Fourier chopper and the sample place and is designed to remove fast neutrons and  $\gamma$ -rays from the beam, as well as reduce losses when transporting the thermal neutron flux to the sample position. The new neutron guide is plane-parallel in the horizontal direction (opening width – 10 mm) and linearly converging in the vertical plane (inlet and outlet opening heights are 126 mm and 50 mm, respectively). The sections of the new neutron guide have a supermirror Ni/Ti coating,  $m = 2$ . The radius of curvature of the neutron guide is  $R = 1900$  m, while the characteristic wavelength is  $\lambda_c = 0.95$  Å. At the exit of the mirror neutron guide, an automated diaphragm made of boron carbide with an adjustable aperture  $(0\text{--}30)\times(0\text{--}80)$  mm is installed to form an incident beam of the required dimensions.

Fourier chopper. The FSS Fourier chopper is designed for fast modulation of the primary neutron beam and consists of a rotor disk with a diameter of 570 mm fastened to the axis of the engine, and a stator plate fixed to the platform. During the experiment, the disk rotational speed varies from 0 to 2000 rpm according to a certain law (frequency window). The chopper disk has 1024 slits 0.75 mm wide. Metallic gadolinium is used as a neutron absorber. An incremental magnetic encoder is installed on the axis of the electric motor to measure the speed and

acceleration of the disk and generate a pickup signal fed to the RTOF analyzer. A significant disadvantage of this chopper is its low maximum rotational speed ( $\Omega_{\max} = 2000$  rpm) due to its design. Therefore, in 2020, to improve the resolution of the FSS, a new Fourier chopper with a maximum rotational speed of  $\Omega_{\max} = 6000$  rpm (previously used on the FSD diffractometer and upgraded) was installed. The chopper is mounted on a high-precision positioner, which allows the Fourier chopper to be remotely moved into and out of the neutron beam as needed, and quickly switch between the TOF (high luminosity) and RTOF (high resolution) modes. The results of test experiments showed that after replacing the Fourier chopper, the resolution of the FSS was significantly improved due to a reduction in the time component of the resolution function and approximately corresponds to the resolution of the ASTRA 90°-detectors on the FSD diffractometer.



**Fig. 10.** a) Sample position on the FSS. One can see the HUBER goniometer with a sample, radial collimators in front of the Ost and West  $\pm 90^\circ$ -detectors, and the end part of the neutron guide with a diaphragm for the incident beam. b) Comparison of diffraction patterns measured before and after the replacement of the neutron guide.



**Fig. 11.** a) Comparison of high-resolution RTOF diffraction patterns measured using a standard iron sample with the old ( $\Omega_{\max} = 2000$  rpm) and new ( $\Omega_{\max} = 6000$  rpm) Fourier choppers in the range  $d_{hkl} = 0.55 \div 1.033$  Å. b) Comparison of the resolution functions  $\Delta d/d$  measured using a standard iron sample with the old ( $\Omega_{\max} = 2000$  rpm) and new ( $\Omega_{\max} = 6000$  rpm) choppers.

The **detector system** on the FSS consists of two Ost and West  $\pm 90^\circ$ -detectors, assembled from 12 and 15 PEMs, respectively, with NE912 ( $^6\text{Li}$ ) scintillation glasses glued on them. The elements of each detector are located in space in accordance with the time-focusing surface. The range of  $2\theta$  angles in the scattering plane are  $8^\circ$  for the Ost detector and  $10^\circ$  for the West detector. If necessary, radial collimators with a spatial resolution of 1 or 2 mm can be placed in front of the detectors to define a small gauge volume in the depth of the sample. Radial collimators are mounted on movable platforms, which allows them to be remotely moved into and removed from the scattered beam, ensuring the required formation of the scattered neutron beam in the experiment.

The FSS equipment includes a **sample table** consisting of goniometer modules (X, Y, Z,  $\Omega$ ) with a load capacity of 60 kg and additional vertical adjustment. For experiments at high temperatures, an MF2000 halogen-lamp mirror furnace with temperature control by a Lakeshore controller (model 325) is used.

**Data acquisition electronics.** As with other Fourier diffractometers, the FSS is equipped with MPD-32 RTOF-analyzers for list-mode data acquisition, which makes it possible to set the required parameters of the time-of-flight scale and ensure high-precision electronic focusing for individual detector elements. The experiment is controlled using the SONIX+ software package in Windows OS (PC).

#### *Main parameters of FSS*

<b>Curved neutron guide</b>	mirror, Ni/Ti coating (m = 2)
- length, m	17.32
- radius of curvature, m	1900
<b>Neutron beam size at sample position (variable), mm</b>	$(0 \div 10) \times (0 \div 50)$
<b>Moderator-to-sample distance, m</b>	26.5
<b>Chopper-to-sample distance, m</b>	11.43
<b>Fourier chopper (disk)</b>	high-strength Al alloy
- outer diameter, mm	570
- slit width, mm	0.75
- number of slits	1024
- max. rotational speed, rpm.	6000
- max. beam modulation frequency, kHz	102.4
<b>Thermal neutron pulse width:</b>	
- in low-resolution mode, $\mu\text{s}$	340
- in high-resolution mode, $\mu\text{s}$	9.8
<b>Neutron flux at sample position, <math>\text{n}/\text{cm}^2 \cdot \text{s}^{-1}</math></b>	$1.2 \cdot 10^6$
<b>Detectors (<math>2\theta = \pm 90^\circ</math>)</b>	$^6\text{Li}$ , with time focusing
Detector resolution $\Delta d/d$ ( $d = 2 \text{ \AA}$ )	$4 \cdot 10^{-3}$
<b><math>d_{\text{hkl}}</math>-range, <math>\text{\AA}</math></b>	$0.5 \div 3.5$

#### ***4.2. Proposals for modernization of FSS***

The FSS project is planned to be implemented in two stages. In the framework of stage I, the major part of work on the adaptation of the diffractometer at the IBR-2 beamline 13 was completed, and the first test experiments were performed. At this stage, the available units of the FSS diffractometer were mainly used. The plan of activities for stage II (2026-2031) for the modernization of the FSS involves work on the enhancement of the diffractometer luminosity, reduction of the background, improvement of the Fourier analysis parameters and equipping the diffractometer with additional sample environment devices.

##### ***1) Detector system***

To increase the luminosity of the diffractometer, it is necessary to develop and construct a new wide-aperture ZnS(Ag)-scintillator-based detector system with a combined use of electronic and time focusing at scattering angles of  $2\theta = \pm 90^\circ$ . For this purpose, it is planned to use a detector system design similar to the multi-module ASTRA detectors on the FSD diffractometer. Each  $90^\circ$ -detector of FSS will cover a range of angles in the horizontal scattering plane of  $\Delta(2\theta) = 30^\circ$  and in the vertical (azimuthal) plane of  $\Delta\phi = 24^\circ$ . The total solid angle of the new detector system will be approximately 0.52 sr.

##### ***2) Fourier chopper***

To improve the quality of correlation analysis, it is planned to replace the Fourier chopper with an advanced version similar to that developed for the FSD diffractometer—with a vacuum housing, stable pick-up signals, cut-out slits in the rotor and stator, high rotational speed (up to 6000 rpm), flexible control system, and equipped with a high-precision mechanical positioner.

##### ***3) Beam-forming system***

The proposed detector system requires the installation of wide-aperture multi-slit radial collimators in front of  $90^\circ$  detectors. This system will make it possible to regulate the shape of the incident and scattered neutron beams, and thus, to define a gauge volume with characteristic dimensions of several cubic millimeters inside the sample under study. The collimators will be equipped with movable platforms with stepper motors, which will allow them to be remotely moved in and out of their working position.

##### ***4) Goniometer***

For large-sized samples and additional equipment, a new goniometer ( $X, Y, Z, \Omega$ ) with a wide range of linear displacements ( $\sim 200$  mm) and a larger load capacity ( $\sim 300$  kg) will be installed.

##### ***5) Data acquisition system***

For list-mode data acquisition at the FSS, the CAEN digitizer is planned to be used. It features a resolution of 8 ns and a large data format (64 bits), which improves the correlation data acquisition process. In addition, for the CAEN digitizer, a new, improved algorithm for processing signals from the ZnS scintillation detector was developed by the specialists of the SC Department. Test experiments conducted at the FSD in 2025 demonstrated that the CAEN digitizer allows registering high-quality raw data and enables accurate reconstruction of chopper sweep functions and high-resolution neutron diffraction patterns.

#### ***4.3. Operation of the instrument with a cold moderator***

The majority of structural materials studied at the FSS have a relatively small unit cell parameter, so the greater part of the observed diffraction peaks are in the region  $d_{hkl} \approx 0.6 - 2.5$  Å. In addition, the installation of a cold moderator for IBR-2 beamline 13 is not planned. Thus, experiments at the FSS will be carried out using thermal neutrons.

#### **4.4. Expected technical parameters after modernization**

After modernization, the FSS diffractometer will have an advanced wide-aperture detector system ( $\sim 0.52$  sr) with a high level of resolution ( $\Delta d/d \approx 4 \cdot 10^{-3}$ ), which will significantly improve the accuracy of determining the structural parameters of various structural materials.

#### **4.5. Relevance of the instrument development for the concept of a suite of spectrometers within the project of a new neutron source at JINR**

For long-pulse neutron sources, the only practical way to achieve high resolution is to use the reverse time-of-flight method in combination with a Fourier chopper (RTOF method), which provides an optimal balance between resolution and luminosity. The experience gained from upgrading the FSS will be extremely useful for the development of spectrometers for the new neutron source at FLNP.

### **5. Expected scientific results, comparison with the world level**

At present, all the unique Fourier diffractometers in the world (HRFD, FSD and FSS) operate on the IBR-2 pulsed reactor. These instruments provide an optimal balance between luminosity and resolution in diffraction experiments on a long-pulse neutron source, so further development of these Fourier diffractometers should be given special attention. It is expected that after modernization, the main parameters of the FSS (resolution, luminosity) will be comparable to the parameters of the FSD diffractometer. Thus, the improved characteristics of the FSS will allow us to continue work on the further development of the correlation technique, as well as to test new equipment (detectors, detector electronics, and data acquisition electronics). In addition, the gain in the luminosity and the high-precision beam-forming system will enable experiments to study residual stresses in various industrial samples within a reasonable exposure time, as well as accurately determine the level of microstrains and characteristic crystallite sizes from the profiles of diffraction peaks.

### **6. Requested resources, costs and time frames of instrument modernization**

The costs of the FSS units to be manufactured and upgraded are given in **Table 3 (Section 4)**.

## **References**

1. J. Schröder, V.A. Kudryashev, J.M. Keuter, H.G. Priesmeyer, J. Larsen, and A. Tiitta, FSS-a novel RTOF-diffractometer optimized for residual stress investigations. *Journal of Neutron Research*, 1994, 2(4), pp. 129-141.
2. Bokuchava, G.D., Kruglov, A.A., Papushkin, I.V., et al., Neutron Fourier Stress Diffractometer FSS at the IBR-2 Reactor: Results of Upgrading and Prospects for Further Development, *Journal of Surface Investigation*, 2022, 16(2), 191–200.
3. Gizo Bokuchava, Neutron Fourier Stress Diffractometer FSD at the IBR-2 pulsed reactor, *Crystals*, 8 (2018) 318.
4. A.M. Balagurov, Scientific Reviews: High-Resolution Fourier Diffraction at the IBR-2 Reactor, *Neutron News*, 2005, 16-3, pp. 8-12.

## RTD — Neutron Diffractometer (Real-Time Diffraction)

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### 1. Abstract

Modern advances in technology and microelectronics place increasing demands on the creation of materials with improved functional characteristics both under normal conditions and in the processes of transition of substances from one thermodynamic phase to another under changing external conditions (temperature, pressure, etc.). Establishing the relationship between the structural features of materials and their physical characteristics is an important scientific task, the solution of which will lead to an understanding of the microscopic mechanisms and processes responsible for the formation of desired properties, as well as to the unlocking of their full potential under the influence of external factors. The RTD (real-time diffraction) neutron diffractometer is used to address such challenges.

The advantage of RTD is the possibility to follow the kinetics of processes in condensed matter in real time with a minimum measurement time interval during which the necessary statistics can be collected. The experience gained during the operation of the RTD allows us to outline ways for its further development.

### 2. Scientific program, relevance and comparison with the world level

The RTD diffractometer (Real Time Diffraction) [1, 2] is an experimental instrument located on beamline 6a of the IBR-2 pulsed reactor. Figure 12 illustrates a general view of the instrument, including the layout of the main components, designed according to the classical scheme of a time-of-flight spectrometer for elastic neutron scattering. The specialization of the RTD is determined by its interplanar spacing resolution ( $\Delta d/d \sim 0.01$ ) and the neutron flux at the sample position ( $\sim 5 \times 10^6 \text{ n/cm}^2/\text{s}$ ). These characteristics ensure the efficiency of the instrument in studying the structural properties of materials in real time.

The acquisition of experimental data is performed by:

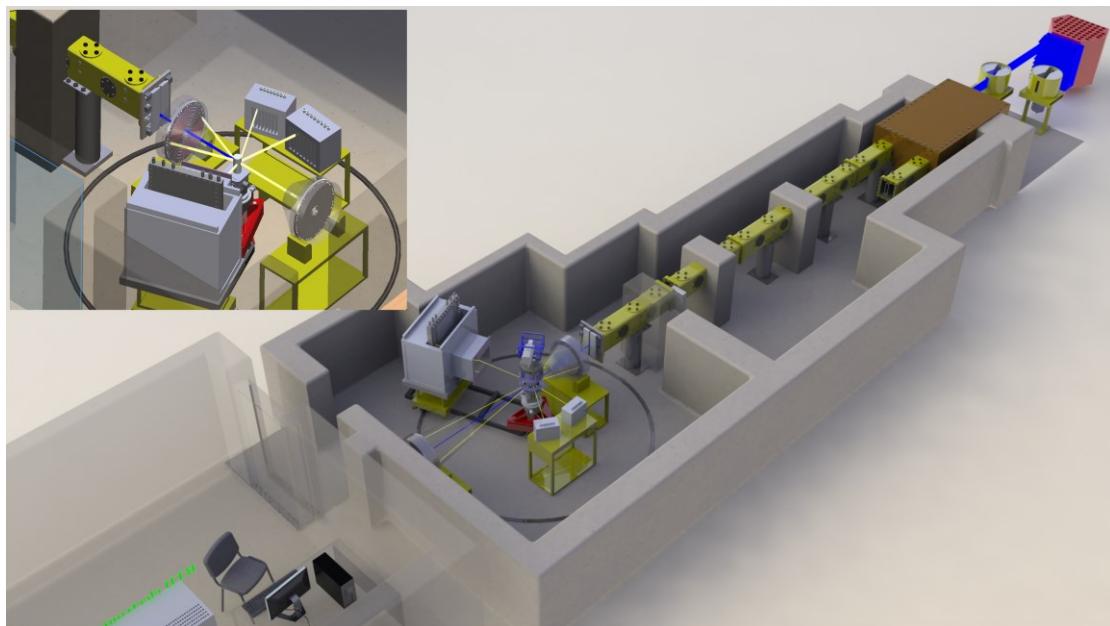
- 1) 8-ring backscattering detector ( $2\theta = 156^\circ - 171^\circ$ ) in the range of interplanar spacings  $d_{hkl} = 0.5-6 \text{ \AA}$ ;
- 2) 9-ring detector for small scattering angles of  $2\theta \sim 1.0-4.5^\circ$  divided into 16 independent sectors, additional measurement of azimuthal coordinates of scattered neutrons in the  $d_{hkl}$ -range from 15  $\text{\AA}$  to 300  $\text{\AA}$  and greater;
- 3) three detector modules comprising 8 counters each at  $2\theta \sim 30^\circ - 138^\circ$  in the range of  $d_{hkl} = 0.5 - 23 \text{ \AA}$ ;

4) 2D position-sensitive detector (PSD) with a variable angle  $2\theta \sim 5^\circ - 140^\circ$ , diffraction is observed up to  $d_{\max} = 80 \text{ \AA}$ .

The 2D PSD makes it possible to perform experiments with single-crystal samples. Moreover, the combination of the 2D detector and TOF technique for collecting diffraction data opens up the possibility of studying the effects of diffuse scattering in single crystals and analyzing deviations of the local structure from the long-range order.

Due to its medium resolution and wide range of interplanar spacings ( $d_{\text{hkl}} \sim 0.5 - 300 \text{ \AA}$ ), the high-luminosity RTD diffractometer can be used to specify the crystal structure of polycrystals of medium complexity. The use of high- and low-temperature add-on modules allows for more detailed research into crystal and magnetic structures in the region of phase transitions in the temperature range from 4 to 1400 K.

Thus, the RTD is an efficiently functioning spectrometer, on the basis of which scientific research is carried out in cooperation with organizations and a wide community of users from Russia, Azerbaijan, Belarus, Romania, Serbia and other countries.



*Fig. 12. Layout of the RTD diffractometer.*

### 3. Scientific and methodological groundwork laid in FLNP JINR

With the start of operation of the IBR-2 reactor (mid-1980s), systematic studies of a wide range of materials and phenomena were initiated, covering the study of cement hydration, the kinetics of water sorption/desorption by lipid membranes [3], solid-phase synthesis of HTSC compounds [4], the effect of H<sub>2</sub> on Y-123 HTSC [5], the kinetics of phase transitions in TiD<sub>x</sub> [6] and the phase transformations of high-pressure metastable ice [7].

The high luminosity ( $\sim 5 \times 10^6 \text{ n/cm}^2/\text{s}$ ) and wide wavelength range (from 1 to 18  $\text{\AA}$ ) of the RTD diffractometer allow obtaining information in the range of interplanar spacings from 0.5 to 300  $\text{\AA}$ . Over the past years, the diffractometer has been successfully used to study a variety of objects and processes in real time, including nanomaterials [8, 9], crystalline [10] and magnetic structures, chemical reactions, phase transitions (crystallization, solidification, hydration/dehydration kinetics), diffusion of light elements in ceramics, domain structure and diffuse scattering in single crystals, as well as long-period [3], incommensurate and modulated structures. Test experiments [11] were conducted to study structural transformations in electrode materials (both in specialized electrochemical cells and in commercial lithium-ion batteries). The

analysis of the scattering intensity made it possible to track the evolution of the phase composition of the electrodes and the lithium content in the cathode. Studying the positions of diffraction peaks, in turn, allowed us to obtain data on changes in the sizes of the unit cell during cyclic charging and discharging.

#### 4. Current status of the instrument and proposals for its modernization

##### 4.1. *Information on the current status of the instrument*

The diffractometer is equipped with the following detector systems:

- Three detector modules with eight SNM-17 counters each for studying the crystal and magnetic structure of powders. The counters (18 mm in diameter and 180 mm in length) are filled with  $^3\text{He}$  under a pressure of 8 atm.
- Ring detector with eight concentric rings at large scattering angles (156-171°) (backscattering).
- 2D position-sensitive detector (active area of  $225 \times 225$  mm, spatial resolution of  $\sim 2$  mm) for studying single crystals and ordered structures with large interplanar spacings.
- Ring detector with nine concentric rings and azimuthal sensitivity at small scattering angles of 1.0-4.5° (operated in adjustment and test modes).

The equipment of the RTD diffractometer includes a three-axis goniometer, a low-temperature add-on module (4-320 K) for a closed-cycle refrigerator and high-temperature furnaces (300-1000 K).

The characteristics of the diffractometer and its sample environment equipment make it possible to study [8, 12 - 21]:

1. processes in real time such as:
  - a) solid-phase chemical reactions,
  - b) crystallization,
  - c) hydration - dehydration,
  - d) phase transitions;
2. crystal structure of powders and single crystals in a wide temperature range from 4 K to 1000 K;
3. magnetic structure;
4. phase transitions;
5. diffuse scattering in imperfect crystals;
6. domain structures;
7. superstructure reflexes of low intensity ( $\sim 0.1\text{-}0.01\%$  of the main peak intensity) in modulated structures;
8. low-dimensional structures with a large unit cell;
9. incommensurate modulated magnetic structures.

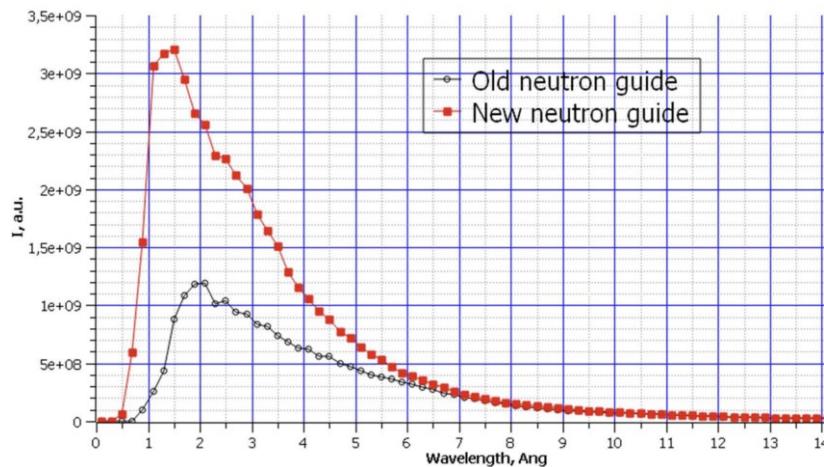
**Main parameters of RTD**

<b>Neutron guide</b>	Ni, mirror
<b>Neutron guide cross-section</b>	15 mm × 180 mm
<b>Moderator-to-sample distance</b>	23.85 m
<b>Sample-to-detector distance</b>	0.15 - 2.0 m, variable
<b>Neutron flux at sample position</b>	$\sim 5 \times 10^6$ n/cm <sup>2</sup> /s
<b>Wavelength range</b>	1.0 - 18 Å
<b>Scattering angle range</b>	1 - 170°
<b>Interplanar spacing range</b>	0.6 - 300 Å
<b>Resolution (<math>\Delta d/d</math>) :</b>	
θ = 80°, d = 2 Å	1 %
θ = 10°, d = 60 Å	10 %

**4.2. Detailed description of proposals for modernization of the instrument**

The main characteristics of the RTD diffractometer that affect the quality of the obtained data are the luminosity, the range of available interplanar spacings and the resolution.

To improve the informativeness of experiments, the planned upgrade of the diffractometer should be aimed at increasing its luminosity. For example, according to preliminary calculations, replacing the existing 12-meter curved section and the final 2-meter straight section of the neutron guide with new ones with a supermirror coating of  $m \approx 2$  will increase the neutron beam intensity approximately by a factor of 3 (Fig. 13).



**Fig. 13.** Calculated values of neutron beam intensity (total integral flux) on the existing (old) and new ( $m \approx 2$ ) neutron guides.

The next stage of modernization of the diffractometer involves minimizing the background level, upgrading the software, and developing the sample environment system to control external conditions on the sample during the experiment (temperature, electric/magnetic fields, gas atmosphere, vacuum). It is expected that this will not only allow more experiments to be conducted in the same amount of time, but also significantly expand the possibilities for studying the structural features of samples under controlled external conditions.

### 4.3. Current technical parameters of the instrument in thermal and cold modes of the moderator

The advantage of a cold moderator over a grooved thermal moderator is due to an increase (by a factor of  $\sim 5$ -10) in the flux of long-wave neutrons, which enhances the efficiency of studying structures with large unit cell parameters ( $> 300 \text{ \AA}^3$ ), the diffraction peaks of which are concentrated in the range of interplanar spacings  $d_{\text{hkl}} \approx 2.5 - 10.0 \text{ \AA}$ . A cold moderator ensures high-quality registration of peaks in the region of large  $d_{\text{hkl}}$  on the RTD, which corresponds to the optimal moderator temperature of  $\sim 100 \text{ K}$ . Considering the duration of measurements from 2 to 20 hours required for *in situ* experiments and for the analysis of magnetic and long-period structures (biomembranes, the family of MIL-101 metal-organic frameworks (MOFs)), an important condition is the stability of the cold moderator for at least 24 hours.

### 4.4. Expected technical parameters after modernization of the instrument

The replacement of the neutron guide, according to the calculations presented in Fig. 13, will increase the intensity of the neutron beam by a factor of  $\sim 3$  and proportionally shorten the data collection time, which is a prerequisite for recording processes occurring in condensed matter in real time. Increasing the solid angle  $\Omega_d$  of the backscattering detector from the current 0.8 sr to 2 sr by replacing it will enhance the luminosity by a factor of 2.5. The installation of detector collimators oriented strictly toward the sample will decrease the background level, which will lead to a reduction in the statistical error in the measured spectrum and an increase in the accuracy of determining structural parameters. A low background level will also allow for more accurate detection of the presence or absence of low-intensity diffraction peaks. The use of a cold moderator on the RTD diffractometer (beamline 6a) will improve the quality of neutron diffraction patterns in the range of interplanar spacings of more than  $3.6 \text{ \AA}$ . The installation of new goniometric devices, positioners, and sample environment equipment will make it possible to expand the experimental possibilities of the neutron diffractometer.

#### *Expected characteristics of the new neutron guide*

<b>Length</b>	14.0 m
<b>Width (constant)</b>	15 mm
<b>Entrance height</b>	200 mm
<b>Exit height</b>	100 mm
<b>Supermirror coating</b>	$m = 2$
<b>Reflectivity (at <math>\theta_c</math>)</b>	(96 - 98)%
<b>Radius of curvature</b>	1850 m
<b>Characteristic wavelength</b>	1.25 $\text{\AA}$
<b>Length of mirror section</b>	0.5 m
<b>Waviness of mirror section</b>	$\leq 1.5 \cdot 10^{-4}$
<b>Glass thickness</b>	$\sim 15 \text{ mm}$
<b>Vacuum level</b>	$\leq 0.1 \text{ MmHg}$

### 4.5. Relevance of the instrument development for the concept of a suite of spectrometers within the project of a new neutron source at JINR

The analysis of the possibilities of the RTD shows that there is a clear tendency towards its application for an increasingly wider range of scientific problems. Its key advantage lies in the combination of high intensity, which compensates for its medium resolution (compared to high-resolution analogs), and versatility, allowing the study of magnetic, long-period structures and even structural features of biological objects, such as lipid membranes. It is important that the instrument can be used for both static and dynamic (real-time) measurements. The latter particularly benefit from the unique characteristic of the IBR-2 source—its comparatively low

pulse frequency, which favorably distinguishes it from other spallation sources. Considering the above factors and the extensive and successful experience gained in operating the instrument, the inclusion of the RTD diffractometer into the development plan of the FLNP spectrometer complex is a logical and necessary step with good scientific prospects. Due to the dynamically developing possibilities of using neutron diffraction in the research of biologically significant materials, including protein crystals, there is growing demand for such measurements within the IBR-2 User Program (e.g., Laboratory for ageing and age-related neurodegenerative diseases, MIPT; Department of Medical Physics, KFU). Due to its high luminosity and wide range of interplanar spacings, the RTD is the most suitable instrument for such measurements. In this regard, optimization of the detector system in the small-angle scattering region, enabling the study of structures with characteristic parameters of the order of 100-300 Å, as well as equipping the sample environment system with goniometers capable of maintaining low temperatures, are of particular relevance and demand.

## 5. Expected scientific results, comparison with the world level

Equipping the RTD diffractometer with a full suite of sample environment systems (including cryogenic add-on modules, a furnace, an electrochemical cell, and a goniometer) will open up possibilities for conducting a *significantly wider range of studies*. At the same time, the implementation of all proposals on the modernization of the RTD (replacement of the neutron guide, increase in the solid angle of the detector, use of improved scintillators, installation of collimators, etc.) will directly *improve the characteristics of the diffractometer and speed up the data collection process*. Today, the RTD is a flexible instrument for conducting effective research using neutron diffraction on powders and single crystals in the field of condensed matter. The coverage of scattering angles from 1 to 170 degrees by the detector system and the real-time resolution of the diffractometer are comparable to those of the cold neutron diffractometer DMC (PSI), the multi-physics instrument MPI BL16 at the CSNS accelerator (Hong Kong, China), and the high-intensity two-axis diffractometer D20 in medium-resolution mode (ILL, France). The undeniable advantage of the RTD over the above-mentioned instruments is the possibility to collect complete information on all measured interplanar spacings of the material under study in very short periods of time, down to one neutron pulse, despite the fact that the RTD is inferior to them in terms of the magnitude of the neutron flux at the sample position, being approximately 5-20 times lower.

## 6. Requested resources, costs and time frames of instrument modernization

The main units to be upgraded are the mirror neutron guide, the backscattering detector, the detector system positioned at scattering angles of 30°-138°. The list of required sample environment equipment and approximate costs are given in **Table 4 (Section 4)**. The project will be realized by the staff of the RTD group of the Diffraction Sector with the assistance of employees from the SC Department.

**Plan-scheme of activities for the modernization of the RTD diffractometer**

№	Description	2026	2027	2028	2029	2030
1	Mirror neutron guide ( $m \approx 2$ )					
2	Backscattering detector system ( $\Omega_d \approx 2 \text{ sr}$ ), electronic components, slit collimators					
3	90° ZnS scintillation detector system and electronic components					
4	Rotating platform for small-angle scattering detector					
5	High-temperature furnace (1800°C)					
6	Electrochemical cell capable of passing high current (up to 10 A) through the sample across a temperature range of 20-900°C					
7	Huber three-circle goniometer with a refrigerator (down to 4 K)					
8	Mini-furnace for the Huber three-circle goniometer					
9	Refrigerator with shaft sample loading for a temperature range of 8 - 290 K					
10	Cryostat with possibility of generating a magnetic field on the sample					
11	Add-on module for generating a magnetic field from 0 to 2 T					
12	Modernization of electronics and software					
13	Radiation shielding screen with Pb glass "TISSA-RP" for beamlines 5-6					
14	Infrastructure					

**References**

1. A.M. Balagurov, A.I. Beskrovnyy, V.V. Zhuravley, G.M. Mironova, I.A. Bobrikov, D. Neov, S.G. Sheverev. Neutron Diffractometer for Real-Time Studies of Transient Processes at the IBR-2 Pulsed Reactor. *J. Surf. Invest. X-ray, Synchr. Neutr. Techniq.* 10(3) (2016) 467-479.
2. A.M. Balagurov, G.M. Mironova. "Real-time neutron diffraction studies," *Crystallography Reports*, v. 36, 1991 (in Russian).
3. N.Y. Ryabova, M.A. Kiselev, A. I. Beskrovnyi and A.M. Balagurov / Investigation of the structure of multilayer lipid membranes by real-time neutron diffraction // *Physics of the Solid State* 52 (5) (2010) P.1050-1058.
4. Balagurov A.M., Mironova G.M. Neutron diffraction study of the synthesis of  $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$  ceramics // *Superconductivity: Physics, Chemistry, Engineering*, 1990, Vol. 3, pp. 615-624 (in Russian).
5. A.M. Balagurov, G.M. Mironova, L.A. Rudnickij, and V.Yu. Galkin / Time-resolved neutron diffraction investigation of the effect of hydrogen on the high-T<sub>c</sub> superconductor  $\text{YBa}_2\text{Cu}_3\text{O}_{7-y}$  // *Physica C: Superconductivity* 172 (1990) 331-334.
6. Balagurov A.M., Bashkin I.O., Kolesnikov A.I., Malyshev V.Yu., Mironova G.M., Ponyatovsky E.G., Fedotov V.K. / Neutron diffraction studies of kinetics of phase transition in  $\text{TiD}_{0.74}$  // *Sov. Phys. Solid State* 33, 711 (1991).
7. A.M. Balagurov, O.I. Barkalov, A.I. Kolesnikov, G.M. Mironova, E.G. Ponyatovskii, V.V. Sinitsyn, and V.K. Fedotov, *Pis'ma Zh. Eksp. Teor. Fiz.* 53, (1991) 30.

Proposals for Development of a Suite of Instruments for Condensed Matter Research  
at the IBR-2 Reactor in 2026-2030

8. A.I. Beskrovny, S.G. Vasilovsky, S.B. Vakhrushev, D.A. Kurdyukov, O.I. Zvorykina, A.A. Naberezhnov, N.M. Okuneva, M. Tovar, E. Rysiakiewicz-Pasek, P. Jagus. Temperature dependences of the order parameter for sodium nitrite embedded in porous glasses and opals // *Physics of Solid State*. 52(5) (2010) 1021-1025.
9. Jaźdżewska M., K. Domin, M. Śliwińska-Bartkowiak, A.I. Beskrovnyi, D.M. Chudoba, T.V. Nagorna, K. Ludzik, D.S. Neov. "Structural properties of ice in confinement" *Journal of Molecular Liquids*, 283 (2019) 167-173.
10. Dmitriev A.I., Kaminsky V.M., Lashkarev G.V., Butorin P.E., Kovalyuk Z.D., Ivanov V.I., Beskrovny A.I. / Neutron diffraction studies of negative thermal expansion in an InSe layered crystal // *Physics of Solid State*. 51(11) (2009) 2207-2212.
11. I.A. Bobrikov, A.M. Balagurov, Hu Chih-Wei, Lee Chih-Hao, Chen Tsan-Yao, Deleg Sangaa, and D.A. Balagurov. Structural evolution in LiFePO<sub>4</sub>-based battery materials: In-situ and ex-situ time-of-flight neutron diffraction study // *J. Power Sources* 258 (2014) 356.
12. L.Mestres, M.L.Martinez-Sarrión, M.Morales, A.I.Beskrovnyi, I.Natkaniec, I.G.Shelkova, L.S.Smirnov, L.A.Shuvalov / X-ray and neutron diffraction study of the Rb<sub>2-x</sub>(NH<sub>4</sub>)<sub>x</sub>SO<sub>4</sub> system // *J. Phys. Soc. Jpn.* 67 (1998) P.1636-1642.
13. V.K.Fedotov, V.E.Antonov, A.I.Kolesnikov, A.I.Beskrovny, G.Grosse, F.E.Wagner / Neutron diffraction investigation of  $\gamma$ -manganese hydride // *Sol. St. Comm.*, 107(12), (1998) P.787-790.
14. A.I. Beskrovnyy and Z. Jirák. Structural modulation in Bi<sub>2</sub>Sr<sub>2</sub>Ca<sub>0.4</sub>Y<sub>0.6</sub>Cu<sub>2</sub>O<sub>8+d</sub>. *Journal of Physics: Conference Series*. V. 340, (2011) P. 405-410.
15. V.Yu. Pomjakushin, A. A. Zakharov, A. M. Balagurov, F. N. Gygax, A. Schenck, A. Amato, D. Herlach, A. I. Beskrovny, V. N. Duginov, Yu. V. Obukhov, A.N.Ponomarev, and S. N. Barilo / Microscopic phase separation in LaCuO<sub>4+y</sub> induced by the superconducting transition // *Phys. Rev. B*, 58(18) (1998) P.12350-12354.
16. L.M. Plyasova, V. F. Anufrienko, A.I. Beskrovnyi, I.Y. Molina et al. Influence reductive-oxidative processings on magnetic properties of chromite copper. *Journal of Structural Chemistry*, 43(2) (2002), p.274.
17. Z.Surowiec, M.Wiertel, A.I.Beskrovny, J.Sarzyński and J.J.Milczarek / Investigations of microscopic magnetic properties of the pseudo-binary system (Zr<sub>1-x</sub>Ti<sub>x</sub>)Fe<sub>2</sub> // *J.Phys.:Condens.Matter* v.15 (2003) P.6403-6414.
18. V. Likodimos, N. Guskos, S. Glenis, R. Szymczak, A. Bezkrovnyi, M. Wabia, J. Typek, G.Gasiorek, M. Kurzawa, I. Rychlowska-Himmel, and A. Blonska-Tabero / Magnetic properties of the antiferromagnetic site-disordered vanadate Zn<sub>2</sub>FeV<sub>3</sub>O<sub>11</sub> // *Eur.Phys.J. B* 38 (2004) P.13-18.
19. A.I. Beskrovnyi, Z.Jirak, M.Nevriva, and I.G. Shelkova / Neutron diffraction study of the modulated structure of Bi<sub>2</sub> Sr<sub>3-x</sub>Cu<sub>2</sub>O<sub>8+y</sub>(x~0.6) // *Physica C* 206 (1993) P.27-32.
20. Monika Jazdzewska, Małgorzata M. Śliwińska-Bartkowiak, Anatoly I. Beskrovnyy, Sergey G. Vasilovskiy, Siu-Wa Ting, Kwong-Yu Chan, Liangliang Huang and Keith E. Gubbins / Novel ice structures in carbon nanopores: pressure enhancement effect of confinement // *Phys. Chem. Chem. Phys.*, 13, (2011) P.9008-9013.
21. Monika Jazdzitska, Małgorzata Śliwińska-Bartkowiak, Kamila Domin, Dorota M Chudoba, Anatoly I Beskrovnyi, Dimitr S Neov, and Keith E Gubbins. Structure of ice confined in carbon and silica nanopores. *Bull. Mater. Sci.* (2019) 42:184 © Indian Academy of Sciences <https://doi.org/10.1007/s12034-019-1846-9>.

## DN-6 — Neutron Diffractometer for Ultrahigh-Pressure Research

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### 1. Abstract

Back in 1993, on the basis of the high-pressure neutron diffraction technique, the DN-12 diffractometer for studying micro-samples and allowing for research in the pressure range up to 7 GPa was developed and built in cooperation with the NRC “Kurchatov Institute” at the pulsed IBR-2 reactor at FLNP JINR. During the period of its operation, a wealth of experience has been gained both in the design, development and modernization of the instrument, as well as in the development of the high-pressure neutron diffraction technique. In 2013, a basic version of the new DN-6 diffractometer was built on channel 6B with significantly higher luminosity. Further modernization of this diffractometer made it possible to considerably expand the available range of high pressures up to 40 GPa. The planned modernization of the DN-6 is mainly aimed at improving its technical parameters and developing methods for producing high pressures with the aim of further expanding the range of pressures covered in experiments to 50 GPa.

### 2. Scientific program, relevance and comparison with the world level

Recently, there has been significant progress in the study of materials under extreme conditions. High pressure often gives rise to new physical phenomena in materials, including, in addition to pressure-induced superconductivity, various changes in magnetic states, insulator-metal transitions, spin crossover, structural and electronic phase transitions [1]. In addition, high-pressure studies offer unique opportunities to study microscopic mechanisms of the formation of physical phenomena in functional materials by analyzing the response of various properties to changes in structural parameters during compression of the crystal lattice. Also, under high pressures and temperatures, it is possible to synthesize new metastable forms of materials with unusual properties.

At present, neutron scattering experiments with high-pressure cells are carried out only in a few most advanced neutron centers in the world [1-8]. This is due to the fact that this kind of experiments can be done only at high-flux neutron sources on instruments equipped with advanced detector systems that provide good experimental statistics for appropriate data analysis. Until recently, the application of neutron methods, as a rule, has been restricted to the pressure range of 1-2 GPa [2], because of the use of relatively large samples in cells of the cylinder-piston type. The development of the method of neutron investigations at high pressures employing the technique of sapphire/diamond anvils in combination with low-background neutron diffraction has allowed us to extend the pressure range in experiments to several tens of GPa. Further development of the high-pressure technique, which is aimed at extending the accessible pressure range up to 30-50 GPa, requires an increase in the neutron flux at the sample position and the solid angle of the detector system [1].

The optimum combination of the intense neutron beam of beamline 6B, mirror neutron guide and a unique multidetector system of the DN-6 diffractometer makes it possible to perform experiments at a pressure of up to 13 GPa using new high-pressure sapphire-anvil cells. The use of high-pressure diamond-anvil cells allowed experiments with very small sample volumes (0.02 - 0.1 mm<sup>3</sup>) and pressures of up to 39 GPa, which makes it possible to rank the DN-6 diffractometer among the world's best instruments for structural neutron studies at high pressures. It should be noted that experimental neutron studies at sufficiently high pressures can be performed only on a few instruments in the world's advanced neutron scattering research centers, including the DISK diffractometer [2] at the IR-8 research reactor of NRC 'Kurchatov Institute' (Moscow, Russia); PERL diffractometer [3] at ISIS RAL (UK); G6.1 diffractometer at LLB (Saclay, France); high-pressure setup for the HRPT diffractometer [4, 5] at the SINQ neutron spallation source (Paul Scherrer Institute, Villigen, Switzerland); high-pressure setup at D20 in ILL (Grenoble, France); HiPPO diffractometer [6] at the Los Alamos Neutron Science Center (USA); PLANET diffractometer [7] at J-PARC (Ibaraki, Japan) and the SNAP diffractometer [8] at the SNS spallation neutron source (ORNL, Oak Ridge, USA).

### 3. Scientific and methodological groundwork laid in FLNP JINR

The work on the development and construction of the new DN-6 diffractometer [1] on beamline 6B of the IBR-2 reactor was a logical continuation of the work on the DN-12 diffractometer [2] and was aimed at significantly increasing the luminosity of the instrument and the range of pressures achievable in experiments. At present, the DN-6 diffractometer is mainly used for investigations of atomic and magnetic structures of condensed matter in experiments at ultrahigh pressures. Further development of the neutron diffraction technique for experiments with ultra-small sample volumes (down to thousandths of a cubic millimeter) was achieved by enhancing the neutron flux at the sample position and increasing the solid angle of the detector system. The intensity of the neutron beam of the DN-6 diffractometer on beamline 6B, equipped with a neutron guide section with parabolic vertical focusing, exceeds the intensity of the neutron beam of beamline 12 by a factor of ~20. The use of a cold neutron moderator at this beamline will significantly widen the possibilities for studying magnetic structures in crystals at high pressures. The optimum combination of the intense neutron beam at beamline 6B, use of a mirror neutron guide and the unique multi-detector system at DN-6 makes it possible to perform diffraction experiments with sample volumes of down to 0.0001 mm<sup>3</sup>.

At present, this unique experimental base makes it possible to conduct world-class experiments and obtain new cutting-edge scientific results [9-11]. Using the DN-6 diffractometer, a number of studies of the crystal and magnetic structure of materials were carried out in an extended pressure range of up to 39 GPa using high-pressure diamond-anvil cells.

The magnetic structure of the high-pressure orthorhombic phase of Fe<sub>3</sub>O<sub>4</sub> magnetite, formed at P > 25 GPa at room temperature, was determined. A study was conducted on the effect of high pressure in the pressure range up to 35 GPa and low temperatures in the range of 5-300 K on the magnetic structure of chromium oxide Cr<sub>2</sub>O<sub>3</sub> [11] (eskolaite). It was established that the main antiferromagnetic state of eskolaite is stable in the entire studied range of pressures and temperatures, and the existence of a magnetic phase transition, previously suggested on the basis of experiments on the generation of the second optical harmonic, was not confirmed.

The advantage of the DN-6 diffractometer is that it can be effectively used to study the effects of high pressure on a wide range of materials with medium neutron scattering lengths or magnetic moments in the ultra-high pressure range. For example, the magnitude of the ordered magnetic moment of Cr<sup>3+</sup> ions in Cr<sub>2</sub>O<sub>3</sub> [11] at low temperature is about 3 μB, which is also typical for many other transition metal oxides.

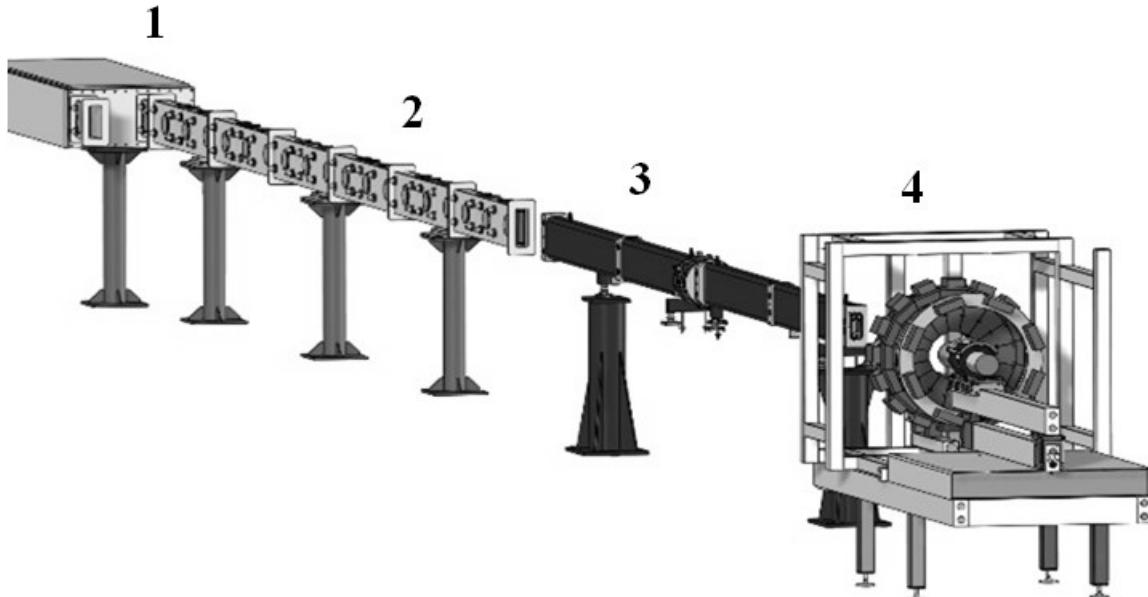
#### 4. Current status of the instrument and proposals for its modernization

##### 4.1. Current status and main units of DN-6

The main components of the new DN-6 spectrometer are shown in Fig. 14.

##### Main characteristic parameters of DN-6 diffractometer

<b>Thermal neutron flux at sample position</b>	$\sim 5 \times 10^7$ n/cm <sup>2</sup> /s
<b>Experimental <math>d_{hkl}</math>-range</b>	
at scattering angle $2\theta = 90^\circ$ :	5.7 Å
at scattering angle $2\theta = 42^\circ$ :	11.2 Å
<b>Resolution</b>	
$\Delta d/d$ at $d = 2$ Å:	
at scattering angle $2\theta = 90^\circ$ :	0.025
at scattering angle $2\theta = 42^\circ$ :	0.030
<b>Characteristic measurement time for one diffraction pattern:</b>	
typical sample volume $V \sim 50$ mm <sup>3</sup>	0.1 h
small sample volume $V \sim 1$ mm <sup>3</sup>	2-4 h
ultra-small sample volume $V \sim 0.01$ mm <sup>3</sup>	20-40 h
<b>Temperature range</b>	5-320 K



**Fig. 14.** General layout of the DN-6 diffractometer at beamline 6B of the IBR-2 reactor. 1 – neutron beam splitter; 2 – curved section of the neutron guide in vacuum housing; 3 – parabolic focusing section of the neutron guide in vacuum housing; 4 – detector system and sample environment system.

The optimum combination of the intense neutron beam of beamline 6B, the use of a mirror neutron guide, and the unique multi-detector system of the DN-6 diffractometer allows for diffraction experiments to be carried out with samples with volumes of down to 0.01 mm<sup>3</sup>.

The neutron beam is formed using a three-section neutron guide. The first section is a neutron-optical splitter that divides the incident neutron beam into two beams for the DN-6 diffractometer and the adjacent RTD diffractometer on beamline 6a, respectively. It is followed by a 20-m curved section (coating  $m = 3$ ) manufactured by SwissNeutronics (Switzerland) with a total radius of curvature of 1860 m. The neutron beam after this section measures  $165 \times 15 \text{ mm}^2$ . A parabolic system with the vertical focusing and coating  $m = 3$  was manufactured by SwissNeutronics (Switzerland) and installed as the third end section of the neutron guide. The focused beam, measuring  $10 \times 10 \text{ mm}$ , is located at a distance of 870 mm from the end section of the neutron guide. The new focusing section of the neutron guide increases the total neutron flux at the sample position by a factor of  $\sim 6$ . The total length of the entire neutron guide from the surface of the neutron moderator to the sample position is 30.5 m.

A helium refrigerator ( $T \geq 4 \text{ K}$ ) and high-pressure cells of different designs (including those with diamond anvils) are used to modify the sample environment. To extend the accessible pressure range, new pressure cells with sapphire and diamond anvils are being designed and manufactured.

A special frame provides fastening of the collimation system, detector system in a protective casing and helium refrigerator on one platform (Fig. 9). Low sample temperatures are achieved using a specially designed cryostat based on a closed-cycle helium refrigerator providing minimum temperatures of  $T \sim 5 \text{ K}$ .

A special frame provides attachment of the collimation system, the detector system in a protective casing and the helium refrigerator on one platform. Low sample temperatures are achieved using a specially designed cryostat based on a closed-cycle helium refrigerator, providing minimum temperatures of  $T \sim 5 \text{ K}$ .

The DN-6 diffractometer operates in the time-of-flight mode. It is equipped with two ring detectors for different scattering angles (Fig. 14). A photograph of one of the ring detectors is shown in Fig. 15.



**Fig. 15.** Detector section of the DN-6 diffractometer. The detectors are housed in boron-polyethylene casings for suppressing background. The system consists of sixteen sectoral collimation modules and detector units. Each ring detector consists of 96 individual  ${}^3\text{He}$ -counters arranged in six rings of 16 detectors each. The detector system provides neutron detection in the range of scattering angles of  $87\text{--}93^\circ$  and  $40\text{--}44^\circ$ . Neutron diffraction patterns are obtained by summing the spectra from each detector element with an appropriate correction for the scattering angle.

#### 4.2. Detailed description of proposals for modernization of the instrument

1. For experiments at high pressures, it is necessary to purchase diamond anvils, as well as a set of specialized equipment for working with high-pressure sapphire-anvil cells.

2. To expand the range of pressures at the sample position, it is necessary to develop new, more advanced types of diamond-anvil cells.
3. To vacuum sections of the neutron guide of beamline 6B, it is necessary to purchase vacuum pumping stations based on spiral or forevacuum pumps and special accessories for their operation.
4. To accurately position the sample relative to the detector system, it is necessary to purchase an endoscope and a video monitoring system.
5. To improve background conditions on the sample, it is necessary to purchase cadmium foils and boron-polyethylene plates.

#### ***4.3. Current technical parameters of the instrument in thermal and cold modes of the moderator***

The new DN-6 diffractometer is located on beamline 6B, where the option of operation of the moderator in the cold mode is provided, which makes it possible to increase the intensity of the incident cold neutron beam in the wavelength range of 4-13 Å by a factor of ~7-10, and this, in turn, significantly improves the quality of measured diffraction data in the range of interplanar spacings  $d_{hkl} > 4 \text{ \AA}$ .

#### ***4.4. Expected technical parameters after modernization of the instrument***

On the DN-6 diffractometer on beamline 6B, in the cold moderator mode, the intensity of the cold neutron flux in the wavelength range of 4-13 Å increases by a factor of ~7-10, and this, in turn, significantly improves the quality of measured diffraction data in the range of interplanar spacings  $d_{hkl} > 4 \text{ \AA}$ .

#### ***4.5. Relevance of the instrument development for the concept of a suite of spectrometers within the project of a new neutron source at JINR***

The methodological and scientific development of neutron diffraction studies at high pressures is one of the promising and relevant areas in the FLNP Scientific Program, including for implementation at the new neutron source.

#### ***5. Expected scientific results, comparison with the world level***

On the DN-6 diffractometer, a high-pressure sapphire-anvil cell technique is used for research in the pressure range of up to 13 GPa, which was also implemented in studies on DISK (NRC "Kurchatov Institute"), DN-12 (FLNP JINR, Dubna) and G6.1 (LLB, Saclay, France) diffractometers. The use of high-pressure diamond-anvil cells made it possible to carry out experiments with ultra-small sample volumes of 0.02-0.1 mm<sup>3</sup> at pressures up to 39 GPa, which allows the DN-6 diffractometer to be classified as one of the best instruments in the world for structural neutron studies at high pressures. It should be noted that the joint efforts of a collaboration of researchers from Universite P.&M.Curie (Paris, France) and the University of Edinburgh (Edinburgh, UK) led to the development of a technique of high-pressure cells with anvils made of tungsten carbide and sintered diamond with a toroidal profile. These cells are widely used in experiments on research instruments of foreign pulsed and stationary neutron sources, including the PEARL diffractometer, ISIS RAL (UK); PLANET diffractometer, J-PARC (Japan); SNAP diffractometer, SNS (ORNL, USA); HRPT diffractometer, SINQ (PSI, Switzerland); D20 diffractometer, HFR (ILL France); HiPPO diffractometer, LANSCE (LANL, USA). However, it should be noted that to date, most neutron diffraction experiments at ultra-high pressures above 20 GPa in other neutron centers have been carried out only for a limited number of model systems with large neutron scattering lengths or ordered magnetic moment values. The

advantage of the DN-6 diffractometer is that it can be effectively used to study the effects of high pressure on a wide range of materials with medium neutron scattering lengths or magnetic moments in the ultra-high pressure range. It is expected that further development of the DN-6 diffractometer will make it possible to conduct diffraction experiments with record-low volumes of studied samples in an extended range of ultra-high pressures up to 50 GPa in the low-temperature region of 5-300 K. Among the expected results of future research are pressure-induced structural and magnetic phase transitions in ferroelectrics, multiferroics, low-dimensional magnets, including van der Waals materials, etc.

## 6. Requested resources, costs and time frames of instrument modernization

The costs of the new detector ring and equipment for performing experiments with high pressures are given in **Table 5 (Section 4)**.

### Plan-scheme of activities for the modernization of the DN-6 diffractometer

№	Description of activities	2026	2027	2028	2029	2030
1.	Set of high-pressure diamond-anvil cells					
2.	Equipment for preparing high-pressure diamond-anvil cells for the experiment: a gasket drilling machine, a gas charge for cells, sets of diamond anvils, gaskets, and carbide supports					
3.	Development of drawings for a new type of diamond-anvil cells					
4.	Purchase of vacuum pumping stations based on spiral or forevacuum pumps and special accessories for their operation					
5.	Sample-positioning system based on an endoscope and a video monitoring system					
6.	Purchase of materials for background shielding and structural components					

### References

1. D.P. Kozlenko, S.E. Kichanov, E.V. Lukin, B.N. Savenko “The DN-6 Neutron Diffractometer for High-Pressure Research at Half a Megabar Scale”, *Crystals*, 8, 331 (2018)
2. Glazkov, V.P.; Naumov, I.V.; Somenkov, V.A.; Shilshtein, S.Sh. Superpositional many-detector system and neutron diffraction of microsamples, *Nucl. Instrum. Methods Phys. Res.* **1988**, 264, 367-374, DOI: 10.1016/0168-9002(88)90925-4.
3. Bull, C.L.; Funnell, N.P.; Tucker, M.G.; Hull, S.; Francis, D.J.; Marshall, W.G. PEARL: the high pressure neutron powder diffractometer at ISIS, *High Press. Res.* **2016**, 36, 493-511, DOI: 10.1080/08957959.2016.1214730.
4. Klotz, S.; Strässle Th.; Rousse G.; Hamel G.; Pomjakushin, V. Angle-dispersive neutron diffraction under high pressure to 10 GPa, *Appl. Phys. Lett.* **2005**, 86, 031917, DOI: 10.1063/1.1855419.
5. Klotz, S.; Strässle, Th.; Lebert, B.; d'Astuto, M.; Hansen, Th. High pressure neutron diffraction to beyond 20 GPa and below 1.8 K using Paris-Edinburgh load frames, *High Press. Res.* **2016**, 36, 73-78, DOI: 10.1080/08957959.2015.1136624.
6. Zhao, Y.; Zhang, J.; Xu, H.; Lokshin, K.A.; He, D.; Qian, J.; Pantea, J.; Daemen, L.L.; Vogel, S.C.; Ding, Y.; Xu, J. High-pressure neutron diffraction studies at LANSCE, *Appl. Phys. A* **2010**, 99, 585-599, DOI: 10.1007/s00339-010-5640-1.

7. Arima, H.; Hattori, T.; Komatsu, K.; Abe, J.; Utsumi, W.; Kagi, H.; Suzuki, A.; Suzuya, K.; Kamiyama, T.; Arai, M.; Yagi, T. Designing PLANET: Neutron beamline for high-pressure material science at J-PARC, *J. Phys.: Conf. Ser.* **2010**, 215, 012025, DOI:10.1088/1742-6596/215/1/012025.
8. Iizuka, R.; Yagi, T.; Gotou, H.; Komatsu, K.; Kagi, H.; An opposed-anvil-type apparatus with an optical window and a wide-angle aperture for neutron diffraction, *High Press. Res.* **2012**, 32, 3, 430-441, DOI: 10.1080/08957959.2012.722213.
9. Golosova, N.O.; Kozlenko, D.P.; Dubrovinsky, L.S.; Cerantola, V.; Bykov, M.; Bykova, E.; Kichanov, S.E.; Lukin, E.V.; Savenko, B.N.; Ponomareva, A.V.; Abrikosov, A.I. Magnetic and structural properties of FeCO<sub>3</sub> at high pressures, *Phys. Rev. B* **2017**, 96, 13, 134405, DOI:10.1103/PhysRevB.96.134405.
10. Belozerova, N.M.; Kichanov, S.E.; Jirak, Z.; Kozlenko, D.P.; Kacenka, M.; Kaman, O.; Lukin, E.V.; Savenko, B.N. High pressure effects on the crystal and magnetic structure of nanostructured manganites La<sub>0.63</sub>Sr<sub>0.37</sub>MnO<sub>3</sub> and La<sub>0.72</sub>Sr<sub>0.28</sub>MnO<sub>3</sub>, *J. Alloys Compd.* **2015**, 646, 998-1003, DOI: 10.1016/j.jallcom.2015.06.154.
11. Golosova, N.O.; Kozlenko, D.P.; Kichanov, S.E.; Lukin, E.V.; Liermann, H.-P.; Glazyrin, K.V.; Savenko, B.N. Structural and magnetic properties of Cr<sub>2</sub>O<sub>3</sub> at high pressure, *J. Alloys Compd.* **2017**, 722, 1, 593-598, DOI: 10.1016/j.jallcom.2017.06.140.

## DN-12 — Neutron Diffractometer for Investigations of Microsamples at High Pressures

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### 1. Abstract

At the IBR-2 reactor, a specialized diffractometer DN-12 for investigations of microsamples was developed and constructed in collaboration with researchers from the NRC “Kurchatov Institute” for conducting neutron diffraction studies in the high-pressure range up to 8 GPa. It is planned to carry out work on modernization of the DN-12, aimed at improving its technical parameters and expanding the available pressure range.

### 2. Scientific program, relevance and comparison with the world level

Neutron diffraction is the most direct and informative method for studying the crystal structure and magnetic ordering in materials. Because of the low intensities of neutron sources, which are many orders of magnitude lower than those of synchrotron sources, performing neutron studies in a fairly wide pressure range (up to 5-7 GPa and higher) is limited by the need to use rather large sample volumes (at least several mm<sup>3</sup>) and became possible only relatively recently. The corresponding neutron experimental technique has been developed only at a few most advanced neutron research centers in the world, including the Frank Laboratory of Neutron Physics at JINR (Dubna).

The advanced methodological base of the DN-12 diffractometer [1] provides possibilities for performing a comprehensive analysis of both the crystal and magnetic structures (from the same set of experimental data) of fairly complex materials using high-pressure sapphire-anvil cells. At present, neutron scattering experiments using high-pressure cells are carried out only in a few of the most advanced research laboratories in the world. This is due to the fact that this kind of experiments can be done only at high-flux neutron sources on instruments equipped with advanced detector systems that provide good experimental statistics for data analysis. The development of the method of neutron investigations at high pressures employing the technique of sapphire/diamond anvils in combination with low-background neutron diffraction has made it possible to expand the pressure range in these experiments to several tens of GPa. Further development of the high-pressure technique, which is aimed at expanding the range of achievable pressures up to 12-15 GPa, requires an increase in the neutron flux at the sample position and an increase in the solid angle of the detector system.

The optimum combination of the intense neutron beam of beamline 12 at the IBR-2 high-flux pulsed reactor, the use of a supermirror neutron guide, and the unique multidetector system of the DN-12 diffractometer makes it possible to perform experiments with ultra-small sample volumes of down to 1 mm<sup>3</sup> at pressures of up to 8 GPa.

At present, the DN-12 diffractometer is mainly used to study the atomic and magnetic structures of condensed matter at high pressures (0 - 8 GPa) and low temperatures (10 - 300 K). It should be noted that the DN-12 diffractometer provides a slightly better resolution in interplanar

spacings compared to the DN-6, therefore, in the case of studying functional materials, it allows one to obtain more precise structural information and, in this regard, is complementary to the DN-6. Neutron diffraction experiments on the DN-12 spectrometer allow one to simultaneously determine the characteristics of the crystal and magnetic structure of functional materials over a wide range of temperatures and high external pressures. Over a fairly long period of experimental studies on the DN-12 spectrometer and DN-6 diffractometer, a new scientific direction has been formed, consisting in a systematic study of the relationship between changes in the crystal and magnetic structures and the physical properties of entire classes of materials [2-11]. This experimental approach makes it possible to identify microscopic mechanisms underlying the observed physical phenomena and effectively separate the contributions of competing interactions due to their different dependence on pressure. Below is a review of several recent studies on pressure-induced changes in the crystal and magnetic structures of various functional materials.

### 3. Scientific and methodological groundwork laid in FLNP JINR

Over the long period of operation of the DN-12 diffractometer, a number of successful studies have been performed, which, in fact, laid the foundation for a new scientific direction consisting in the systematic simultaneous investigation of the crystal and magnetic structures of entire classes of functional materials widely used in various technologies. The structural and magnetic phase diagrams of bulk and nanostructured perovskite-like manganites with the colossal magnetoresistance effect  $R_{1-x}A_xMnO_3$  ( $R = La, Pr$ ,  $A = Ca, Sr$ ) were studied in a wide range of concentrations  $x$ , and a number of new pressure-induced magnetic phase transitions were discovered [2-5], in particular, transitions from the FM state or pseudo-CE AFM state to the A-type AFM state in compositions with a concentration  $x \sim 0.25-0.3$ . In recent studies, structural aspects of the formation of magnetic states in nanostructured manganites  $La_{0.63}Sr_{0.37}MnO_3$  and  $La_{0.72}Sr_{0.28}MnO_3$  under high pressures were investigated [5]. While the bulk analogs of these compounds are ferromagnets, in their nanostructured form, an antiferromagnetic phase is observed to appear already at atmospheric pressure, and with increasing pressure, an increase in the proportion of this magnetic phase is detected with a simultaneous suppression of the initial ferromagnetic phase.

A series of studies were carried out to investigate the pressure-induced effects of spin crossover on the magnetic states and properties of complex cobalt oxides of various compositions [6, 7]. Cobalt oxides have a unique feature – the ability to change the spin state of  $Co^{3+}$  ions when temperature or pressure varies. Depending on the balance of comparable values of exchange energy  $J_H$  and crystal field splitting energy  $\Delta_{CF}$ , the diamagnetic low-spin LS ( $t_{2g}^6, S = 0$ ), and magnetic intermediate-spin IS ( $t_{2g}^5e_g^1, S = 1$ ) or high-spin HS ( $t_{2g}^4e_g^2, S = 2$ ) states can be realized. In particular, in the layered compound  $LaSrCoO_4$  with a tetragonal crystal structure of  $I4/mmm$  symmetry, in the pressure range up to 5.8 GPa, an anomalous behavior of thermal volume expansion was revealed [7], which indicates a pressure-induced spin crossover in  $Co^{3+}$  ions.

A study was conducted to investigate the effect of high pressure on the structural and magnetic properties of  $PbFe_{0.5}Nb_{0.5}O_3$ ,  $RMn_2O_5$  ( $R = Y, Bi$ ) multiferroic materials. The  $PbFe_{0.5}Nb_{0.5}O_3$  compound is an unusual example of a relaxor multiferroic with pronounced magnetoelectric coupling. Under normal conditions,  $PbFe_{0.5}Nb_{0.5}O_3$  [2] has a rhombohedral structure of  $R3m$  symmetry, and with a decrease in temperature in this compound below  $T_N = 155$  K, a G-type antiferromagnetic order is formed, which remains stable at high pressures up to 6.4 GPa.

A systematic study of the effect of high pressure on the crystal and magnetic structures of rare-earth and cobalt (R-Co) intermetallic compounds was conducted [8, 9]. Above the Curie temperature, the  $RCo_2$  ( $R = Tb-Er$ ) compounds crystallize in the cubic Laves phase of  $Fdm$  symmetry. The occurrence of ferrimagnetic ordering of rare-earth and cobalt sublattices leads to a reduction in the symmetry of the crystal structure to tetragonal  $I4_1/amd$  ( $R = Dy, Ho$ ) or rhombohedral  $m$  ( $R = Tb, Er$ ). Systematic neutron diffraction studies of the atomic and magnetic

structure of  $RCO_2$  compounds under temperature variations in the range of 10-300 K and at pressures in the range of 0-5 GPa have shown that the traditional concept of itinerant-electron metamagnetism does not allow an adequate description of the behavior of the magnetic properties of the entire class of these materials.

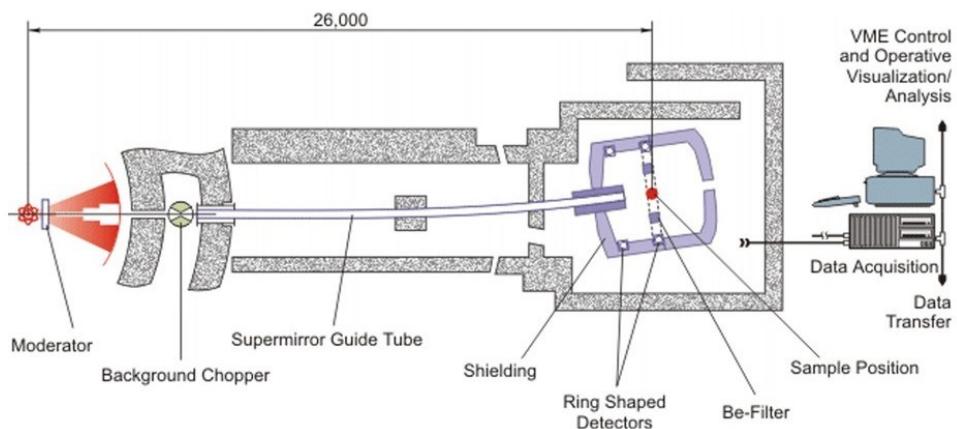
#### 4. Current status of the instrument and proposals for its modernization

##### 4.1. Information on the current status of the instrument

The schematic diagram of the DN-12 time-of-flight diffractometer on beamline 12 of the IBR-2 pulsed reactor is shown in Fig. 16. It consists of the following main components: a drum-type neutron beam chopper for improving background conditions at the sample position; a curved supermirror neutron guide in an evacuated casing with a length of 26 m; a beam collimation system; a ring detector system in a protective housing; an electronic system for control, acquisition and processing of neutron data.

The detector system of the DN-12 (Fig. 17) consists of two rings with radii of 393 and 342 mm, located at a distance of 386 mm from each other. The first ring detector contains 96 counters in a single helium-filled volume [10]. It is used to detect scattered neutrons in the  $2\theta$  angle range from 88 to 92°. The second ring detector consists of 48 individual neutron counters, independently detecting neutrons and arranged in three rings of 16 counters each. Both detector rings are placed in boron-polyethylene housings to improve background conditions at the sample position. The protective housing with two detector rings can be moved along the neutron beam axis, providing a neutron scattering angle ( $2\theta$ ) range from 45.5 to 90°. The MPD-32 electronic module collects data from the detector system. The SONIX+ software package implements procedures and algorithms for automating experiments and visualizing experimental data.

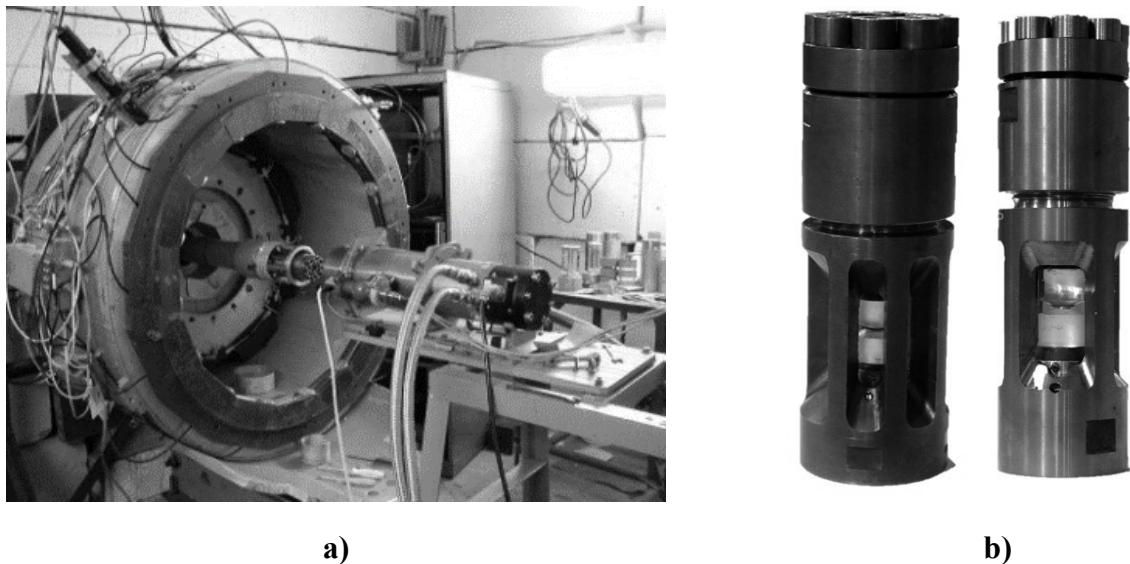
A closed-cycle helium refrigerator is used to provide low temperatures at the sample position, which makes it possible to conduct neutron experiments in the temperature range from 10 to 300 K. At present, the DN-12 spectrometer is equipped with a system of cooled superconducting magnets. This system will allow experiments to be carried out in magnetic fields up to 4 T and low temperatures down to 10 K using high-pressure sapphire-anvil cells. The equipment of the system of cooled superconducting magnets is currently being tested.



**Fig. 16.** Schematic representation of the main components of the DN-12 diffractometer.

### *Main parameters of the DN-12 diffractometer*

The DN-12 diffractometer is equipped with high-pressure cells of various designs. High-pressure sapphire-anvil cells are used for research in the pressure range up to 7 GPa (DN-12) or 10-12 GPa (DN-6) over a wide temperature range. Their design is characterized by wide-aperture side windows for unobstructed passage of scattered neutrons. The location of these windows matches the geometry of the detector system of both diffractometers. Anvils for high-pressure cells are made of leucosapphire single crystals, the upper end surfaces of which are polished to optical clarity. To achieve a quasi-hydrostatic pressure distribution, a hemispherical hole is drilled in the center of the anvil working area. The non-uniformity of pressure distribution across the surface of the sample in anvils with such holes usually does not exceed 10-15%. A typical sample volume in a high-pressure sapphire-anvil cell is approximately  $2 \text{ mm}^3$ . Reducing the anvil working area and the corresponding sample volume to  $0.5 \text{ mm}^3$  makes it possible to achieve a maximum pressure of up to 11-12 GPa.



**Fig. 17.** a) Photo of the DN-12 diffractometer detector system with a high-pressure cell and a horizontal cryostat based on a closed-cycle helium refrigerator, placed for the neutron experiment. b) High-pressure cells used for experiments on the DN-12 neutron diffractometer: a high-pressure sapphire-anvil cell made of beryllium bronze alloy; a compact high-pressure sapphire-anvil cell made of high-strength steel.

#### **4.2. Detailed description of proposals for modernization of the instrument**

1. The detector system is planned to be upgraded to increase its solid angle. This will enhance the luminosity of the diffractometer severalfold. To modernize the detector system, it is required to purchase neutron counters, preamplifier boards and electronic modules.
2. Conducting experiments at high pressures requires the purchase of sapphire anvils and the development of drawings for a new type of sapphire-anvil cells.
3. It is necessary to purchase a set of special equipment for the operation of high-pressure cells with sapphire anvils.
4. To expand the range of tasks solved on the DN-12 spectrometer, it is planned to use a cryogenic system for cooling a permanent magnet (up to 5 T) and simultaneous cooling of the high-pressure cell—a cryocooler system. This device will enable diffraction experiments to be conducted at high pressure in a magnetic field. Therefore, one of the main tasks in the near future will be commissioning and conducting test experiments with the magnetic cryocooler. In addition, it is required to design, develop and manufacture a mounting fixture for the cryocooler on the DN-12 diffractometer. Work is currently being completed on the design and manufacture of a permanent superconducting magnet, which will expand the experimental possibilities of the diffractometer.

#### **4.3. Current technical parameters of the instrument in thermal and cold modes of the moderator**

The DN-12 is located on beamline 12 of IBR-2, where the installation of the cold moderator is not planned.

#### **4.4. Expected technical parameters after modernization of the instrument**

It should be noted that besides the DN-12 diffractometer, neutron studies under high pressure can be performed in the NRC ‘Kurchatov Institute’ (DISK diffractometer at the IR-8 reactor), ILL (France, D20 diffractometer), ISIS RAL (UK, PEARL diffractometer), LLB (France, G6.1 diffractometer), ORNL (USA, SNAP diffractometer). However, in most cases, there are a number of limitations that do not allow conducting experiments at a qualitatively comparable level. For

example, the DISK and G6.1 diffractometers do not have a high enough resolution to analyze fine features of the atomic structure of structurally complex materials. The PEARL diffractometer has a limited low-temperature range (down to 80 K) and a limited range of covered interplanar spacings, which is insufficient for analyzing the magnetic structure. The closest in experimental capabilities is the SNAP diffractometer (ORNL, USA). However, the limited range of covered interplanar spacings at this diffractometer is also insufficient for a comprehensive analysis of the magnetic structure of materials. Therefore, in other neutron centers, the diffraction experiments with high pressures can only be performed to analyze separately either the crystal structure of relatively simple materials or the magnetic structure. After upgrading the detector system, it is expected that the collection of scattered neutrons will double, as well as the possibility of conducting neutron diffraction experiments to study the P-T-H phase and magnetic diagrams of materials.

#### ***4.5. Relevance of the instrument development for the concept of a suite of spectrometers within the project of a new neutron source at JINR***

The methodological and scientific development of neutron diffraction studies at high pressures is one of the promising and relevant areas in the FLNP Scientific Program, including for implementation at the new neutron source.

#### ***5. Expected scientific results, comparison with the world level***

The DN-12 diffractometer provides possibilities for routine investigations of wide classes of materials in a wide range of high pressures (at present, in experiments it is possible to achieve pressures up to 7-8 GPa) and at low temperatures. After upgrading the detector system, the luminosity is expected to double, the available pressure range will expand to 12 GPa, and it will become possible to conduct neutron diffraction experiments to study the P-T-H phase and magnetic diagrams of various materials, including complex magnetic oxides, multiferroics, low-dimensional magnets, and ferroelectrics. It should be noted that the DN-12 has a slightly better resolution ( $\Delta d/d$ ) compared to the DN-6 diffractometer, which allows it to be used to obtain more precise information for materials with a relatively complex crystal structure in the range of thermodynamic parameters achievable at this instrument.

The appropriate configuration of the neutron guide system and multi-component detector system in combination with a set of high-pressure sapphire-anvil cells makes the parameters of the DN-12 diffractometer comparable or superior to that of the most advanced dedicated instruments at other leading neutron scattering research centers mentioned above. The possibility to conduct measurements simultaneously at low temperatures and magnetic fields under relatively high pressures (above 2 GPa) is unique and currently unavailable at other neutron centers.

#### ***6. Requested resources, costs and time frames of instrument modernization***

The costs of the new ring detector and equipment for performing experiments under high pressures are given in **Table 6 (Section 4)**.

**Plan-scheme of activities for the modernization of the DN-12 diffractometer**

Description of activities	2026	2027	2028	2029	2030
Commissioning of a cryogenic system for cooling a permanent magnet (up to 5 T) and simultaneous cooling of a high-pressure cell - cryocooler system					
Purchase of cryocooler system accessories: mechanical fasteners, system positioning device, radiation shielding, vacuum equipment and accessories, vacuum sensors					
Upgrade of the detector system to increase the solid angle and install a cryocooler system. Purchase of neutron counters and data acquisition electronics.					
Purchase of high-pressure cells and accessories (sapphire anvils, carbide supports, gaskets, tools)					
Boron-polyethylene casing for the upgraded detector ring					
Long working distance monocular microscope for adjusting anvils in high-pressure cells and its accessories					

**References**

1. Kozlenko D.P., Kichanov S.E., Lukin E.V., Savenko B.N., Crystallography Reports, 2021, Vol. 66, No. 2, pp. 303–313.
2. Kozlenko D.P., Chan T.A., Kichanov S.E., et al. // JETP Letters. 2010. V. 92. P. 590.
3. Vu M.T., Kozlenko D.P., Kichanov S.E., et al. // J. Alloys Compd. 2016. V. 681. P. 527.
4. Kichanov S.E., Kozlenko D.P., Khiem L.H., et al. // Chemical Physics. 2020. V. 528. P. 110541.
5. Belozerova N.M., Kichanov S.E., Jirák Z., et al. // J. Alloys Compd. 2015. V. 646. P. 998.
6. Golosova N.O., Kozlenko D.P., Nicheva D., et al. // Journal of Magnetism and Magnetic Materials. 2020. P. 166874.
7. Lis O.N., Kichanov S.E., Kozlenko D.P., et al. // Journal of Magnetism and Magnetic Materials. 2019. V. 487. P. 165360.
8. Burzo E., Vlaic P., Kozlenko D.P., et al. // J. Alloys Compd. 2017. V. 724. P. 1184.
9. Kozlenko D.P., Burzo E., Vlaic P., et al. // Scientific reports. 2015. V. 5. P. 8620.
10. Belushkin A.V., Manoshin S.A., Kozlenko D.P., Kichanov S.E. // Nucl. Instrum. Methods Phys. Res. A. 2018. V. 892. P. 48.

# EPSILON — Neutron Diffractometer for measuring crystallographic texture and internal strains

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**Collaborating organizations:** NRC "Kurchatov Institute", Moscow, Russia  
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## 1. Abstract

The EPSILON diffractometer is positioned on beamline 7A1. The long neutron flight path from the pulsed source (~107 m) allows for diffraction experiments to be carried out with very high resolution. A decision was made to upgrade this diffractometer to solve various scientific problems requiring the measurement of crystallographic texture and analysis of internal strains/stresses. The main advantage of the EPSILON diffractometer is the simultaneous detection of a large number of crystallographic lattice planes in combination with high resolution. This opens up possibilities for studying multiphase materials containing phases with low crystallographic symmetries, which is typical for a number of materials: ceramics, industrial materials, archaeological materials, and rocks.

## 2. Scientific program, relevance and comparison with the world level

Many materials, including metals, alloys, ceramics, composites, and rocks, consist of grains of different sizes, shapes, and orientations. Crystallographic texture is the preferred orientation of crystallites within a given phase volume in a polycrystalline material relative to a chosen coordinate system [1]. Conventional processing of materials at room or high temperatures, such as casting, extrusion, rolling or forging, inevitably leads to a reorientation of crystal grains and, thereby, towards achieving a preferred orientation. Due to the anisotropy of the properties of individual crystallites, the texture affects mechanical and physical properties such as, for example, strength, plasticity or electrical conductivity. Therefore, knowledge of texture and its evolution is often used to optimize material properties in applied industrial processing technologies or to obtain information about geological processes in Earth sciences. Recently, neutron texture analysis methods have been used to study the manufacturing features of archaeological artifacts. In addition to neutron texture analysis, the EPSILON diffractometer [2] is useful for quantitatively assessing the resulting distribution of residual stresses. For example, welding processes cause profound changes in the microstructure, locally affecting the composition and mechanical properties of the material. The analysis of residual stresses [3] using neutron diffraction takes into account the influence of local changes in composition by measuring unstressed samples taken from areas away from the weld pool and heat-affected zones.

The project proposes the modernization of the EPSILON neutron diffractometer to expand its possibilities for conducting experiments to measure texture and internal strains/stresses in various materials.

## 3. Scientific and methodological groundwork laid in FLNP JINR

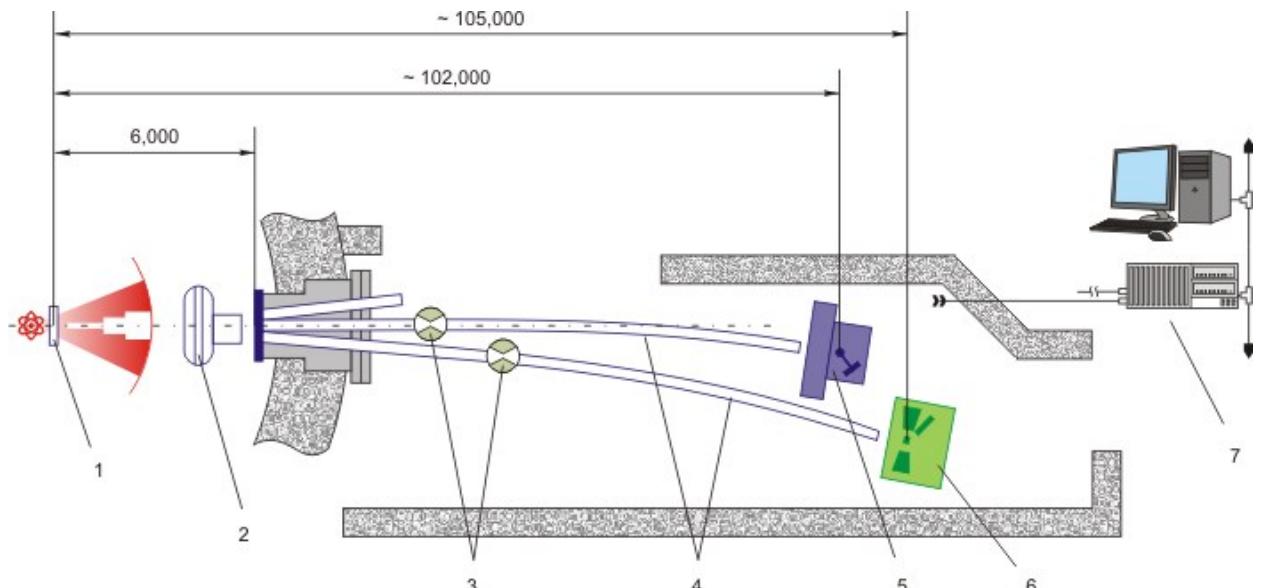
In FLNP, vast experience has been gained both in operating time-of-flight neutron texture diffractometers [4] and in processing the obtained data. The experience accumulated at FLNP in developing diffractometers makes it possible to implement the proposed development variant for

EPSILON. The scientific team of the Laboratory has significant experience in methodological developments in the field of neutron diffraction [5, 6] and neutron tomography [7, 8].

#### 4. Current status of the instrument and proposals for its modernization

##### 4.1. Information on the current status of the instrument

The main units of the EPSILON diffractometer are shown in Fig. 18. Its main parameters are presented in the table below.

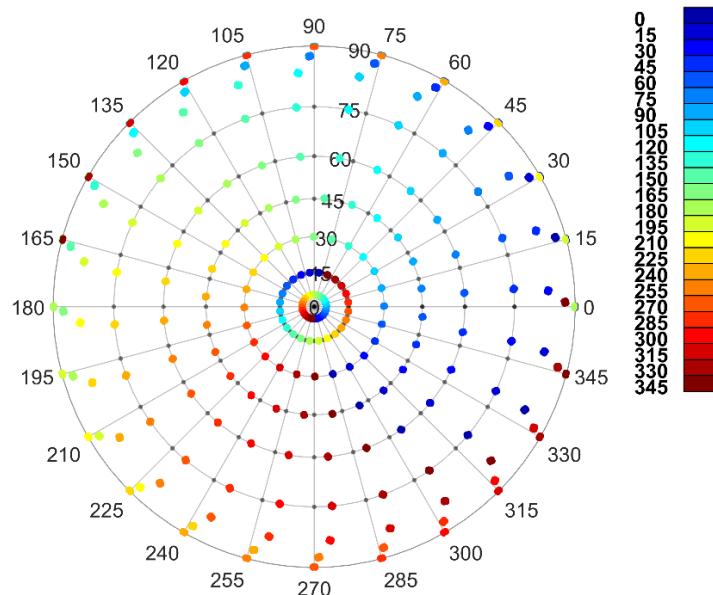


**Fig. 18.** Schematic representation of the main units of the EPSILON diffractometer. 1 – Moderator, 2,3 – Choppers, 4 – Neutron guide, 5 – SKAT diffractometer, 6 – EPSILON diffractometer, 7 – PC-based experiment control.

At present, the EPSILON diffractometer has been modified to conduct neutron texture analysis experiments. For this purpose, nine detector modules, consisting of nine helium counters each, were placed at specific angular positions to maximize pole figure coverage (Fig. 19). At the moment, the detector modules are positioned at angles (relative to the horizontal – X-axis) of  $7.5^\circ$ ,  $-21.3^\circ$ ,  $42.9^\circ$ ,  $65.9^\circ$ ,  $90^\circ$ ,  $118.8^\circ$ ,  $141.8^\circ$ ,  $180^\circ$ ,  $203^\circ$ . This makes it possible to obtain a quasi-regular  $15^\circ$  grid with 24 rotations of a sample.

**Main parameters of EPSILON**

Thermal neutron flux at sample position, $\text{n/cm}^2 \cdot \text{s}^{-1}$	$\sim 1 \cdot 10^6$
Moderator-to-sample distance, m	107
Sample-to-detector distance, m	0.7
Wavelength range, Å	up to 7.1
Scattering angle range	90°
$d_{\text{hkl}}$ -range, Å	up to 5.1
Resolution ( $\Delta d/d, d = 2 \text{ \AA}$ ): at $2\theta = 90^\circ$	0.004
x- y-travel, mm	120
z-travel, mm	40
Sample size, mm	30-60
Characteristic measurement time, h	15-30
Detectors:	81 $^3\text{He}$ counters 120×10 mm



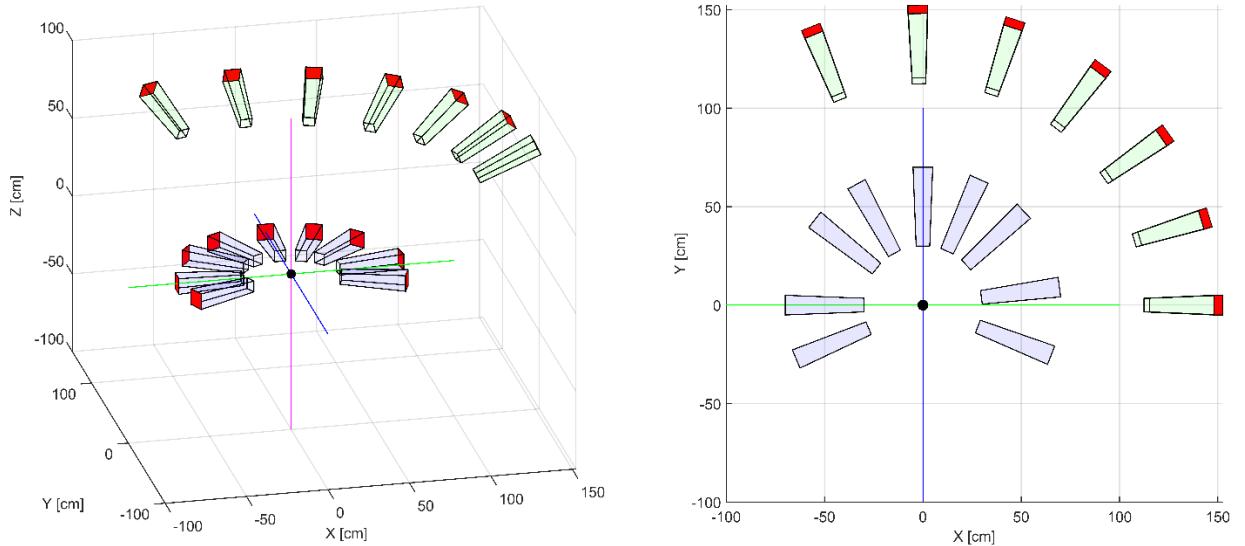
**Fig. 19.** Location of detector centers on the pole figure when rotating a sample in 15° increments.

**4.2. Detailed description of proposals for modernization of the instrument**

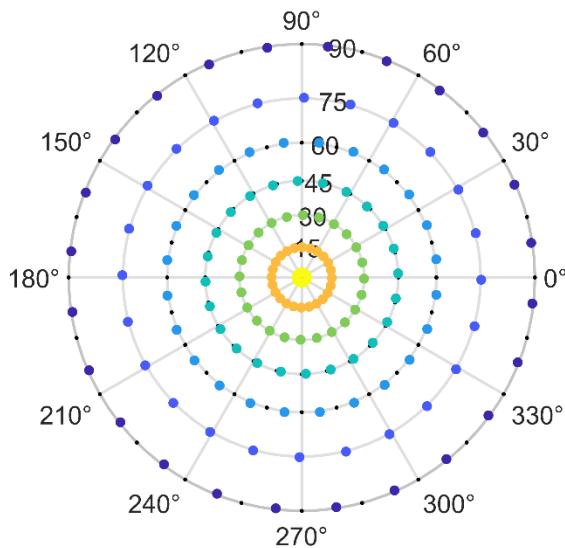
1. It is planned to modernize the detector system. The upgrade includes the purchase and installation of new discriminators, a new high-voltage power supply unit, and the development and construction of a second detector ring (Fig. 20) with an angle of  $2\theta = 65^\circ$  based on helium counters (purchase of helium counters and preamplifiers). The second detector ring consists of seven detector modules, each containing five individual helium

counters. The arrangement of the detector modules on the ring provides a quasi-regular 15-degree grid for pole figure measurements (Fig. 21).

2. To select the optimal ratio of spectral resolution to measurement time, detachable collimators are required. As a result of modifications to the detector system design carried out in 2025, it became possible to quickly install and remove collimators with different angular divergence. It is planned to design, develop and manufacture detachable collimators with different angular divergence both for the existing detector system and for the second ring with an angle of  $2\theta = 65^\circ$ .
3. To expand the range of scientific tasks to be solved with the EPSILON spectrometer, it is planned to use a uniaxial press (up to 200 kN) with a heating element. This device will allow diffraction experiments to be carried out at high temperatures of up to  $600^\circ\text{C}$  while simultaneously applying uniaxial loading. The implementation of these plans requires the purchase of a ready-made commercial product or the design, development and manufacture of a unique press.
4. To expand the range of scientific tasks to be solved with the EPSILON spectrometer, it is planned to equip it with a special heating chamber-furnace that allows for sample rotation. This device will allow diffraction texture experiments to be performed at high temperatures of up to  $600^\circ\text{C}$ . The implementation of these plans requires the purchase of a ready-made commercial product or the design, development and manufacture of a unique chamber-furnace.
5. A coarser angular measurement grid compared to the SKAT diffractometer allows for several texture experiments to be conducted during one day. This requires the development of an automatic sample-changing system. It is planned to design, develop and manufacture such a system.



**Fig. 20.** The arrangement of two detector rings at scattering angles  $2\theta = 90^\circ$  and  $65^\circ$ .



**Fig. 21.** Location of the centers of the detectors of the second ring  $2\theta = 65^\circ$  on the pole figure when rotating a sample in  $15^\circ$  increments.

#### **4.3. Current technical parameters of the instrument in thermal and cold modes of the moderator**

The EPSILON diffractometer is installed on beamline 7A, which is equipped with a cold moderator. When operating in cold moderator mode, the incident cold neutron flux intensity in the wavelength range of 4-13 Å increases by a factor of 7-10, significantly improving the quality of measured diffraction data in the interplanar spacing range  $d_{hkl} > 4$  Å.

#### **4.4. Expected technical parameters after modernization of the instrument**

The modernization of the EPSILON diffractometer is expected to result in an increase in the  $d_{hkl}$ -range by a factor of 1.3; an increase in spectral resolution and the possibility of adapting the ratio of spectral-resolution/scattered neutron collection to experimental requirements; as well as the possibility of conducting neutron diffraction experiments with materials under uniaxial loading at temperatures up to 600°C. An automatic sample-changing system will allow for the maximum number of experiments to be conducted around the clock, thereby saving up to 2 hours of experimental time per day and minimizing wear on the shutter opening/closing mechanism.

#### **4.5. Relevance of the instrument development for the concept of a suite of spectrometers within the project of a new neutron source at JINR**

It is planned to build neutron diffractometers for measuring pole figures and internal strains/stresses at the FLNP new neutron source. The proposed development of the spectrometer into a combined stress/texture diffractometer with a sample environment system and automatic sample changer expands the range of scientific tasks and optimizes the experiment time. The methodological and scientific development of this type of spectrometer is one of the promising and relevant directions in planning the FLNP scientific program.

### **5. Expected scientific results, comparison with the world level**

The EPSILON diffractometer and its scientific program correspond to the highest world level in the field of stress diffractometry. The conducted modification of the instrument, which allows for crystallographic texture measurements, expands the range of scientific tasks to be solved, which makes this spectrometer one of the few capable of measuring pole figures and internal strains. However, the sample environment system is not equipped with high-temperature

heating equipment, and the  $d_{hkl}$ -range needs to be expanded. The proposed variant of development of EPSILON will enable experiments to measure crystallographic textures at various temperatures, including studying changes in crystallographic textures during phase transitions; recording reflections with high  $d_{hkl}$  values, which is relevant for rocks; and experiments to study the effect of temperature and applied uniaxial pressure on stresses arising in materials. Thus, the scope of research topics on EPSILON will expand, which will contribute to the formation of sustained interest in experiments in the fields of geophysics and materials science.

## **6. Requested resources, costs and time frames of instrument modernization**

The costs of new equipment for conducting experiments are given in **Table 7 (Section 4)**.

### **Plan-scheme of activities for the modernization of the EPSILON diffractometer**

<b>Description of activities</b>	<b>2026</b>	<b>2027</b>	<b>2028</b>	<b>2029</b>	<b>2030</b>
Development of a second detector ring for a scattering angle of $2\theta = 65^\circ$					
Purchase of electronics and high-voltage unit					
Design and manufacture of collimators for detector modules					
Design, manufacture and purchase of a furnace					
Design and manufacture of high-temperature press					
Design and manufacture of an automated sample-changing system					

### **References**

1. Lobanov M.L., Yurovskikh A.S., Kardonina N.I., Rusakov G.M. Methods for studying textures in materials. Ekaterinburg, Ural University Press (Publishing house of the Ural Federal University), 2014 (in Russian).
2. Walther, K., Scheffzuek, C., Frischbutter, A. (2000). Neutron time-of-flight diffractometer epsilon for strain measurements: layout and first results. *Physica B, Condensed Matter* 276-278, 130-131.
3. Gary S. Schajer, Philip S. Whitehead Hole Drilling and Ring Coring // Practical residual stress measurement methods / Edited by Gary S. West Sussex. United Kingdom : John Wiley & Sons Ltd, 2013. P. 29-64.
4. T. Lychagina, D. Nikolayev Quantitative comparison of the measured crystallographic textures, *Journal of Applied Crystallography*, 2016, 49(4), 1290-1299.
5. D.P. Kozlenko, S.E. Kichanov, E.V. Lukin, B.N. Savenko “The DN-6 Neutron Diffractometer for High-Pressure Research at Half a Megabar Scale”, *Crystals*, 8, 331 (2018)
6. Kozlenko, D.P., Kichanov, S.E., Lukin, E.V. *et al.* High-Pressure Neutron Diffraction Study of the Crystal and Magnetic Structure of Materials at the Pulsed Reactor IBR-2: Current Opportunities and Prospects. *Crystallogr. Rep.* **66**, 303–313 (2021).
7. D.P. Kozlenko, S.E. Kichanov, E.V. Lukin, A.V. Rutkauskas, G.D. Bokuchava, B.N. Savenko, A.V. Pakhnevich, A.Yu. Rozanov “Neutron Radiography Facility at IBR-2 High Flux Pulsed Reactor: First Results” *Physics Procedia*, 69, 87-91 (2015).
8. D.P. Kozlenko, S.E. Kichanov, E.V. Lukin, A.V. Rutkauskas, A.V. Belushkin, G.D. Bokuchava, B.N. Savenko “Neutron radiography and tomography facility at IBR-2 reactor”, *Physics of Particles and Nuclei Letters*, 13, 3, 346-351 (2016).

## SKAT — Neutron Diffractometer

<b>Leader:</b>	D.I. Nikolaev
<b>Main participants:</b>	T.I. Lychagina, E. Lukin, I. Zel', G.M. Aydanov, employees of NICM Department
<b>Collaborating organizations:</b>	FSUE Central Research Institute of Structural Materials "Prometey" (NRC "Kurchatov Institute"); FSUE All-Russian Scientific Research Institute of Aviation Materials "VIAM" (NRC "Kurchatov institute"); Borissiak Paleontological Institute RAS; National Research Nuclear University MEPhI; IMET RAS; Moscow Polytechnic University

### 1. Abstract

The SKAT diffractometer is successfully operating on beamline 7A-2 of the IBR-2 pulsed reactor. During its operation, a wealth of experience has been gained in respect to the design, development and modernization of the instrument, as well as the development of the technique for measuring spectra to obtain pole figures. Pole figures are necessary to calculate orientation distribution functions, which provide a complete quantitative description of the crystallographic texture in polycrystalline samples. The large cross-section of beamline 7A-2 ( $50 \times 95 \text{ mm}^2$ ) made it possible to build a second ring with detectors, which is a natural way to almost double the efficiency of the instrument. A significant improvement in the quality of data obtained in experiments can be achieved by replacing the neutron guide with a more technologically advanced one.

### 2. Scientific program, relevance and comparison with the world level

The measurement of the crystallographic texture of polycrystals is of great interest for the study of metals, alloys, rocks and ceramics, as well as biological objects. Using the SKAT diffractometer, crystallographic textures of all the above types of polycrystals were successfully measured. The SKAT diffractometer is one of the best neutron instruments in the world for studying crystallographic textures. During the operation of the diffractometer, dozens of scientific articles have been published in international peer-reviewed journals; examples of some publications can be found in [1-15].

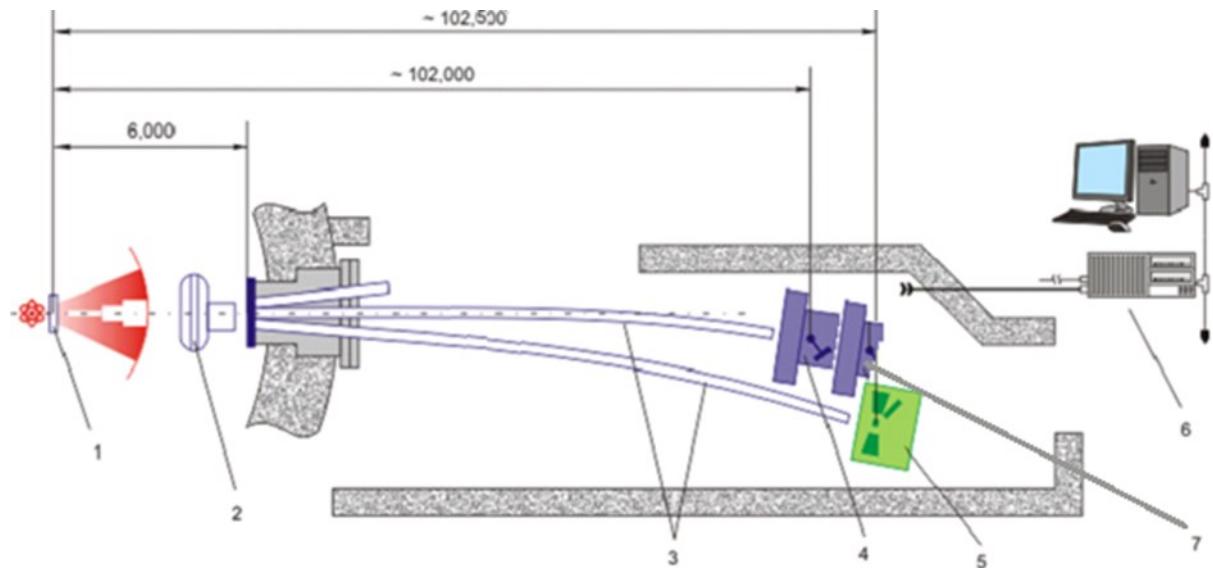
### 3. Scientific and methodological groundwork laid in FLNP JINR

During the operation of the SKAT diffractometer, extensive experience has been accumulated in respect to the design, development, and modernization of the instrument, as well as the development of the technique for measuring spectra and software for obtaining pole figures.

### 4. Current status of the instrument and proposals for its modernization

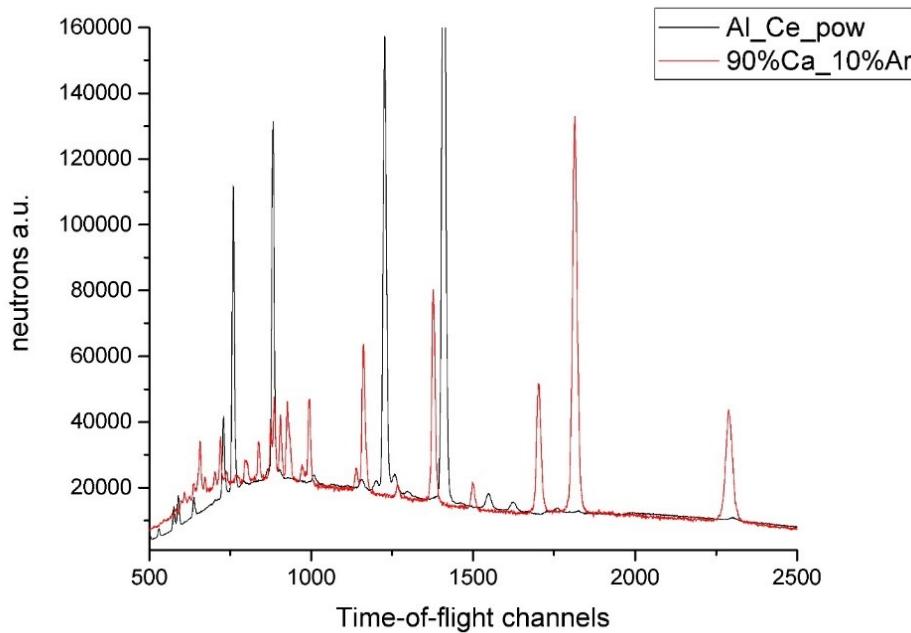
#### 4.1. Information on the current status of the instrument

At present, detectors have been installed on the second ring and first experiments have been conducted to simultaneously measure various powder samples using both rings, allowing us to conclude that independent, simultaneous measurement of two samples is feasible. The schematic diagram of the instrument is shown in Fig. 22.



**Fig. 22.** Schematic diagram of the SKAT instrument: 1 – moderator, 2 – background chopper, 3 – curved neutron guides (evacuated), 4 – first ring of the SKAT texture diffractometer, 5 – EPSILON-MDS diffractometer, 6 – experiment control and data acquisition system, 7 – second ring of the SKAT texture diffractometer.

An example of summed spectra is shown in Fig. 23.



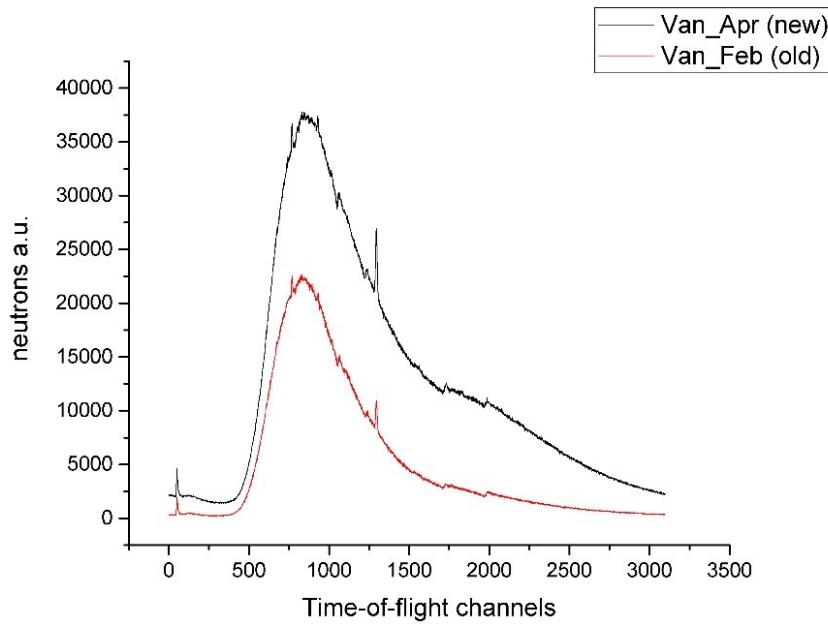
**Fig. 23.** Summed spectra from two simultaneously measured powders.

*Main parameters of the SKAT diffractometer*

<b>Flight path</b>	~ 104 m
<b>Scattering angles <math>2\theta</math></b>	90°
$\lambda_{\max}$	7.0 Å
<b><math>2\theta</math> parameters</b>	$2\theta = 90^\circ$
<b>Maximum resolution <math>\Delta d/d</math></b>	$5.0 \cdot 10^{-3}$
<b>Flight path of the scattered beam</b>	1.10 m
<b>Neutron guide</b>	Cross-section: 50 mm (width) $\times$ 95 mm (height) Radius of curvature: 13400 m Coating: natural Ni (m = 1)
<b>Detectors</b>	Set of 19 detectors based on ${}^3\text{He}$ tubes, $P = 4.5$ bar $\varnothing = 60$ mm.
<b>Collimators</b>	Soller type, Gd coating Angular divergence: 18' / 45' Cross-section: $55 \times 55$ mm $^2$
<b>Sample positioning</b>	3-axis goniometer
<b>Data processing</b>	SONIX+ software package for Windows

**4.2. Detailed description of proposals for modernization of the instrument**

Figure 24 shows the summed spectra for vanadium measured with the old and new detectors on the first ring.



**Fig. 24.** Summed spectra for vanadium.

The spectrum in April 2025 was recorded in cold moderator mode, unlike the spectrum measured in February 2025. This explains the slight difference in the shape of the spectra.

Nevertheless, it can be seen from Fig. 24 that the new detectors can significantly improve the intensity of the measured spectra.

The following changes are proposed:

- Purchase and install new detectors on the second ring, which will significantly improve the intensity of the measured spectra;
- Purchase and install new collimators, which will reduce the intensity loss of the measured spectra;
- Purchase and install new high-voltage units, which will improve the reliability of the measurements.

#### ***4.3. Current technical parameters of the instrument in thermal and cold modes of the moderator***

The SKAT diffractometer allows for the effective study of reflexes in the range of 0.6-5 Å, i.e., for the reconstruction of pole figures corresponding to the crystallographic indices of planes with interplanar spacing in the specified range. The cold moderator provides a better intensity of diffraction reflexes in the range of 2.8-5 Å, with a deterioration in intensity in the range of 0.6-2.7 Å. For structural and biological materials, almost all reflexes are in the range of up to 2.5 Å. At the same time, many geomaterials have reflexes in the range of 2.8-5 Å. Therefore, measurements of geomaterial samples are most effective with the cold mode of the moderator. For measurements of samples of structural and biological materials, the operation of the moderator in the thermal mode is more preferable.

#### ***4.4. Expected technical parameters after modernization of the instrument***

##### ***Main expected parameters of the second ring of the SKAT diffractometer***

<b>Flight path</b>	~ 105 m
<b>Scattering angles <math>2\theta</math></b>	90°
$\lambda_{\max}$	7.0 Å
<b><math>2\theta</math> parameters</b>	$2\theta = 90^\circ$
<b>Maximum resolution <math>\Delta d/d</math></b>	$5.0 \cdot 10^{-3}$
<b>Flight path of the scattered beam</b>	1 m
<b>Neutron guide</b>	Cross-section: 50 mm (width) × 47 mm (height) Radius of curvature: 13400 m Coating: natural Ni (m = 1)
<b>Detectors</b>	Set of 19 detectors based on $^3\text{He}$ tubes, $P = 4.5$ bar $\varnothing = 50$ mm.
<b>Collimators</b>	Soller type, Gd coating Angular divergence: 30' Cross-section: $55 \times 55$ mm $^2$
<b>Sample positioning</b>	1-axis goniometer
<b>Data processing</b>	SONIX+ software package for Windows

#### **4.5. Relevance of the instrument development for the concept of a suite of spectrometers within the project of a new neutron source at JINR**

The relevance of the development of the diffractometer is reflected in the fact that there are 1-2 texture instruments in each neutron center in the world. In addition, FLNP receives numerous external beamtime applications for neutron texture measurements.

#### **5. Expected scientific results, comparison with the world level**

The parameters of the SKAT diffractometer meet international standards, which allows us to confidently speak about the competitiveness of the SKAT as compared to the world analogues. At present, there are about ten instruments (with different degrees of readiness and availability) throughout the world, which can be used to measure full pole figures for coarse-grained samples. In this regard, the parameters of the Russian neutron source—the IBR-2 reactor—allow us to participate in the overall development of neutron sources and improve the SKAT instrument to maintain its high world-class level in terms of global research infrastructure.

#### **6. Requested resources, costs and time frames of instrument modernization**

Upgrading the second detector ring requires purchasing new neutron counters, preamplifier boards, electronics modules, collimators, and high-voltage units.

The costs of the new detector ring and experimental equipment are presented in **Table 8 (Section 4)**.

#### **References**

1. T. Lychagina, D. Nikolayev. Quantitative comparison of the measured crystallographic textures, Journal of Applied Crystallography, 2016, 49(4), 1290-1299.
2. D. Nikolayev, T. Lychagina, A. Zisman, E. Yashina. Directly verifiable neutron diffraction technique to determine retained austenite in steel Advanced Engineering Materials, 2018, DOI 10.1002/adem.20170559.
3. D. Nikolayev, T. Lychagina, M. Rusetsky, A. Ulyanenkov, A. Sasaki. Internal optimization of the texture component approximation method, IOP Conference Series: Materials Science and Engineering, V. 82, Proc. XVII Int. Conf. on Textures of Materials, 2015, <http://dx.doi.org/10.1088/1757-899X/82/1/012007>.
4. T. Lychagina, D. Nikolayev, A. Sanin, J. Tatarko, K. Ullemeyer. Investigation of wheel steel crystallographic texture changes due to modification and thermo-mechanical treatment, IOP Conference Series: Materials Science and Engineering, V. 82, Proc. XVII Int. Conf. on Textures of Materials, 2015, <http://dx.doi.org/10.1088/1757-899X/82/1/012107>.
5. A.A. Zisman, D.I. Nikolaev, T.A. Lychagina, E.A. Yashina. On the Size Effect for Neutron Diffraction by Two-Phase Structures and Applicability of Composite Standards in Analysis of Residual Austenite in Steels. Technical Physics 63 (2018) 1696-1702.
6. A. Ziza, M. Mikhailov, V. Tsukanov, D. Nikolaev, T. Lychagina. A study of retained austenite transformation during high-strength Cr-Ni-Mo-V steel tempering. Lett. Mater. 8 (2018) 146-151.
7. M.L. Fedoseev, S.N. Petrov, A.K. Islamov, N.F. Drozdova, T.A. Lychagina, D.I. Nikolaev. Multimethodological approach to the study of carbide particle dimensions in high-strength steel. Lett. Mater. 8 (2018) 323-328.
8. Nikolayev D, Lychagina T, Pakhnevich A. 2019 Experimental neutron pole figures of minerals composing the bivalve mollusc shells // Springer Nature Applied Sciences V. 1, <https://doi.org/10.1007/s42452-019-0355-1>.
9. Nekhoroshkov P., Zinicovscaia I., Nikolayev D., Lychagina T., Pakhnevich A., Yushin N., Bezuindenhou J. "Effect of the elemental content of shells of the bivalve mollusks (*Mytilus galloprovincialis*) from Saldanha Bay (South Africa) on their crystallographic texture", Biology 10(11) (2021) 1093.
10. A.V. Pakhnevich, D.I. Nikolayev, T.A. Lychagina. Comparison of the Crystallographic Texture of the Recent, Fossil and Subfossil Shells of Bivalves, Paleontological Journal, V.55, pp. 589–599, (2021).

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at the IBR-2 Reactor in 2026-2030

11. Fedoseev M.L., Petrov S.N., Nikolaev D.I., Beskrovny A.I., Lychagina T.A. X-ray and neutron diffraction study of high-strength steel. Methodological aspects. *Voprosy Materialovedeniya*. 2022;(1(109)):07-15. (in Russian) <https://doi.org/10.22349/1994-6716-2022-109-1-07-15>.
12. Pakhnevich, A.; Nikolayev, D.; Lychagina, T. Crystallographic Texture of the Mineral Matter in the Bivalve Shells of *Gryphaea dilatata* Sowerby, 1816, *Biology*, 2022, 11, 1300. <https://doi.org/10.3390/biology11091300>.
13. Pakhnevich, A.; Nikolayev, D.; Lychagina, T.; Balasoiu, M.; Ibram, O. Global Crystallographic Texture of Freshwater Bivalve Mollusks of the Unionidae Family from Eastern Europe Studied by Neutron Diffraction, *Life*, 2022, 12, 730. <https://doi.org/10.3390/life12050730>.
14. Pakhnevich, A.; Nikolayev, D.; Lychagina, T. Global Crystallographic Texture of Pyrite in Fossil Wood (Jurassic, Oryol Region, Russia), *Minerals* 2023, 13, 1050. <https://doi.org/10.3390/min13081050>.
15. A. Pakhnevich, D. Nikolayev and T. Lychagina. Comparison of the global crystallographic texture of minerals in the shells of *Bathymodiolus thermophilus* Kenk et B.R. Wilson, 1985 and species of the genus *Mytilus* Linnaeus, 1758. *Journal of Structural Biology*, Volume 216, Issue 4, December 2024, 108126.

## YuMO — Small-Angle Neutron Scattering Instrument

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**Collaborating organizations:** MSU, MIPT, A.N. Belozersky Institute of Physico-Chemical Biology of MSU, PNPI (NRC “Kurchatov Institute”)

### 1. Abstract

The project aims at the modernization and development of the small-angle neutron scattering instrument operating on beamline 4 of the IBR-2 reactor. The implementation of the project will allow us to improve the quality of obtained data (by changing the detector type, which will gain azimuthal sensitivity), expand the range of available scattering vectors, increase the experimental data acquisition rate, and improve the resolution and background conditions. The development and construction of new position-sensitive detectors, including a direct-beam detector, will make it possible to conduct experiments with anisotropic samples, and expand the possibilities of the sample environment system (magnetic field, fluid rotation, etc.). As a result of the modernization, the already existing wide community of users of the instrument will be significantly enlarged.

### 2. Scientific program, relevance and comparison with the world level

Small-angle neutron scattering is widely applied in the study of the nanoscale structure of matter, being an effective method for investigating fundamental problems and solving important technological challenges. The YuMO small-angle scattering spectrometer is constantly being upgraded [1-18]. Using the YuMO instrument (Fig. 21), research is conducted in the field of condensed matter physics, physical chemistry of dispersed systems, aggregates of surfactants, biophysics and biology, polymeric substances, metallurgy, materials science, etc. [19-70]. The most important feature of small-angle scattering is the possibility of analyzing the structure of disordered systems. This method, for example, is often the only way to obtain structural information about systems with chaotic and partially ordered distribution of density inhomogeneities of 10-10,000 Å in size. It makes it possible to study the dispersed structure of alloys, powders, glasses (phase separation mechanisms, particle size and degree of polydispersity), structural features of polymers in various states of aggregation, weight and geometric characteristics of biological macromolecules and their complexes, biological nanoscale structures such as biological membranes and viruses. The significant difference in the coherent neutron scattering lengths of hydrogen and deuterium together with the possibility of specific deuteration of macromolecules and nanoscale structures make the small-angle neutron scattering technique an indispensable tool for studying biological, colloidal objects, as well as polymers and liquid crystals.

### 3. Scientific and methodological groundwork laid in FLNP JINR

One of the first neutron small-angle instruments that appeared in the world was the setup at ILL. At time-of-flight sources, the first small-angle instrument was built at JINR, at IBR-30 in 1975. Over the past nearly half a century, numerous techniques and developments have been proposed and developed, including a two-detector data acquisition system, direct axial geometry, long neutron flight path and collimation length, the use of a vanadium calibration standard during experiments, and specific detector geometries, including a direct-beam detector. In terms of methodological work, programs for raw data treatment and fitting scattering curves have been successfully created and are being successfully developed in collaboration with colleagues from LIT JINR, and theoretical work on small-angle scattering on deterministic fractals awaits the implementation of corresponding experiments.

The YuMO small-angle scattering spectrometer is the most in-demand instrument at the IBR-2 reactor. During its operation, several hundred journal papers have been published based on the results of experiments conducted at YuMO. The range of research areas using the YuMO instrument is also constantly expanding.

### 4. Current status of the instrument and proposals for its modernization

#### 4.1. Information on the current status of the instrument

At present, the YuMO instrument has a number of unique features, including a two-detector data acquisition system, direct axial experiments, specific geometry of detectors, the use of a direct-beam detector, a wide range of wavelengths, a chopper, and relatively complete automation of the experiment. Primary data processing is carried out almost simultaneously with measurements. Additional sample environment options have been implemented for generating temperature, pressure, and magnetic field on the sample [1-18].

The instrument has a high flux at the sample position, and due to its two-detector system, – a wide dynamic range. The modernization of the instrument carried out earlier opened up new possibilities. At the same time, the creation of a cold moderator with an additional water shell around the thermal and cold parts provides a wide range of wavelengths and requires fine tuning and alignment of the chopper and the collimation system.

As part of the User Program, specialists of the YUMO group have created a sample preparation room with extensive sample storage and preparation options. The instrumentation of the room is constantly being expanded and upgraded to extend the possibilities of technical support for users.

#### 4.2. Detailed description of proposals for modernization of the instrument

The parameters of the instrument that will be improved as a result of the implementation of the project are: the range of available scattering vectors  $Q_{\min} - Q_{\max}$ , the rate of collection of experimental data, the type of neutron detectors, resolution and background conditions.

1. The extension of the Q-range will be achieved by replacing the detectors, eliminating the air gaps along the neutron flight path, and improving the collimation of the neutron beam. Two position-sensitive detectors (PSD) and one direct-beam detector are to be installed. The first PSD will be located in the nearest position (2–8 meters from the sample), in the case of the second PSD, there will be a possibility of changing positions and placing it at a distance from 5 to 13 meters from the sample. The introduction of a two-detector system will require, in its turn, changes in the collimation system, electronic equipment (data acquisition system) and qualitative changes in the software. In addition, a combined analysis of experimental data from different detectors will require methodological improvements in the existing approach. These changes will be based on methodological [7-13] and scientific [14]

achievements, as well as on already partially developed software [4], which will significantly reduce the corresponding project costs.

2. The enhancement of resolution (first of all of spatial resolution of PSD) will require improving the spatial alignment of the detectors, which in turn will call for the development, construction and installation of alignment mechanisms for large-area detectors and vanadium standards.
3. An increase in the data collection rate will be achieved by means of simultaneous detection of scattered neutrons by several detectors arranged in such a way as to cover the entire required (available) range of scattering vectors, by increasing the efficiency and active areas of the detectors, as well as by improving the background conditions.
4. Since the width of the resolution function for the momentum transfer at YuMO is mainly determined by the angular contribution (the contribution from the pulse width is small), its reduction will be achieved by improving the spatial resolution of the detector and of the corresponding collimator.
5. The possibility to measure samples with small scattering cross-sections will be provided by significantly reducing the background with the help of a new chopper and a new collimation system, as well as by using advanced detectors and detector electronics.
6. The range of sample environment equipment will be expanded to enable investigations with additional conditions at the sample. This, in the first place, concerns the possibility of conducting rheological studies, studies under high pressure and in a magnetic field, experiments with sample rotation and in an extended temperature range.
7. The modernization of the instrument will be accompanied by corresponding changes in the software.

#### ***4.3. Current technical parameters of the instrument in thermal and cold modes of the moderator***

Almost all present-day small-angle scattering instruments operate on cold neutron sources. This is primarily due to the need to perform experiments over the widest possible range of momentum transfer  $Q$  and to achieve the lowest possible  $Q$ . The installation of a cold moderator at a steady-state reactor results not only in an increase in the wavelength (and consequently in a decrease in the value of the scattering vector modulus and the appearance of the possibility of studying larger objects), but also in the gain in the neutron flux by more than one order of magnitude. Therefore, there developed a world-wide paradigm of the necessity to use cold moderators. The situation at the IBR-2 pulsed reactor is quite different. The low average reactor power (up to 2 MW) is compensated by the wide range of used wavelengths due to the time-of-flight method. As a result, the YuMO instrument with a direct view of the grooved moderator at room temperature makes it possible (in the absence of a neutron guide) to provide a high neutron flux at the sample position [12].

The use of a cold moderator gives a small gain in the range of small angles with a non-optimal detector position. The flux in the used wavelength range decreases in the cold moderator mode. It is known that the background component at the reactor is determined by the reactor power. Therefore, the signal-to-noise ratio also decreases. This makes it impossible to conduct experiments with low forward scattering intensity, which nowadays make up the majority of experiments with biological and polymeric materials.

The new cold moderator (with mesitylene) and additional cylindrical volumes around it made it possible to increase the flux by a factor of 1.4. But the significant loss in the flux when operating in the thermal mode (especially taking into account the reduction in reactor power) clearly requires continuous operation of the cold moderator. The proposals regarding the design and requirements for the cold moderator outlined in the previous Blue Book 2021-2025

([https://flnp.jinr.int/images/Books/Blue\\_books/Blue\\_Book\\_21-25.pdf](https://flnp.jinr.int/images/Books/Blue_books/Blue_Book_21-25.pdf)) have been implemented. Now both the chopper and the collimator will need to be adapted to operate with the cold moderator.

#### **4.4. Expected technical parameters after modernization of the instrument**

##### **Main parameters of the existing and upgraded YuMO instrument on IBR-2 beamline 4**

<b>Parameters</b>	<b>Before modernization</b>	<b>After modernization</b>
<b>Flux at the sample</b>	$10^7 - 4 \cdot 10^7 \text{ n/(s cm}^2)$	$10^7 - 4 \cdot 10^7 \text{ n/(s cm}^2)$
<b>Wavelength range</b>	from 0.5 Å to 8 Å	from 0.5 Å to 10 Å
<b>Q-range</b>	$8 \cdot 10^{-3} - 0.5 \text{ \AA}^{-1}$	$4 \cdot 10^{-3} - 0.7 \text{ \AA}^{-1}$
<b>Size range of objects under study</b>	10 - 500 Å	8 - 1000 Å
<b>Measured scattering cross section</b>	0.01 cm <sup>-1</sup>	0.005 cm <sup>-1</sup>
<b>Calibration method</b>	V standard during experiment	V, H <sub>2</sub> O, graphite
<b>Collimation type</b>	Axial	Axial
<b>Detector system</b>	2 detectors of scattered neutrons, direct-beam detector	2 PSD of scattered neutrons, direct-beam PSD, monitor
<b>Automatic sample changer</b>	25 samples in thermostatted box	Non-standard samples
<b>Temperature range</b>	from -20°C to +130°C	from -20°C to +200°C
<b>Q-resolution</b>	5 - 20%	2 - 8%
<b>Controlled parameters</b>	Starts, temperature, V-standard, sample position	Starts, temperature, V-standard, sample position, detector position, monitor, chopper, reactor power

#### **4.5. Relevance of the instrument development for the concept of a suite of spectrometers within the project of a new neutron source at JINR**

The key parameter for a small-angle neutron diffractometer is the flux at the sample. At the new neutron source of FLNP, the flux is planned to be increased by a factor of 10. This can significantly improve the parameters of the instrument, reduce the measurement time per experiment, and improve the resolution of the diffractometer. Using a start frequency of 5 Hz or even 10 Hz without an additional satellite peak is the preferred option for the YuMO instrument. An important aspect of this project is the continuation of operation of the spectrometer within the User Program.

The ever-increasing needs of users for specific methods of sample preparation and characterization immediately before experiments require ongoing technical support of the sample preparation room and the development of its instrumentation. Support of the sample preparation room includes servicing its equipment, purchasing and manufacturing specialized cuvettes, sample holders, and consumables.

#### **5. Expected scientific results, comparison with the world level**

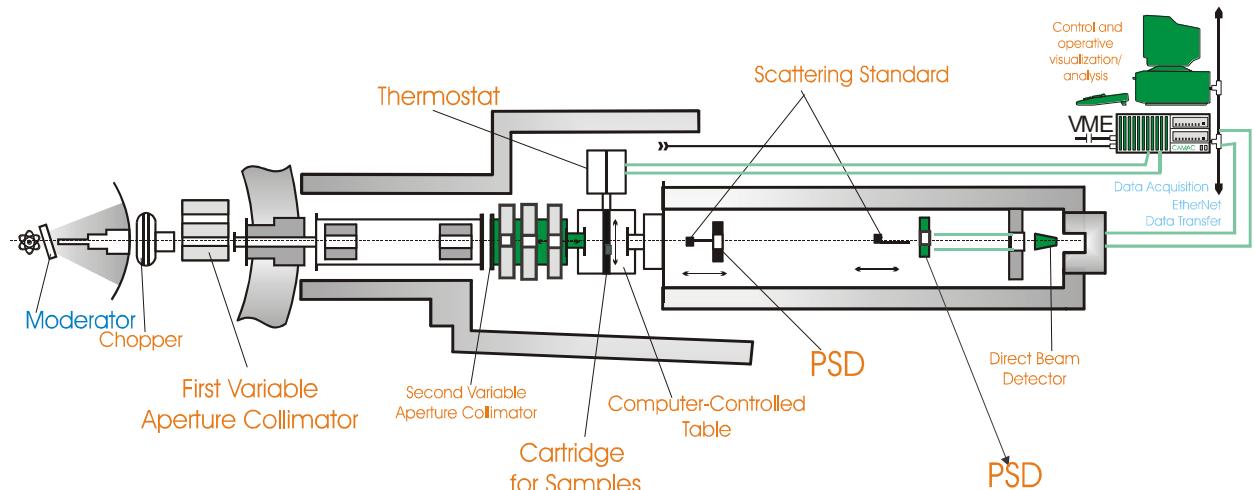
Small-angle neutron scattering is one of the key methods for studying the structure of condensed matter, as evidenced by three international conferences held in Dubna. Every year, up to 100 experiments, both methodological and scientific, are carried out with the instrument. The YuMO spectrometer is the most in-demand instrument within the user policy at IBR-2. It is

expected that the demand for SANS measurements will only grow in the coming years. First of all, this is due to the wide variety of tasks solved with YuMO, ranging from biology to materials science. At the same time, the instrument already allows performing kinetic measurements with a 1-minute exposure. At the new source, with a 10-fold increase in the flux, it will be possible to carry out measurements with a time resolution of tens of seconds, which will significantly expand the range of tasks to be solved. The replacement of the ring detectors with PSD detectors (including the direct-beam detector) will not only broaden the spectrum of research tasks (experiments with a magnetic field, rheological devices, etc.), but also improve the resolution and qualitatively change the data acquisition system. Judging by the publications, some of which are listed below, many publications are already at the highest world level (Scientific Reports, 2019. 9(1): p. 15852. – journal from the Nature Publishing Group, Macromolecules, 2017. 50(1): p. 339-348, BBA, etc.). Taking into account the upcoming changes in the quality of the instrument, it is expected that the level of publications will continue to grow in both quantity and quality. Thus, the modernization will make it possible to qualitatively change the instrument by 2030, provided the project is properly funded.

## 6. Requested resources, costs and time frames of instrument modernization

The work on the project will mainly be carried out by the employees of two FLNP Departments, LIT and the SANS group (YuMO).

The cost and desired timing of production (purchase) of some YuMO units within the project are given in **Table 9 (Section 4)**.



**Fig. 25.** Schematic diagram of the modernized YuMO instrument.

**Plan-scheme of activities for the modernization of the YuMO spectrometer**

<b>№</b>	<b>Description of activities</b>	2025	2026	2027	2028	2029
1	Development and construction of 2D direct-beam detector based on solid-state converter					
2	Development and construction of large-area 2D detectors					
3	Development of algorithms and software for data acquisition from a new type of position-sensitive detector					
4	Replacement, installation and adjustment of limit switches of the adjustable collimator					
5	Optimization of chopper operation					
6	Optimization of the collimation system					
7	Modernization of the units of the instrument, sample table and sample environment system					
8	Support and development of the sample preparation room					

**References**

1. A.I. Kuklin et al., “Small-Angle Neutron Scattering at the Pulsed Reactor IBR-2: Current Status and Prospects,” *Crystallography Reports* 2021 66:2, vol. 66, no. 2, pp. 231–241, Apr. 2021, doi: 10.1134/S1063774521020085.
2. A.V. Vlasov et al., “The Possibilities of Studying Biological Objects on a Pulsed Reactor,” *Biophysics (Russian Federation)*, vol. 68, no. 2, pp. 207–222, Apr. 2023, doi: 10.1134/S0006350923020240/METRICS.
3. Ivankov O.I., Kovalev Y.S., Utrobin P.K., Kutuzov S.A., Soloviev A.G., Rulev M.I. and Gordeliy V.I., Neutronographic investigations of supramolecular structures on upgraded small-angle spectrometer YuMO. *Journal of Physics: Conference Series*, 2017. 848(1): p. 012010.
4. Soloviev A.G., Solovjeva T.M., Ivankov O.I., Soloviov D.V., Rogachev A.V. and Kuklin A.I., SAS program for two-detector system: seamless curve from both detectors. *Journal of Physics: Conference Series*, 2017. 848(1): p. 012020.
5. Kuklin A.I., Ivankov A.I., Soloviov D.V., Rogachev A.V., Kovalev Y.S., Soloviev A.G., Islamov A.K., Balasoiu M., Vlasov A.V., Kutuzov S.A., Sirotin A.P., Kirilov A.S., Skoi V.V., Rulev M.I. and Gordeliy V.I. High-throughput SANS experiment on two-detector system of YuMO spectrometer. in *Journal of Physics: Conference Series*. 2018.
6. Kuklin A.I., Rogachev A.V., Soloviov D.V., Ivankov O.I., Murugova T.N., Chupin V.V., Rulev M.I., Skoi V.V., Kucerka N., Vlasov A.V. and Gordeliy V.I., SANS investigations of biological objects on a YuMO spectrometer: results and possibilities. *Journal of Bioenergetics and Biomembranes*, 2018. 50(6): p. 555–555.
7. Soloviev A., Kutuzov S., Ivankov O. and Kuklin A., Primary Data Treatment Software for Position-Sensitive Detector of Small-Angle Neutron Scattering Spectrometer in the Isotropic Pattern Scattering Case. *EPJ Web Conf.*, 2018. 173: p. 05015.
8. Zabelskii D.V., Vlasov A.V., Ryzhykau Y.L., Murugova T.N., Brennich M., Soloviov D.V., Ivankov O.I., Borshchevskiy V.I., Mishin A.V., Rogachev A.V., Round A., Dencher N.A., Büldt G., Gordeliy V.I. and Kuklin A.I., Ambiguities and completeness of SAS data analysis: Investigations of apoferritin by SAXS/SANS EID and SEC-SAXS methods. *J. Phys. Conf. Ser.*, 2018. 994.
9. Erhan R.V., Manoshin S., Pepy G., Kuklin A.I., Belushkin A.V. and Zamfir N.V., A concept for the modernization of a SANS instrument at the IBR-2M pulsed reactor. *Nuclear Instruments and Methods*

in Physics Research Section A: Accelerators, Spectrometers, Detectors and Associated Equipment, 2011. 634 (1, Supplement): p. S161-S164.

- 10. Kuklin A.I., Kovalev Y.S., Kutuzov S.A., Ivankov O.I., Rogachev A.V., Soloviov D.V. and Gordeliy V.I. The status and perspectives of small-angle neutron scattering YUMO spectrometer: Book of abstracts of the SANS-YuMO user Meeting. in SANS-YuMO user Meeting at the start-up of scientific experiments on IBR-2M devoted to the 75th anniversary of Yu.M Ostanevich's birth. 2011. FLNP, Dubna, Russia.
- 11. Kuklin A.I., Rogachev A.V., Cherny A.Y., E.B. D., Islamov A.K., Kovalev Y.S., Murugova T.N., Soloviev D.V., Ivankov O.I., Soloviev A.G. and Gordeliy V.I., Do the size effects exist? Romanian Journal of Physics, 2011. 56(1-2): p. 134-140.
- 12. Kuklin A.I., Rogov A.D., Gorshkova Y.E., Utrobin P.K., Kovalev Y.S., Rogachev A.V., Ivankov O.I., Kutuzov S.A., Soloviev D.V. and Gordeliy V.I., Analysis of Neutron Spectra and Fluxes Obtained with Cold and Thermal Moderators at IBR-2 Reactor: Experimental and Computer Modelling Studies. Peper Letters, 2011. 8(2): p. 200-217.
- 13. Kuklin A.I., Soloviev D.V., Rogachev A.V., Utrobin P.K., Kovalev Y.S., Balasoiu M., Ivankov O.I., Sirotin A.P., Murugova T.N., Petukhova T.B., Gorshkova Y.E., Erhan R.V., Kutuzov S.A., Soloviev A.G. and Gordelii V.I., New opportunities provided by modernized small-angle neutron scattering two-detector system instrument (YuMO). Journal of Physics: Conference Series, 2011. 291(1): p. 012013.
- 14. Kuklin A.I., Utrobin P.K., Ivankov O.I., Sirotin A.P., Soloviov D.V. and Gordeliy V.I. The hydrostatic high pressure setup for the small angle neutron scattering spectrometer Yumo: Book of abstracts of SANS-YUMO user meeting. in SANS-YuMO user Meeting at the start-up of scientific experiments on IBR-2M devoted to the 75th anniversary of Yu.M Ostanevich's birth. 2011. FLNP, JINR, Dubna, Russia.
- 15. Kuklin A.I., Balasoiu M., Kutuzov S.A., Kovalev Y.S., Rogachev A.V., Erhan R.V., Smirnov A.A., Kirilov A.S., Ivankov O.I., Soloviov D.V., Kappel W., Stancu N., Cios M., Cios A. and Gordeliy V.I., Magnetic system for small angle neutron scattering investigations of nanomaterials at YuMO-SANS instrument. Journal of Physics: Conference Series, 2012. 351(1): p. 012022.
- 16. Kuklin A.I., Murugova T.N., Ivankov O.I., Rogachev A.V., Soloviov D.V., Kovalev Y.S., Ishchenko A.V., Zhigunov A., Kurkin T.S. and Gordeliy V.I., Comparative study on low resolution structures of apo ferritin via SANS and SAXS. Journal of Physics: Conference Series, 2012. 351(1): p. 012009.
- 17. Nyam-Osor M., Soloviov D.V., Kovalev Y.S., Zhigunov A., Rogachev A.V., Ivankov O.I., Erhan R.V. and Kuklin A.I., Silver behenate and silver stearate powders for calibration of SAS instruments. Journal of Physics: Conference Series, 2012. 351(1): p. 012024.
- 18. Soloviev A.G., Murugova T.N., Islamov A.H. and Kuklin A.I., FITTER. The package for fitting a chosen theoretical multi-parameter function through a set of data points. Application to experimental data of the YuMO spectrometer. Journal of Physics: Conference Series, 2012. 351(1): p. 012027.
- 19. Zeleňáková A., Hrubovčák P., Kapusta O., Kučerka N., Kuklin A., Ivankov O. and Zeleňák V., Size and distribution of the iron oxide nanoparticles in SBA-15 nanoporous silica via SANS study. Scientific Reports, 2019. 9(1): p. 15852.
- 20. Shibaev A.V., Abrashitova K.A., Kuklin A.I., Orekhov A.S., Vasiliev A.L., Iliopoulos I. and Philippova O.E., Viscoelastic Synergy and Microstructure Formation in Aqueous Mixtures of Nonionic Hydrophilic Polymer and Charged Wormlike Surfactant Micelles. Macromolecules, 2017. 50(1): p. 339-348.
- 21. Shibaev A.V., Makarov A.V., Kuklin A.I., Iliopoulos I. and Philippova O.E., Role of Charge of Micellar Worms in Modulating Structure and Rheological Properties of Their Mixtures with Nonionic Polymer. Macromolecules, 2018. 51(1): p. 213-221.
- 22. Gibhardt H., Haramagatti C.R., Islamov A.K., Ivankov O.I., Kuklin A.I. and Eckold G., Ordering Phenomena in Surfactant Systems: From Micellar Solutions to Gel and Crystalline Phases. Zeitschrift Fur Physikalische Chemie-International Journal of Research in Physical Chemistry & Chemical Physics, 2015. 229(10-12): p. 1869-1885.
- 23. Selivanova O.M., Surin A.K., Ryzhykau Y.L., Glyakina A.V., Suvorina M.Y., Kuklin A.I., Rogachevsky V.V. and Galzitskaya O.V., To Be Fibrils or To Be Nanofilms? Oligomers Are Building Blocks for Fibril and Nanofilm Formation of Fragments of Abeta Peptide. Langmuir, 2018. 34(6): p. 2332-2343.

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at the IBR-2 Reactor in 2026-2030

24. Shibaev A.V., Tamm M.V., Molchanov V.S., Rogachev A.V., Kuklin A.I., Dormidontova E.E. and Philippova O.E., How a Viscoelastic Solution of Wormlike Micelles Transforms into a Microemulsion upon Absorption of Hydrocarbon: New Insight. *Langmuir*, 2014. 30(13): p. 3705-3714.
25. Shibaev A.V., Kuklin A.I. and Philippova O.E., Different responsiveness to hydrocarbons of linear and branched anionic/cationic-mixed wormlike surfactant micelles. *Colloid and Polymer Science*, 2018.
26. Zabrodskaya Y.A., Lebedev D.V., Egorova M.A., Shaldzhyan A.A., Shvetsov A.V., Kuklin A.I., Vinogradova D.S., Klopov N.V., Matusevich O.V., Cheremnykh T.A., Dattani R. and Egorov V.V., The amyloidogenicity of the influenza virus PB1-derived peptide sheds light on its antiviral activity. *Biophysical Chemistry*, 2018. 234: p. 16-23.
27. Feldman T.B., Ivankov O.I., Kuklin A.I., Murugova T.N., Yakovleva M.A., Smitienko O.A., Kolchugina I.B., Round A., Gordeliy V.I., Belushkin A.V. and Ostrovsky M.A., Small-angle neutron and X-ray scattering analysis of the supramolecular organization of rhodopsin in photoreceptor membranes. *Biochimica et Biophysica Acta (BBA) - Biomembranes*, 2019.
28. Lebedev D.V., Zabrodskaya Y.A., Pipich V., Kuklin A.I., Ramsay E., Sokolov A.V., Elizarova A.Y., Shaldzhyan A.A., Grudinina N.A., Pantina R.A., Wu B., Shtam T.A., Volnitskiy A.V., Shmidt A.E., Shvetsov A.V., Isaev-Ivanov V.V. and Egorov V.V., Effect of Alpha-Lactalbumin and Lactoferrin Oleic Acid Complexes on Chromatin Structural Organization. *bioRxiv*, 2019: p. 675223.
29. Nabiyev A.A., Olejniczak A., Pawlukojc A., Balasou M., Bunoiu M., Maharramov A.M., Nuriyev M.A., Ismayilova R.S., Azhibekov A.K., Kabyshev A.M., Ivankov O.I., Vlase T., Linnik D.S., Shukurova A.A., Ivanshina O.Y., Turchenko V.A. and Kuklin A.I., Nano-ZrO<sub>2</sub> filled high-density polyethylene composites: Structure, thermal properties, and the influence  $\gamma$ -irradiation. *Polymer Degradation and Stability*, 2019: p. 109042.
30. Bairamukov V.Y., Kuklin A., Orlova D. and Lebedev V., Structure of Yttrium Bis-Phthalocyanine Pyrolyzed Derivatives. *Journal of Surface Investigation: X-ray, Synchrotron and Neutron Techniques*, 2019. 13(5): p. 793-801.
31. Vlasov A., Murugova T., Grudinin S., Ivankov O., Soloviov D., Rogachev A., Round A., Ryzhykau Y., Mishin A., Balandin T., Borshchevskiy V., Gordeliy V. and Kuklin A., Protein structure and structural ordering versus concentration dependence. *Febs Journal*, 2014. 281: p. 593-594.
32. Murugova T.N., Vlasov A.V., Ivankov O.I., Rogachev A.V., Ryzhykau Y.L., Soloviov D.V., Zhigunov A., Zinovev E.V., Kovalev Y.S., Round A., Gordeliy V.I. and Kuklin A.I., Low resolution structural studies of apoferritin via SANS and SAXS: the effect of concentration. *Journal of Optoelectronics and Advanced Materials*, 2015. 17(9-10): p. 1397-1402.
33. Kulvelis Y.V., Ivanchev S.S., Primachenko O.N., Lebedev V.T., Marinenko E.A., Ivanova I.N., Kuklin A.I., Ivankov O.I. and Soloviov D.V., Structure and property optimization of perfluorinated short side chain membranes for hydrogen fuel cells using orientational stretching. *Rsc Advances*, 2016. 6(110): p. 108864-108875.
34. Boikova A.S., Dyakova Y.A., Ilina K.B., Konarev P.V., Kryukova A.E., Kuklin A.I., Marchenkova M.A., Nabatov B.V., Blagov A.E., Pisarevsky Y.V. and Kovalchuk M.V., Octamer formation in lysozyme solutions at the initial crystallization stage detected by small-angle neutron scattering. *Acta Crystallogr D Struct Biol*, 2017. 73(Pt 7): p. 591-599.
35. Cherny A.Y., Anitas E.M., Osipov V.A. and Kuklin A.I., Small-angle scattering from the Cantor surface fractal on the plane and the Koch snowflake. *Physical Chemistry Chemical Physics*, 2017. 19(3): p. 2261-2268.
36. Golub M., Combet S., Wieland D.C.F., Soloviov D., Kuklin A., Lokstein H., Schmitt F.J., Olliges R., Hecht M., Eckert H.J. and Pieper J., Solution structure and excitation energy transfer in phycobiliproteins of *Acaryochloris marina* investigated by small angle scattering. *Biochimica et Biophysica Acta (BBA) - Bioenergetics*, 2017. 1858(4): p. 318-324.
37. Shibaev A.V., Makarov A.V., Aleshina A.L., Rogachev A.V., Kuklin A.I. and Philippova O.E., Structure and oil responsiveness of viscoelastic fluids based on mixed anionic/cationic wormlike surfactant micelles. *Journal of Physics: Conference Series*, 2017. 848(1): p. 012019.
38. Bratanov D., Kovalev K., Machtens J.-P., Astashkin R., Chizhov I., Soloviov D., Volkov D., Polovinkin V., Zabelskii D. and Mager T., Unique structure and function of viral rhodopsins. *Nature communications*, 2019. 10(1): p. 1-13.
39. Kulvelis Y.V., Primachenko O., Odinokov A., Shvidchenko A., Bayramukov V.Y., Gofman I., Lebedev V., Ivanchev S., Vul A.Y. and Kuklin A., Composite proton-conducting membranes with nanodiamonds. *Fullerenes, Nanotubes and Carbon Nanostructures*, 2020. 28(2): p. 140-146.

40. Almasy L., Kuklin A.I., Požar M., Baptista A. and Perera A., Microscopic origin of the scattering pre-peak in aqueous propylamine mixtures: X-ray and neutron experiments versus simulations. *Physical Chemistry Chemical Physics*, 2019. 21(18): p. 9317-9325.
41. Kazantsev A.S., Vlasov A.V., Ryzhykau Y.L., Zabelskii D.V., Murugova T.N., Ivankov O.I., Rogachev A.V., Zinovev E.V., Kurbatov N.M., Gordeliy V.I. and Kuklin A.I., SAXS studies of apoferritin in different pH with consideration of dimers. *Journal of Bioenergetics and Biomembranes*, 2018. 50(6): p. 548-548.
42. Nabihev A.A., Linnik D.S., Gorshkova Y.E., Maharramov A.M., Balasoiu M., Olejniczak A., Ivankov O.I., Kovalev Y.S. and Kuklin A.I., Influence of gamma irradiation on the morphological properties of HDPE+%ZrO<sub>2</sub> polymer nanocomposites. *Romanian Journal of Physics*, 2019. 64(5-6).
43. Kwiatkowski A.L., Molchanov V.S., Sharma H., Kuklin A.I., Dormidontova E.E. and Philippova O.E., Growth of wormlike micelles of surfactant induced by embedded polymer: role of polymer chain length. *Soft Matter*, 2018. 14(23): p. 4792-4804.
44. Rulev M.I., Pavlova A.A., Ivankov O.I., Soloviov D.V., Rogachev A.V., Skoi V.V., Chupin V.V., Gordeliy V.I. and Kuklin A.I., SAS investigations of size changes in lipid vesicles near temperature point of phase transition. *Journal of Bioenergetics and Biomembranes*, 2018. 50(6): p. 569-569.
45. Kulvelis Y.V., Shvidchenko A.V., Aleksenskii A.E., Yudina E.B., Lebedev V.T., Shestakov M.S., Dideikin A.T., Khozyaeva L.O., Kuklin A.I., Török G., Rulev M.I. and Vul A.Y., Stabilization of detonation nanodiamonds hydrogel in physiological media with poly(vinylpyrrolidone). *Diamond and Related Materials*, 2018. 87: p. 78-89.
46. L Anghel, A Rogachev, A Kuklin, RV Erhan.  $\beta$ -Lactoglobulin associative interactions: a small-angle scattering study. *European Biophysics Journal* 48 (3), 285-295.
47. A.I. Kuklin et al., "Small-Angle Neutron Scattering at the Pulsed Reactor IBR-2: Current Status and Prospects," *Crystallography Reports* 2021 66:2, vol. 66, no. 2, pp. 231–241, Apr. 2021, doi: 10.1134/S1063774521020085.
48. A.V. Vlasov et al., "The Possibilities of Studying Biological Objects on a Pulsed Reactor," *Biophysics (Russian Federation)*, vol. 68, no. 2, pp. 207–222, Apr. 2023, doi: 10.1134/S0006350923020240/METRICS.
49. Y. L. Ryzhykau et al., "Ambiguities in and completeness of SAS data analysis of membrane proteins: the case of the sensory rhodopsin II-transducer complex," *Acta Crystallogr D Struct Biol*, vol. 77, no. 11, pp. 1386–1400, Oct. 2021, doi: 10.1107/s2059798321009542.
50. I.O. Bezruchko et al., "Capabilities of SEC-SAXS Method in Research of Polydisperse Systems: The Case of Apoferritin," *Biochem (Mosc) Suppl Ser A Membr Cell Biol*, vol. 19, no. 1, pp. 20–26, Mar. 2025, doi: 10.1134/S1990747824700405/FIGURES/3.
51. S. D. Ivashchenko and A. V. Vlasov, "Elucidating the Distinctions between Open-State Monomers and Dimers of Human Tissue Transglutaminase," *Physics of Particles and Nuclei Letters*, vol. 21, no. 4, pp. 823–826, Aug. 2024, doi: 10.1134/S1547477124701450/METRICS.
52. Y.L. Ryzhykau et al., "Molecular model of a sensor of two-component signaling system," *Sci Rep*, vol. 11, no. 1, p. 10774, Dec. 2021, doi: 10.1038/s41598-021-89613-6.
53. V. V. Sudarev et al., "Ferritin self-assembly, structure, function, and biotechnological applications," *Int J Biol Macromol*, vol. 224, pp. 319–343, Jan. 2023, doi: 10.1016/J.IJBIOMAC.2022.10.126.
54. T. Kondela, E. Dushanov, M. Vorobyeva, K. Mamakulov, E. Drolle, D. Soloviov, P. Hrubovčák, K. Kholmurodov, G. Arzumanyan, Z. Leonenko, N. Kučerka, Investigating the competitive effects of cholesterol and melatonin in model lipid membranes, *Biochimica et Biophysica Acta (BBA) - Biomembranes*, 1863 (2021) 183651. doi:10.1016/j.bbamem.2021.183651
55. S.A. Kurakin, E.V. Ermakova, A.I. Ivankov, S.G. Smerdova, N. Kučerka, The Effect of Divalent Ions on the Structure of Bilayers in the Dimyristoylphosphatidylcholine Vesicles, *Journal of Surface Investigation: X-ray, Synchrotron and Neutron Techniques*, 15 (2021) 211-220. doi:10.1134/S1027451021020075
56. O. Ivankov, T.N. Murugova, E.V. Ermakova, T. Kondela, D.R. Badreeva, P. Hrubovčák, D. Soloviov, A. Tsarenko, A. Rogachev, A.I. Kuklin, N. Kučerka, Amyloid-beta peptide (25–35) triggers a reorganization of lipid membranes driven by temperature changes, *Scientific Reports*, 11 (2021) 21990. doi:10.1038/s41598-021-01347-7
57. S. Kurakin, O. Ivankov, E. Dushanov, T. Murugova, E. Ermakova, S. Efimov, T. Mukhametzyanov, S. Smerdova, V. Klochkov, A. Kuklin, N. Kučerka, Calcium ions do not influence the A $\beta$ (25–35) triggered morphological changes of lipid membranes, *Biophysical Chemistry*, 313 (2024) 107292. doi:10.1016/j.bpc.2024.107292

58. O. Ivankov, T. Kondela, E.B. Dushanov, E.V. Ermakova, T.N. Murugova, D. Soloviov, A.I. Kuklin, N. Kučerka, Cholesterol and melatonin regulated membrane fluidity does not affect the membrane breakage triggered by amyloid-beta peptide, *Biophysical Chemistry*, 298 (2023) 107023. doi:10.1016/j.bpc.2023.107023
59. S. Kurakin, O. Ivankov, V. Skoi, A. Kuklin, D. Uhríková, N. Kučerka, Cations Do Not Alter the Membrane Structure of POPC—A Lipid With an Intermediate Area, *Frontiers in Molecular Biosciences*, 9 (2022) 926591. doi:10.3389/fmolb.2022.926591
60. O. Ivankov, D.R. Badreeva, E.V. Ermakova, T. Kondela, T.N. Murugova, N. Kučerka, Anionic lipids modulate little the reorganization effect of amyloid-beta peptides on membranes, *Gen Physiol Biophys*, 42 (2023) 59-66. doi:10.4149/gpb\_2022052
61. Napol'skiy, F., M. Avdeev, M. Yerdauletov, O. Ivankov, S. Bocharova, S. Ryzhenkova, B. Kaparova, K. Mironovich, D. Burlyayev , V. Krivchenko, On the Use of Carbon Nanotubes in Prototyping the High Energy Density Li-ion Batteries. *Energy Technology*, 2020. 8(6): p. 2000146. DOI: 10.1002/ente.202000146.
62. Siposova, K., V.I. Petrenko, O.I. Ivankov, A. Musatov, L.A. Bulavin, M.V. Avdeev , O. Kyzyma, Fullerenes as an Effective Amyloid Fibrils Disaggregating Nanomaterial. *ACS Applied Materials & Interfaces*, 2020. DOI: 10.1021/acsami.0c07964.
63. Nabiiev, A.A., A. Olejniczak, A.K. Islamov, A. Pawlukojc, O.I. Ivankov, M. Balasoiu, A. Zhigunov, M.A. Nuriyev, F.M. Guliyev, D.V. Soloviov, A.K. Azhibekov, A.S. Doroshkevich, O.Y. Ivanshina , A.I. Kuklin, Composite Films of HDPE with SiO<sub>2</sub> and ZrO<sub>2</sub> Nanoparticles: The Structure and Interfacial Effects. *Nanomaterials*, 2021. 11(10). DOI: 10.3390/nano11102673.
64. Safarik, I., J. Prochazkova, M.A. Schroer, V.M. Garamus, P. Kopcansky, M. Timko, M. Rajnak, M. Karpets, O.I. Ivankov, M.V. Avdeev, V.I. Petrenko, L. Bulavin , K. Pospiskova, Cotton Textile/Iron Oxide Nanozyme Composites with Peroxidase-like Activity: Preparation, Characterization, and Application. *ACS Applied Materials & Interfaces*, 2021. 13(20): p. 23627-23637. DOI: 10.1021/acsami.1c02154.
65. Murugova, T.N., O.I. Ivankov, Y.L. Ryzhykau, D.V. Soloviov, K.V. Kovalev, D.V. Skachkova, A. Round, C. Baeken, A.V. Ishchenko, O.A. Volkov, A.V. Rogachev, A.V. Vlasov, A.I. Kuklin , V.I. Gordeliy, Mechanisms of membrane protein crystallization in 'bicontinuous'. *Scientific Reports*, 2022. 12(1): p. 11109. DOI: 10.1038/s41598-022-13945-0.
66. Petrenko, V.I., L.C. Fernandes, O.I. Ivankov, C.R. Tubio, M. Tariq, J. M.S.S. Esperança, D.M. Correia , S. Lanceros-Mendez, Structural organization of ionic liquids embedded in fluorinated polymers. *Journal of Molecular Liquids*, 2022: p. 119385. DOI: 10.1016/j.molliq.2022.119385.
67. Putz, A.-M., O.I. Ivankov, A.I. Kuklin, V. Ryukhtin, C. Ianăși, M. Ciopec, A. Negrea, L. Trif, Z.E. Horváth , L. Almásy, Ordered Mesoporous Silica Prepared in Different Solvent Conditions: Application for Cu(II) and Pb(II) Adsorption. *Gels*, 2022. 8(7): p. 443. DOI: 10.3390/gels8070443.
68. Tomchuk, O.V., N.O. McHedlov-Petrossyan, O.A. Kyzyma, N.N. Kriklya, L.A. Bulavin, Y.L. Zabulonov, O.I. Ivankov, V.M. Garamus, E. Ōsawa, M.V. Avdeev, Cluster-cluster interaction in nanodiamond hydrosols by small-angle scattering. *Journal of Molecular Liquids*, 2022. 354: p. 118816. DOI: 10.1016/j.molliq.2022.118816.
69. Zeleňáková, A., P. Hrubovčák, A. Berkutova, O. Šofranko, N. Kučerka, O. Ivankov, A. Kuklin, V. Girmán, V. Zeleňák, Gadolinium-oxide nanoparticles for cryogenic magnetocaloric applications. *Scientific Reports*, 2022. 12(1): p. 2282. DOI: 10.1038/s41598-022-06132-8.
70. Krakovský, I., T.V. Tropin, O.I. Ivankov, V. Petrenko, Investigation of the Interaction of Ionic Surfactants with Epoxy-Based Hydrogels by SANS. *Langmuir*, 2025. DOI: 10.1021/acs.langmuir.4c04062.

## GRAINS — Neutron Reflectometer with Horizontal Sample Plane



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### 1. Abstract

The GRAINS (GRAzing Incidence Neutron Scattering) time-of-flight neutron reflectometer with a horizontal sample plane is located at beamline 10-B of the IBR-2 reactor. The instrument is designed to study nanostructured interfaces in the solid and liquid states by measuring the specular reflection coefficient and the intensity of diffuse scattering of a beam of thermal/cold neutrons. The modernization is aimed at improving the quality of neutron reflectometry experiments, including the reduction in the measurement time per reflectivity curve, increasing the range of momentum transfer, reducing the background, and widening of the capabilities of the sample environment system for conducting *in situ* experiments under various conditions.

### 2. Scientific program, topicality and comparison with the world level

The GRAINS (GRAzing Incidence Neutron Scattering) time-of-flight neutron reflectometer with a horizontal sample plane is located at beamline 10-B of the IBR-2 reactor [1,2]. The instrument is designed to study nanostructured interfaces in the solid and liquid states by measuring the specular reflection coefficient and the intensity of diffuse scattering of a beam of thermal/cold neutrons. The classical analysis of specular reflection in neutron reflectometry (NR) allows reconstructing the profile of the scattering length density in the studied object along the direction perpendicular to the interface between the media over a depth of up to several hundred nanometers with a resolution of 1 nm. The analysis of off-specular (diffuse) neutron scattering provides the characterization of lateral correlations on surfaces and interlayer boundaries. The use of specialized liquid cells in neutron reflectometry allows one to vary the contrast in the reflectometry experiment at interfaces with liquid media and to obtain more detailed information on the structure of surface inhomogeneities at a level of 1-100 nm. The cold mode of operation of the neutron moderator available for beamline 10 allows measurements in a wider dynamic range of momentum transfer.

**The scientific program** includes the following areas related to the physics of soft matter:

- Liquid-containing interfaces
- Adsorption of nanoparticles (including magnetic nanoparticles) on solid surfaces;
- Phase separation in liquid solutions and melts at interfaces
- Electrochemical interfaces;
- Thin films, including polymer and composite films;
- Biological macromolecules and lipid layers on the surface;
- Neutron supermirror coatings.

The GRAINS project includes a wide configuration of modern capabilities of neutron reflectometry in horizontal geometry. First of all, it involves the creation of conditions for complex studies of interfaces containing liquid components with the help of unpolarized and polarized neutrons and the testing of methods for obtaining and analyzing specular reflection and off-specular (diffuse) scattering. The GRAINS reflectometer has been included in the FLNP User Program since 2015 and is available for interdisciplinary research [3-16].

### **3. Scientific and methodological groundwork laid in FLNP JINR**

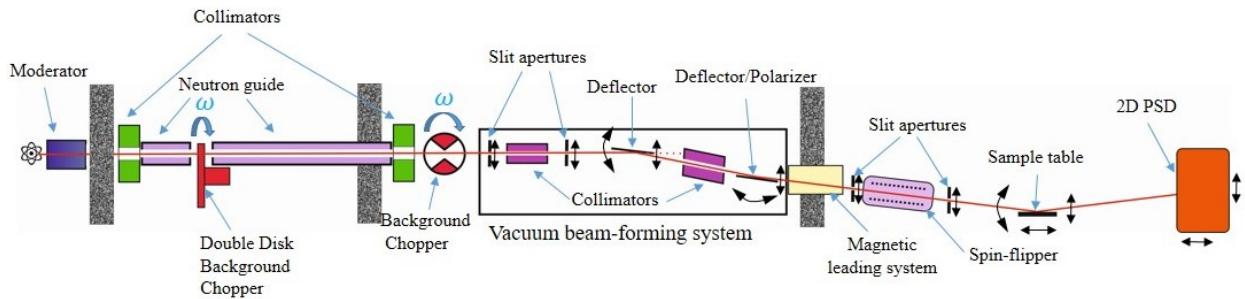
When designing GRAINS, we used the experience of development of two other successfully operating reflectometers at the IBR-2 reactor (REMUR, REFLEX) [17] with a vertical sample plane. The GRAINS reflectometer implements geometry with a horizontal sample plane (vertical scattering plane), which opens up possibilities for studying interfaces with liquid media. So far, two neutron reflectometers of this type have been created at the neutron sources of the Russian Federation: REVERANCE (steady-state reactor VVR-M, PNPI NRC KI, Gatchina) [18, 19] and HORIZON (pulsed spallation source IN-06, INR RAS, Moscow) [20, 21].

### **4. Current status of the instrument and proposals for its modernization**

#### ***4.1. Information about the current status of the instrument***

The GRAINS reflectometer (its schematic layout is shown in Fig. 22) is located at the IBR-2 reactor, beamline 10-B. The main elements of the reflectometer are the moderator, neutron guide, chopper, beam-forming system, sample table and detector system. The characteristics of the units of the GRAINS reflectometer are given in Table 1. The beam is formed using a set of collimating devices and a long (up to 1 m) deflecting mirror – a principal element that separates the thermal neutron beam from fast background neutrons and directs it to the horizontally placed interface at a certain angle. Then, the reflected or scattered beam enters the detector system. The axis of the channel is directed to the moderator, which can operate in two modes: cold (22 K) and thermal (300 K). The experimental samples are fixed in special holders in a horizontal plane on a five-axis goniometric table (HUBER), which is mounted on an anti-vibration platform (JRS Scientific Instruments). The sample holder is provided with thermostating (Julabo F25-MA thermostat) in the temperature range from  $-15$  to  $+180$   $^{\circ}\text{C}$ . The reflectometer is equipped with a platform for remotely changing the position of the sample (up to 10 positions depending on the size of the sample).

Neutrons behind the sample are detected by a detector system consisting of the main large-area gas two-dimensional position-sensitive detector (2D PSD) and an additional standard gas point detector. PSD is a multiwire proportional chamber with delay-line readout with universal data acquisition and storage system (developed in SC Department of DCMRD, FLNP JINR [22, 23]). The detectors are mounted on a movable platform placed behind the sample at a certain distance (Fig. 22); the plane of PSD and the axis of the counter are always perpendicular to the beam axis. The experiment is fully automated, control is provided (including remote access control) using the universal SONIX+ software package for the IBR-2 spectrometers (developed in SC Department of DCMRD, FLNP JINR [24-26]).



*Fig. 22. Schematic layout of the GRAINS reflectometer, current configuration.*

During five years after the GRAINS reflectometer was put into operation and included in the suite of instruments for users, it has proved effective in *in situ* studies of solid/liquid interfaces. This is due to a comparatively small characteristic measurement time per reflectivity curve: 1 hour in the dynamic range  $q_z = 0.05-1.2 \text{ nm}^{-1}$ . Measurements in the maximum possible range using several grazing angles are limited only by strongly scattering systems due to a relatively high background: the minimum reflectivity is  $10^{-5}$ .

#### **4.2. Detailed description of proposals for modernization of the instrument**

The modernization is aimed at improving the quality of NR experiments. The main focus is on expanding the capabilities for conducting interdisciplinary investigations, including the development of a sample environment system for conducting NR experiments under various conditions.

##### **1) Putting into service of additional background chopper**

At present, there is one drum chopper at the instrument: horizontal slit, rotation frequency of 5 Hz, fixed transmission function, released in 2010 (SiNaTech Ltd., Gatchina). This chopper ensures the performance of the reflectometer. There is a disk-type chopper installed in the beamline (under adjustment): two disks with opposite rotation, rotation frequencies of 5 and 10 Hz, variable transmission function, manufactured in 2019 (MIRROTRON Ltd., Budapest).

##### **2) Improvement of the detector system**

Improvement of the detector system basically implies the installation and adaptation of a vacuum tube after the sample holder to reduce the contribution of the air rescattering to the background component after reflection. This will enable experiments on grazing incidence small-angle neutron scattering (GISANS).

##### **3) Replacement of the polarizer**

At present, the polarizer (manufactured in 2010) has become unusable. It needs to be replaced with a fundamental change in the design of the polarizer unit: replacing the metal support of the long (up to 1 m) polarizing mirrors with a polymer one and replacing two mirrors with one long mirror.

##### **4) Magnetic system**

In 2019, for reflectometry measurements in external magnetic fields with controlled field strength, an electromagnet from GMW Associates, USA, was purchased (H-shape, dipole electromagnet, current of up to 70 A, gap of up to 96 mm, maximum field strength of 1.5 T, weight – 700 kg). The installation and adaptation of this magnet require the creation of a special sample unit with the development, manufacture and installation of a magnet cooling and power supply system.

## 5) Sample environment system

The development of a sample environment system is associated with the creation and production of specialized reflectometry cells for *in situ* studies at interfaces of different types (liquid/solid, liquid/air) under various conditions (temperature, external electric field, external magnetic field, humidity, pressure, etc.). It is planned to design and test new specialized cells with adjustable conditions of various types, including changes in: (i) temperature and humidity conditions in the ranges of 10-200°C and 30-100%, respectively; (ii) the electromagnetic field in directions parallel and perpendicular to the surface under study; (iii) the scattering density of the solvent due to isotopic substitution (contrast variation). The work is characterized by high technological requirements for the processing of cell components made of metals and plastics, the use of specialized materials, and the creation of specialized planar structures on the surface of crystalline substrate blocks (silicon, quartz). This work also includes the organization of grinding and redeposition of planar structures on crystalline substrate blocks after the experiment.

### 4.3. Current technical parameters of the instrument in thermal and cold modes of the moderator

<b>Neutron wavelength range, nm</b>	0.05 - 1.0 (cold) 0.05 - 0.7 (thermal)
<b>Grazing angle, mrad</b>	0 - 25
<b><math>q_z</math>-interval covered, nm<sup>-1</sup></b>	0.05 - 2
<b>Angle resolution, %</b>	2 - 10
<b>Neutron flux at sample position, cm<sup>-2</sup>·s<sup>-1</sup></b>	1 (cold) - 2 (thermal) $\times 10^6$
<b>Sample dimensions, cm</b>	(2 $\times$ 2) - (7 $\times$ 15)
<b>Deflecting mirror</b>	Supermirror NiTi, $m = 2$ , $L = 1$ m
<b>Detectors</b>	2D PSD, <sup>3</sup> He, 20 $\times$ 20 cm, spatial resolution 2 $\times$ 2 mm 1D cylindrical counter, <sup>3</sup> He, $\varnothing$ 18 mm, $L = 190$ mm

### 4.4. Expected technical parameters after modernization of the instrument

<b>Neutron wavelength range, nm</b>	0.05 - 1.5 (cold) 0.05 - 0.7 (thermal)
<b>Grazing angle, mrad</b>	0-25
<b><math>q_z</math>-interval covered, nm<sup>-1</sup></b>	0.05-2
<b>Angle resolution, %</b>	2-10
<b>Neutron flux at sample position, cm<sup>-2</sup>·s<sup>-1</sup></b>	1 (cold) - 2 (thermal) $\times 10^6$
<b>Sample dimensions, cm</b>	(2 $\times$ 2) - (7 $\times$ 15)
<b>Deflecting mirror</b>	Supermirror NiTi, $m = 2$ , $L = 1$ m
<b>Detectors</b>	2D PSD, <sup>3</sup> He, 20 $\times$ 20 cm, spatial resolution 2 $\times$ 2 mm 1D cylindrical counter, <sup>3</sup> He, $\varnothing$ 18 mm, $L = 190$ mm
<b>Polarizer</b>	Supermirror, $m = 2$ , $L = 1$ m
<b>Analyzer</b>	Fan-type, $m = 2$
<b>Sample environment system</b>	External magnetic field, 1.5 T Thermostat, -50 $\div$ +150°C

#### **4.5. Relevance of the instrument development for the concept of a suite of spectrometers within the project of a new neutron source at JINR**

The development of reflectometry at IBR-2 in the direction of studying interfaces in soft matter is fully consistent with modern world trends. At almost all neutron sources of both pulsed and steady-state types, neutron reflectometers with a horizontal sample plane have been actively developed and put into operation over the past ten years with an emphasis on the use for soft matter research. It should be noted that the time-of-flight technique is mainly implemented at steady-state sources.

#### **5. Expected scientific results, comparison with the world level**

Layered phospholipid structures, including soybean phospholipids, are of current interest for drug delivery. Neutron reflectometry will be used to study the simultaneous effects of temperature and humidity on the structure of layers on substrates. Using heavy water as a basis for specific humidity conditions during the interaction and absorption of D<sub>2</sub>O by the bilayer at a planar interface allows for changes in the scattering length density causing changes in specular reflection. This makes it possible to track the rate of water penetration and its distribution across the layer depth, as well as to conclude about changes in the structural parameters of the membrane as a whole. Neutron reflectometry applied to planar interfaces will also be used to identify and analyze the interactions of various drugs with the lipid bilayer and their penetration into model membranes. Studying the structure of such systems under various conditions is an important step in developing their practical applications.

Polymer brushes are a class of surface coatings consisting of macromolecules attached to the surface. They have been widely studied in recent years regarding their potential applications, including effective lubricants, stimuli responsive intelligent coatings and protective coatings. A particular interest is related to specific polymer passivations of chemically inert surfaces for studying individual molecules and their associates (micelles). Various methods exist for “grafting” polymer chains onto planar surfaces with varying densities. Neutron reflectometry can be well used for characterization of final polymer layers on quartz surfaces when varying components and synthesis conditions (pH, temperature, salt conditions), including “wet” and “dried” states, to determine the coating density over the interface depth and to study the swelling of polymer chains on surfaces in various environments. Potential for scaling films over large areas for adsorption of macromolecules from solution will be studied.

Neutron reflectometry will be used to study thin composite catalyst films. An effective method for enhancing the activity of catalysts is their distribution on surfaces through nanoscale inclusions in various matrices. The catalytic properties of such films depend on the size of the nanoparticles and the interactions between them. For example, using tetraethoxysilane-based silica sols containing platinum and palladium compounds, it is possible to create effective catalytic layers for metal oxide-based gas sensors, including those sensitive to carbon dioxide. Neutron reflectometry will be used to determine the structural parameters of the films and track the evolution of the nanoparticle depth distribution in different conditions.

A classic problem well studied for bulk systems is the study of phase separation in mixed liquid systems. The semi-empirical theory of this phenomenon created so far allows a fairly complete classification of the types of separation depending on the interaction potentials in the components of solutions. At present, an extremely interesting problem is the study of the behavior of mixed solutions at interfaces. Modern structural methods, such as neutron reflectometry in the horizontal geometry of the sample, will make it possible to advance in solving this problem.

The interest in systems containing magnetic nanoparticles is primarily connected with the possibility to control the properties of these systems by means of an external magnetic field. These

systems include magnetic fluids, magnetic polymer films, Langmuir-Blodgett films with magnetic nanoinclusions, magnetic gels, including elastomers, and other magnetic composite materials. Particular prospects are associated with the use of magnetic nanoparticles in biomedical applications, such as targeted drug delivery to tumors; tumor diagnostics (magnetic resonance imaging), tumor therapy (magnetic hyperthermia).

A remote sample exchange platform (up to 10 positions depending on size) operating at GRAINS enables rapid tests of batches of identical samples, in particular, non-polarizing neutron supermirrors. Multilayer neutron supermirrors are currently used in most experimental neutron scattering facilities. Characterization and control of interlayer roughness are a special focus in developing mass production of supermirrors on extended substrates using magnetron deposition. Various algorithms are being considered for roughness modulation control in a multiparameter system with a large number of internal interfaces. These algorithms can be effectively studied using specular and non-specular neutron reflection. The research will involve step-by-step characterization of the quality of model supermirrors simulating various structures for different deposition stages.

In terms of its characteristics, including the available range of momentum transfer, resolution, and measurement time of a typical reflectivity curve, the GRAINS reflectometer is comparable to other instruments at international research centers—the SURF reflectometer at the ISIS pulsed source, as well as reflectometers on medium-flux steady-state reactors such as GINA (BNC, 10 MW) and NREX+ (MLZ, 20 MW). A significant advantage is the pulsed nature of the neutron source, which enables the efficient use of time-of-flight techniques for *in situ* experiments.

## 6. Requested resources, costs and time frames of instrument modernization

The modernization period is 3 years. The cost of the main units of GRAINS is given in **Table 10 (Section 4)**.

## References

1. M.V.Avdeev, V.I.Bodnarchuk, V.V.Lauter-Pasyuk, H.Lauter, V.L.Aksenov, S.P.Yaradaikin, V.A.Ulyanov, V.A.Trounov, S.I.Kalinin, Project of the new multifunctional reflectometer GRAINS with horizontal sample plane at the IBR-2M pulsed reactor in Dubna *J. Phys.: Conf. Ser.* 251 (2010) 012060
2. M.V. Avdeev, V.I.Bodnarchuk, V.I.Petrenko, I.V.Gapon, O.V.Tomchuk, A.V.Nagorny, V.A.Ulyanov, L.A.Bulavin, V.L.Aksenov. Neutron time-of-flight reflectometer GRAINS with horizontal sample plane at the IBR-2 reactor: possibilities and prospects. *Crystallography reports* 62(6) (2017) 1002-1008
3. Kutsenko V.Y., Artykulnyi O.P., Petrenko V.I., Avdeev M.V., Marchenko O.A., Bulavin L.A., Snegir S.V.. Isotope effect in heavy/light water suspensions of optically active gold nanoparticles. *Applied Nanoscience* 9(5) (2019) pp. 957-963
4. Kiselev M.A., Selyakov D.N., Gapon I.V., Ivankov A.I., Ipatova O.M., Aksenov V.L., Avdeev M.V., Investigation of Nanodrug Phospholipovit by Small-Angle Neutron Scattering. *Crystallography Reports* 64 (2019) 656-661
5. Nagornyi A.V., Petrenko V.I., Rajnak M., Gapon I.V., Avdeev M.V., Dolnik B., Bulavin L.A., Kopcansky P., Timko M.. Particle assembling induced by non-homogeneous magnetic field at transformer oil-based ferrofluid/silicon crystal interface by neutron reflectometry. *Applied Surface Science* 473 (2019) 912–917.
6. M.V.Avdeev, A.A.Rulev, E.E.Ushakova, Ye.N.Kosiachkin, V.I.Petrenko, I.V.Gapon, E.Yu.Kataev, V.A.Matveev, L.V.Yashina, D.M.Itkis. On nanoscale structure of planar electrochemical interfaces metal/liquid lithium ion electrolyte by neutron reflectometry. *Applied Surface Science* 486 (2019) 287–291
7. Hrubovčák P., Dushanov E., Kondela T., Tomchuk O., Kholmurodov K., Kučerka N., “Reflectometry and molecular dynamics study of the impact of cholesterol and melatonin on model lipid membranes”, *Eur. Biophys. J.*, 50 (2021) 1025-1035.

8. Tropin T.V., Karpets M.L., Kosiachkin Ye., Gorshkova Y.E., Gapon I.V., Aksenov V.L., "Evaluation of fullerenes C<sub>60</sub>/C<sub>70</sub> layers in polystyrene thin films by neutron and X-ray reflectometry", Fuller. Nanotub. Carbon Nanostructures, 29 (10) (2021) 819-824.
9. Tomchuk O.V., Krasnikov D.V., Kosiachkin Y.N., Ilatovskii D.A., Avdeev M.V., Nasibulin A.G., "Specular reflectometry studies of alcohol-induced densification for thin films of single-walled carbon nanotubes", J. Surf. Investig.: X-ray Synchrotron Neutron Tech., 15 (2021) 773-776.
10. Artykulnyi O.P., Avdeev M.M., Kosiachkin Y.M., Petrenko V.I., Safarik I., Bulavin L.A., "Neutron investigation of interaction between anionic surfactant micelles and poly(ethylene glycol) polymer brush system", Nucl. Phys. Atom. Energy, 22(2) (2021) 149-156.
11. Kosiachkin Y.N., Gapon I.V., Rulev A.A., Ushakova E.E., Merkel D., Bulavin L.A., Avdeev M.V., Itkis D.M., "Structural Studies of Electrochemical Interfaces with Liquid Electrolytes Using Neutron Reflectometry: Experimental Aspects", J. Surf. Investig: X-ray Synchrotron Neutron Tech., 15(4) (2021) 787-792.)
12. Karpets M., Rajnak M., Petrenko V., Gapon I.V., Avdeev M.V., Bulavin L.A., Timko M., Kopcanský P., Electric field-induced assembly of magnetic nanoparticles from dielectric ferrofluids on planar interface, Journal of Molecular Liquids, 2022, Volume 362, p. 119773.
13. Socoliu V., Avdeev M.V., Kuncser V., Turcu R., Tombácz, E., Vékás L., Ferrofluids and bio-ferrofluids: looking back and stepping forward, Nanoscale, 2022, Volume 14, Issue 13, pp. 4786–4886.
14. Bobrikov I.A., Gapon I.V., Avdeev M.V., Application of Neutron Scattering to Study Materials and Transition Processes in Lithium Energy Storage Devices at the IBR-2 Pulsed Reactor, Physics of Particles and Nuclei, 2022, Volume 53, Issue 3, pp. 674–696.
15. M.V. Avdeev, N.A. Ivanova, Temperature Effect in Aqueous Solutions of Trisiloxane Surfactants at the Interface With Titanium Oxide Studied by Neutron Reflectometry, Journal of Surface Investigation, V. 17, No. 2, pp. 445-449, 2023).
16. Tropin T.V., Avdeev M.V., Aksenov V.L., Temperature Dependence of Structural Parameters of Thin Films of Polystyrene–Fullerene C<sub>60</sub>/C<sub>70</sub> Nanocomposite According to Neutron Reflectometry Data, Journal of Surface Investigation: X-ray, Synchrotron and Neutron Techniques, 2024, Vol. 18, No. 4, pp. 765–769)
17. Aksenov V.L., Nikitenko Yu.V., Crystallography Reports 52(3), 2007, 540–549.
18. Radzhabov, A.K. , Gordeev, G.P. , Aksel'rod, L.A. , Lazebnik, I.M. , Zabenkin, V.N., Crystallography Reports , 52(3) (2007) 561–564
19. Radzhabov A.K., Gordeev G.P., Lazebnik I.M. et al. // Physica B: Cond. Matter. 2007. V. 397. № 1–2. P. 156.
20. Litvin, V.S. , Trunov, V.A. , Bulkin, A.P. et al., Journal of Surface Investigation , 4(6), 2010, 873–878
21. Litvin V.S., Alekseev A.A., Sadykov R.A. et al., J. Phys. Conf. Ser. 2012. V. 340. P. 012032.
22. Belushkin, A.V., Bogdzel', A.A. , Zhuravlev, V.V., et al., Physics of the Solid State , 52(5), 2010, 1025–1028
23. Kulikov S.A., Prikhodko V.I. // Physics of Particles and Nuclei. 2016. V. 47. № 4. P. 702.
24. Petukhova T.B., Kirilov A.S., JINR Communications, Dubna, 2006. P10-2006-27.
25. Kirilov, A.S. , Murashkevich, S.M. , Okulov, R.Yu. , Petukhova, T.B., Instruments and Experimental Techniques , 52(1), 2009, 37–42
26. Kirilov A.S., Physics of Particles and Nuclei Lett. 2016. V. 13. № 1. P. 132.

## REFLEX — Reflectometer with Polarized Neutrons

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**Main participants:** V.V. Sadilov

**Collaborating organizations:** PNPI NRC KI, «DonIPE» (Donetsk)

### 1. Abstract

The REFLEX polarized neutron reflectometer is used for time-of-flight studies of thin-film magnetic and non-magnetic nanostructured objects at room temperature. Its long flight path allows measurements with high time and angular resolution.

In addition to scientific research, the REFLEX reflectometer is used to perform methodological studies on diagnostics of the quality of neutron-optical structures, as well as to study and develop new polarized neutron techniques.

### 2. Scientific program, relevance and comparison with the world level

The REFLEX reflectometer is located on beamline 9 of the IBR-2 reactor. The feature of the beamline is that it is positioned tangentially to the moderator, i.e. the radiating surface is the flat end part of the thermal water moderator. As a result, the neutron flux density on beamline 9 is lower than that on the beamlines of other two IBR-2 reflectometers (REMUR and GRAINS), whose beam axes originate from the flat part of the cold moderator. In addition, the head part of the REFLEX reflectometer, which is located in the ring corridor of the reactor, is not optimal because of the limitations imposed by the technological equipment of the reactor and the system of cold moderators. This limits the possibilities for effective background suppression at the beamline and narrows the possibilities of reflectometry measurements at the instrument. Nevertheless, the availability of a polarized beam and good angular resolution of the instrument make it possible to solve a certain range of problems related to the investigation of magnetism of layered nanosystems, interlayer diffusion processes, surface roughness, etc.

To increase the efficiency of using beamline 9, the REFLEX reflectometer is mainly used in methodological research. It is actively employed for testing new neutron-optical systems, polarization control devices, detector systems, etc. One of the promising areas of methodological development is the creation of a neutron spin-echo instrument with a time-of-flight mode. The spin-echo method is quite well developed for steady-state neutron sources [1-4]. It is based on the use of either coils with a constant magnetic field or radio-frequency spin flippers as precession arms. In both cases, the working spectral region is limited to a value of the order of  $\Delta\lambda/\lambda \sim 15\text{-}20\%$ , which in most cases is substantially less than the total spectral range of the pulsed source. In this regard, the use of the spin-echo technique on pulsed sources is limited, although some attempts have been made to adapt the existing techniques to the time-of-flight mode [5].

For pulsed neutron sources, two methods of using spin-echo techniques have been proposed. The first one consists in using adiabatic RF spin-flippers in the precession arms [6, 7]. This method has been brought to practical implementation at the OFF-SPEC reflectometer of the ISIS pulsed source [8]; this mode is currently undergoing debugging and tuning for the instrument. The second method was proposed in [9] and is based on the use of spin-rotators in the precession arms, in which magnetic fields increase linearly with time. In practice, the magnetic fields in this method are modulated in time (sawtooth-shaped magnetic pulses). This approach in the variant of elastic spin echo for small-angle neutron scattering is being developed at the REFLEX instrument. The wide spectral range that can be used within the framework of this method will cover the area of

studied objects on a scale from 100 Å to 15000 Å, which will significantly supplement the capabilities of existing small-angle scattering instruments at the IBR-2 reactor. Furthermore, the neutron spin-echo method does not require high beam collimation, and polarization analysis is performed for the entire scattered beam without scanning over a specific range of solid angles. This feature compensates for the comparatively low flux at the REFLEX instrument, thus making the method most appropriate for further effective development of the instrument.

### **3. Scientific and methodological groundwork laid in FLNP JINR**

The REFLEX reflectometer team has extensive experience in creating and developing instruments for the IBR-2 reactor and other sources [10-12]. In the frames of the modernization of the REFLEX reflectometer, numerous units and components were successfully designed and integrated into the operating instrument.

As part of the project on the design of a small-angle spin-echo instrument in collaboration with colleagues from the Jülich Research Center (Germany), all elements of the spin-echo setup, including electronic power supply units for spin-rotators, were developed and tested at the IBR-2. Along with this work, the feasibility of implementing this technique at the IBR-2 reactor was experimentally demonstrated.

Using the VITESS software package for Monte Carlo simulation of neutron instruments, a virtual model of the future prototype of a small-angle spin-echo scattering instrument was developed as an option for REFLEX [13] and the main parameters of the setup were optimized.

To date, the necessary power supplies, a cooling system for the power electronics, and the spin-rotator coils have been developed and manufactured.

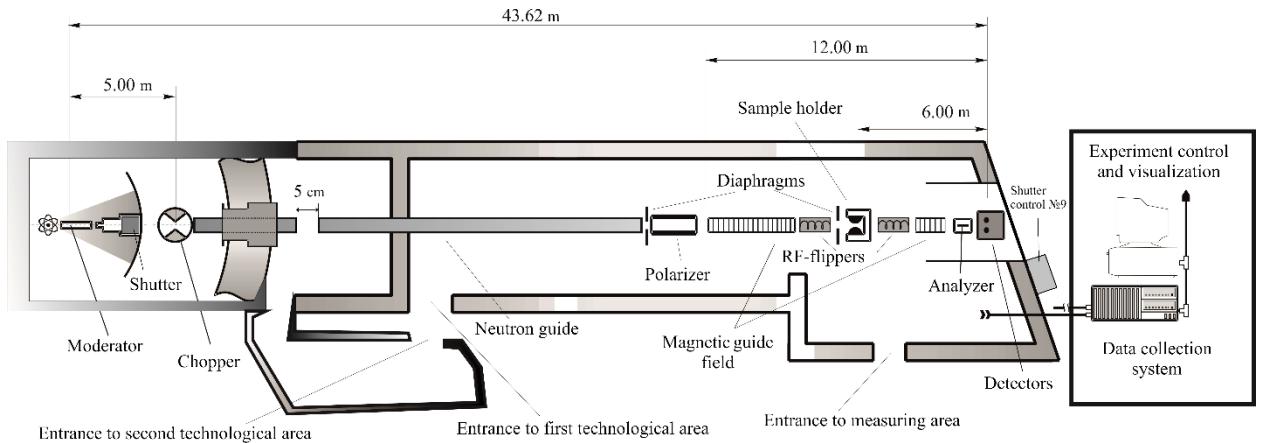
### **4. Current status of the instrument and proposals for its modernization**

The REFLEX instrument is currently included in the FLNP User Program, within the framework of which planned reflectometry experiments with solid samples are carried out. Most of submitted experimental proposals concern the analysis of specular reflection from various layered nanostructures and are aimed at determining the structural characteristics of thin films: layer thicknesses, roughness of interfaces and free surfaces, scattering density characteristics of the material within the layers. For the analysis of structures containing layers of magnetic materials, the REFLEX reflectometer offers a polarized neutron beam option with a possibility of full polarization analysis of the beam scattered (specularly reflected) from the sample.

#### ***4.1. Information about the current status of the instrument***

The equipment upgrade carried out in recent years has significantly improved the REFLEX parameters and simplified the measurement process. However, there are a number of factors that limit the capabilities of the reflectometer. For example, the head part of the REFLEX reflectometer, which is located in the ring corridor of the reactor, is not optimal because of the constraints imposed by the technological equipment of the reactor and the system of cold moderators. This limits the possibilities for effective background suppression at the beamline. A relatively high background imposes limitations on the dynamic range of the measured reflectivity curves. The background chopper installed in the ring corridor was designed to be placed on a flight path twice as long as its actual position, and therefore, the time window of the chopper does not sufficiently suppress the background of thermal neutrons. The first section of the neutron guide is installed at a distance of more than 5 m from the shutter. As a result, the neutron guide collects the flux from an area significantly larger than the shutter opening, which also worsens the background conditions for reflectometry measurements.

Active work is currently underway to improve the background conditions at the REFLEX instrument in order to maximize its potential.



**Fig. 27. Layout of the REFLEX instrument.**

### **Main parameters of the REFLEX reflectometer**

<b>Beam-forming system</b>	supermirror (m=1.2) neutron guide 27 m long, 10×80 mm <sup>2</sup>
<b>Wavelength range</b>	1.4 - 10 Å
<b>Q-range</b>	0.001- 0.13 Å <sup>-1</sup>
<b>Neutron flux at sample position</b>	10 <sup>5</sup> s <sup>-1</sup> cm <sup>-2</sup>
<b>Q-resolution</b>	3 - 10 %
<b>Sample-to-detector distance</b>	2 - 6 m
<b>Minimum sample dimensions</b>	20×20 mm <sup>2</sup>
<b>Magnetic field at sample position</b>	<0.4 T
<b>Spin-flippers</b>	2 radio-frequency adiabatic spin-flippers
<b>Polarizer</b>	Transmission type, V-shape, Fe/Si, m=5
<b>Analyzer</b>	Transmission type, Fe/Si, m=3.6 (also supermirror, m=5, is available)
<b>Detectors</b>	2D PSD 200×200 mm <sup>2</sup> , <sup>3</sup> He; <sup>3</sup> He proportional counter

### **4.2. Detailed description of proposals for modernization of the instrument**

The long-term development of the REFLEX instrument is planned to focus on the following areas:

#### **4.2.1. Sample environment equipment**

- Modernization of the magnetic system at the sample location. The magnet should produce a magnetic field at the sample position of at least 1 T.
- Development and installation of a cryogenic system to generate low temperatures of down to 2 K at the sample.

The modernization of the sample environment system will allow studies of magnetic thin-film structures with a low Curie temperature, superconducting films near the superconducting transition temperature. At present, these studies cannot be performed at the instrument.

#### ***4.2.2. Modernization of automation system***

- Installation of automated collimation systems: a) at the exit of the neutron guide, b) in front of the sample, c) behind the sample.
- Development and installation of a new table at the sample position with a system for adjusting the sample.
- Development of automated control of the position of the detector system (for movement perpendicular to the beam axis when setting up a convenient measurement mode).

#### ***4.2.3. Modernization of background chopper***

The reflectometry method is extremely sensitive to both the quality of neutron pulse formation by the mechanical chopper and background conditions. The available mechanical drum chopper for the REFLEX instrument has a large neutron transmission window (40 ms). During the development of the chopper, there was no complete clarity regarding the possibility of its installation in the ring corridor of the reactor, so the window was designed taking into account the possible placement at a large distance from the moderator. The long-term use of the chopper in the ring corridor has practically exhausted its resource for reliable operation. It is necessary to develop a more reliable design of the chopper with the provided options for regulating the delays and the window width of the neutron pulse. Therefore, the design and installation of a new mechanical chopper on beamline 9 of the IBR-2 reactor is a first-priority task for the development of the REFLEX instrument for its operation in both the reflectometry mode and the spin-echo mode currently under development.

#### ***4.2.4. Background suppression***

The creation of enhanced background shielding around the neutron guide is also an important task for the development of the REFLEX instrument. The section in the beginning of the neutron guide in the ring corridor of the IBR-2 reactor is of particular importance, since at the moment it does not have protection from radiation coming from neighboring beamlines. In the framework of the REFLEX instrument development, it is planned to develop and construct shielding walls and housings made of neutron-absorbing materials (including borated polyethylene) in the IBR-2 reactor ring corridor.

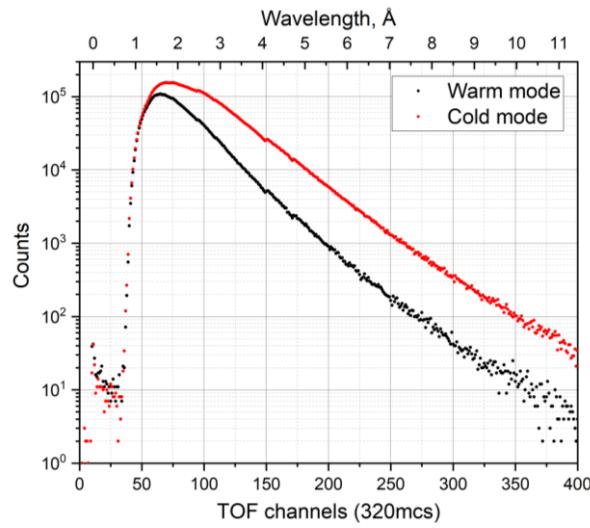
It is also necessary to develop and install background shielding of the detector system, as well as to provide an option for installation of a vacuum tube between the sample and the detector system.

#### ***4.2.5. Creation of a small-angle scattering spin-echo mode***

The spin-echo technique for elastic neutron scattering has been developing at the REFLEX instrument for a long time. Over the next five years, it is planned to create a separate module with a spin-echo small-angle scattering setup, which will allow scientific measurements to be carried out at the request of users. The new option will significantly expand the experimental capabilities of the instrument.

### ***4.3. Current technical parameters of the instrument in thermal and cold modes of the moderator***

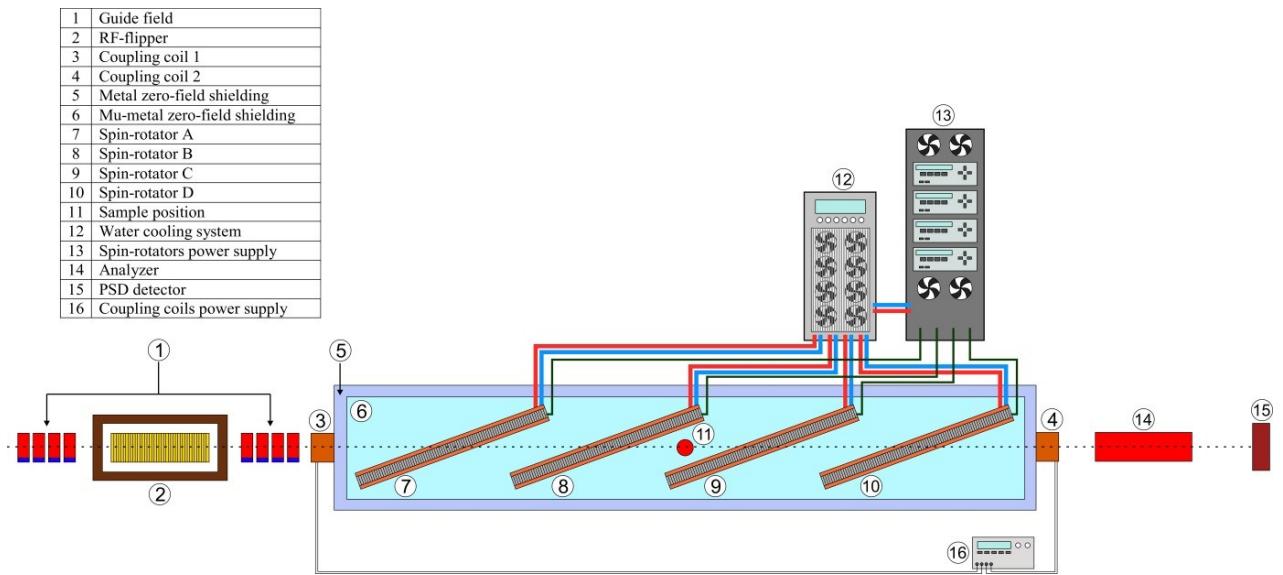
The implementation of the project of the new cold moderator, taking into account the geometry of the beamline, made it possible to increase the intensity of cold neutrons in reflectometric measurements (Fig. 28).



**Fig. 28.** Neutron spectra obtained during operation of the cryogenic (red dots) and thermal (black dots) moderators. The use of a mesitylene-based pelletized cryogenic moderator significantly increases the intensity of cold neutrons. The design of the cryogenic moderator provides visibility of both the thermal and cryogenic spectral regions. This results in an overlap of both spectral ranges. As a result, on beamline 9, an increase in intensity is achieved in the entire spectral range.

#### 4.4. Expected technical parameters after modernization of the instrument

The main project, within which further methodological development of the REFLEX instrument will be carried out, is the creation of a spin-echo small-angle diffractometer. Preliminary calculations and simulation of the future setup, taking into account the actual parameters of the neutron beam on beamline 9 made it possible to estimate the parameters of the future instrument.



**Fig. 29.** Schematic diagram of the spin-echo small-angle diffractometer on beamline 9 of the IBR-2 reactor. The spin-echo scheme is implemented on the basis of spin-rotators (7-10) with a magnetic field inside, linearly increasing along their axes with a time gradient  $\dot{B}$ . The axes of spin-rotators are inclined to the beamline axis to increase sensitivity to the neutron scattering angle.

***Expected technical parameters of the spin-echo small-angle diffractometer***

<b>Wavelength range, Å</b>	1.2 - 6
<b>Spin-echo wavelength range, Å</b>	100 - 15000
<b>Magnetic field gradient, Gauss/s</b>	$3.75 * 10^5$
<b>Pulse frequency, Hz</b>	200
<b>Coil tilt angle, deg</b>	10 - 15
<b>Spin-rotator coil dimensions, mm×mm×mm</b>	350×80×40
<b>Distance between spin-rotators, m</b>	1

***4.5. Relevance of the instrument development for the concept of a suite of spectrometers within the project of a new neutron source at JINR***

The REFLEX instrument is a convenient setup for testing new methods of neutron scattering, which can be implemented at the future neutron source at JINR in the coming years. In this regard, the development of the REFLEX instrument will certainly contribute to the methodological and scientific support of the instrument development regarding the concept of the new source.

**5. Expected scientific results**

The spin-echo small-angle diffractometer operating in the time-of-flight mode will be the first instrument of this kind in Russia. The evaluation of the parameters of the future setup allows us to state that it can significantly expand the capabilities of the existing and future conventional small-angle instruments at the IBR-2 pulsed reactor.

The experience gained in the development of this technique can be applied to both the existing and future pulsed neutron sources.

**6. Requested resources, costs and time frames of instrument modernization**

The terms and cost of modernization of the REFLEX reflectometer are listed in **Table 11 (Section 4)**.

<b>№</b>	<b>Description of activities</b>	2026	2027	2028	2029	2030
1	Polarization analyzer, m = 5					
2	Background shielding for the neutron guide					
3	Mechanical chopper					
4	Automation system					
5	Magnetic system for the sample					
6	Cryogenic system for the sample					
7	Fan polarization analyzer					
8	Spin-echo diffractometer					

## References

1. Mezei F, Z. Phyz. Neutron spin echo: A new concept in polarized thermal neutron techniques. *Z. Physik* 255 (1972) 146.
2. Pynn R, Neutron Spin Echo, *Lecture Notes in Physics*. V 128, pp. 159±177. Berlin: Springer.
3. Rekveldt M Th, Neutron reflectometry and SANS by neutron spin echo. *Physica B* 234–236 (1997) 1135-1137.
4. Golub R., Gähler R. A neutron resonance spin echo spectrometer for quasi-elastic and inelastic scattering. *Phys. Letters A*, (1987) 123(1), 43–48.
5. P. Schleger, B. Alefeld, J.F. Barthelemy, G. Ehlers, B. Farago, P. Giraud, C. Hayes, A. Kollmar, C. Lartigue, F. Mezei, D. Richter, The long wavelength neutron spin-echo spectrometer IN5 at the Institute Laue-Langevin. *Physica B* 241-243(1998) 164-165.
6. W.H. Kraan, S.V. Grigoriev, M.Th.Rekveldt, H.Fredrikze, C.F.de Vroege, J.Plomp, Test of adiabatic spin flippers for application at pulsed neutron sources. *NIM A*, Volume 510, Issue 3, 11 September 2003, Pages 334-345.
7. M.Th. Rekveldt, J. Plomp, W.G. Bouwman, W.H. Kraan, S.V. Grigoriev and M. Blaauw, Spin-echo small angle neutron scattering in Delft. *Review of Scientific Instruments* 76 033901 (2005).
8. Dalglish R M, Langridge S, Plomp J, V.O. de Haan, A.A. van Well, Offspec, the ISIS spin-echo reflectometer. *Physica B* 406 (2011) 2346–2349.
9. Ioffe A. A new neutron spin-echo technique with time-gradient magnetic fields. *NIM A* 586 (2008) 31–35.
10. M.V. Avdeev, V.I.Bodnarchuk, V.V.Lauter-Pasyuk, H.Lauter, V.L.Aksenov, S.P.Yaradaikin, V.A.Ulyanov, V.A.Trounov, S.I.Kalinin, Project of the new multifunctional reflectometer GRAINS with horizontal sample plane at the IBR-2M pulsed reactor in Dubna. *J. Phys.: Conf. Ser.* 251 (2010) 012060.
11. M.V. Avdeev, V.I.Bodnarchuk, V.I.Petrenko, I.V.Gapon, O.V.Tomchuk, A.V.Nagorny, V.A.Ulyanov, L.A.Bulavin, V.L.Aksenov. Neutron time-of-flight reflectometer GRAINS with horizontal sample plane at the IBR-2 reactor: possibilities and prospects. *Crystallography Reports* 62(6) (2017) 1002-1008
12. A.B. Rubtsov, N.F. Miron, V.A. Somenkov, V.I. Bodnarchuk, S.P. Yaradaykin, A neutron reflectometer on thermal neutrons with variable wavelength. *Journal of Surface Investigation: X-ray, Synchrotron and Neutron Techniques* 5 (2011) 105-108
13. [www.helmholtz-berlin.de/vitess](http://www.helmholtz-berlin.de/vitess).

## REMUR — Reflectometer with Polarized Neutrons

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PNPI NRC KI (Gatchina)  
IMP RAS (Nizhny Novgorod)  
KFU, KIMP (Kazan)  
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TPU (Tomsk)  
CSNS (Dongguan, China)

### 1. Abstract

The REMUR time-of-flight neutron reflectometer with a vertical sample plane is located on beamline 8 of the IBR-2 reactor. The instrument is designed to study solid-state heterostructures by measuring the neutron reflection coefficient, the diffuse scattering intensity of a thermal neutron beam, and detecting secondary radiation (gamma-rays, charged particles, fission fragments, etc.). The development of the REMUR reflectometer involves a further extension of the range of the instrument possibilities and is aimed at improving the scattered neutron detection system, enhancing the luminosity, suppressing the background from fast neutrons and gamma-radiation, increasing the polarizing efficiency, and developing sample environment systems, in particular, low-temperature equipment and a mode with an oscillating magnetic field.

### 2. Scientific program, relevance and comparison with the world level

The REMUR instrument is a time-of-flight polarized neutron reflectometer with vertical geometry, located on beamline 8 of the IBR-2 pulsed reactor operating at a repetition rate of 5 Hz. The reflectometer is designed to study the magnetism of thin films. In particular, one of the important tasks is to study proximity effects in low-dimensional ferromagnet/superconductor structures. Another area of research is the development of the method: the possibility of detecting secondary radiation, neutron channeling in a resonator structure, etc. Polarized neutron reflectometry is based on the reflection of a polarized neutron beam from a layered structure. The dependence of the reflection coefficient on the neutron wavelength can be used to determine such structural parameters as the thickness of the layers and the density distribution of matter within the structure. If a magnetic moment exists in the structure, the reflection coefficients differ for different input/output neutron polarizations, allowing us to determine the depth distribution of the magnetic moment, including that directed non-collinearly to the external magnetic field. Neutron scattering in grazing geometry also indicates the presence of inhomogeneities in the structure, which can be detected over a wide range of characteristic sizes, from nanometers to hundreds of microns. Such inhomogeneities include roughness at interfaces, clusters, magnetic domains, superconducting vortices, and so on.

The interaction of various types of order parameters gives rise to unusual phenomena that underlie the development of nano- and microelectronics. In this regard, research into this interaction is important from both fundamental and applied points of view. A number of such phenomena are the result of the interaction between superconducting pairs and magnetization,

which occurs in **ferromagnetic-superconducting structures**. In bilayers, trilayers, and periodic structures, such unusual phenomena as triplet superconductivity, cryptoferromagnetism, the inverse proximity effect, the spontaneous Meissner effect, the paramagnetism of the Meissner state, etc. have been predicted. It should be noted that theoretical predictions are not fully realized in practice due to inhomogeneities in heterostructures under study. In this regard, it is necessary to conduct not only experimental but also theoretical research. Of current interest are heterostructures with magnetic layers having a more complex, specifically helical, magnetic structure. In this case, superconductivity in magnetic layers can occur at higher temperatures.

Experiments with polarized neutrons are rare, however, they allow us to discover phenomena by probing structure at the nanoscale. Recent advances in neutron reflectometry, involving the **detection of secondary radiation**, are already making it possible to study the distribution of elements in magnetic structures. This will allow us, for example, to investigate the nature of Meissner paramagnetism, as well as to determine the degree of polarization of superconducting pairs induced by ferromagnets.

In addition, new research areas that are being developed using the REMUR reflectometer include:

- study of the properties of magnetic and superconducting heterostructures in a high-frequency oscillating magnetic field;
- comparison of the properties of periodic and quasi-periodic Fibonacci systems;
- dynamics of light atoms in layered systems and their influence on structural, magnetic, and superconducting properties;
- study of heterostructures with actinide materials;
- study of type 1.5 superconductivity in layered systems.

The REMUR development project provides wide possibilities of modern neutron reflectometry in vertical geometry. It is primarily aimed at creating conditions for a comprehensive study of interfaces using neutron reflectometry, as well as at developing and testing methods for obtaining and analyzing both specular reflection and non-specular (diffuse) scattering and secondary radiation. The REMUR reflectometer is included in the current FLNP User Program, which ensures its accessibility to the broad scientific community for conducting cutting-edge interdisciplinary investigations into the structure and properties of materials.

### 3. Scientific and methodological groundwork laid in FLNP JINR

Over the past two decades, investigations conducted by the REMUR group have focused mainly on two research areas. The first one concerned research into condensed matter, in particular, the discovery and study of phenomena in ferromagnetic-superconducting structures [1-25]. Structures that differ in the composition of layers and elements were studied. The main results on the effect of superconductivity on magnetic structures, which are characterized by a linear scale from fractions of a nanometer to hundreds of angstroms, are as follows:

- Suppression of magnetization by superconductivity due to the isotropic distribution of the directions of domain moments (cryptoferromagnetism in the domain structure) and the anticollinear ordering of moments of the cluster system (cryptoferromagnetism in the cluster system);
- Temperature reversibility of the inhomogeneous magnetic state;
- Correlation in the behavior of magnetic structures at different spatial scales, in particular, the interaction of the cluster system with the domain structure;
- Two-stage transformation of the domain structure;
- Reorientation of magnetic moments in the system during a zero-field superconducting transition to a spontaneous Meissner phase;

- Diamagnetism of a periodic ferromagnetic-superconducting structure, coherent superconducting length in gadolinium (4 nm);
- Meissner paramagnetism of ferromagnetic-superconducting structures.

The second line of research was related to the development of neutron reflectometry methods. Scattering methods, in particular GIND and GISANS, were further developed. Reflectometry with the detection of secondary radiation (charged particles, gamma-rays and spin-flipped neutrons) was implemented [26-30].

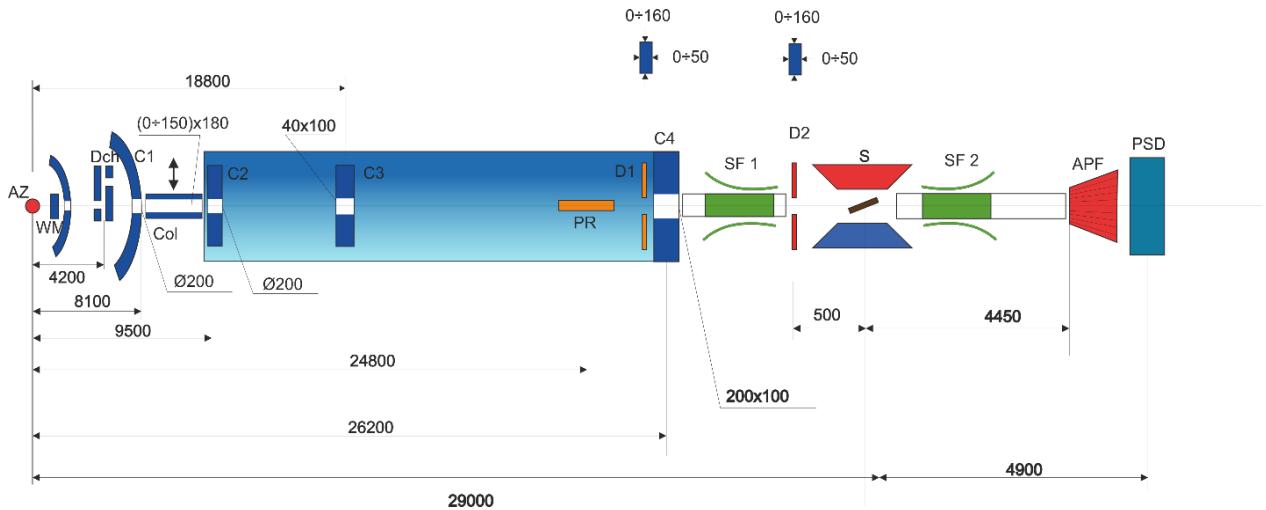
#### 4. Current status of the instrument and proposals for its modernization

##### 4.1. Information on the current status of the instrument

The REMUR reflectometer is located on beamline 8 of the IBR-2 pulsed reactor. The IBR-2 reactor operates with a pulse repetition rate of 5 Hz, and the neutron pulse duration with a 5-cm-thick water moderator is 340  $\mu$ s. The thermal neutron flux in a pulse pulse is  $10^{16}$  n/cm<sup>2</sup>/s at an average reactor power of 2 MW. The sample placement location is at a distance of 29 m from the IBR-2 neutron moderator. The position-sensitive neutron detector is placed at a distance of 5.03 m from the sample position. The neutron wavelength is determined by the neutron time of flight from the moderator surface to the detector. The standard deviation of the wavelength of neutrons registered by the detector is 0.02 Å at a distance of 34 m from the moderator. The reflectometer makes it possible to conduct polarization analysis of non-specular neutron reflection from layered magnetic structures in the wavelength range of 1-10 Å and scattering angles of 1-30 mrad. The polarized neutron channel includes a polarizer, incoming and outgoing neutron spin-flippers, a polarization analyzer, and a neutron detector. The structure under study is located between the spin-flippers. The beam intensity at the sample position is determined by its divergence, which is regulated by a cadmium diaphragm located at the exit of the polarizer. The maximum solid angle of visibility of the polarizer from the sample position is  $\Omega_{ps}=2.0 \cdot 10^{-5}$  sr. The polarized neutron intensity at the sample, with a beam divergence in the horizontal neutron reflection plane of  $2\Delta\theta = 0.7$  mrad is  $2.0 \cdot 10^4$  cm<sup>-2</sup>s<sup>-1</sup>.

The operation of the reflectometer is possible in various moderator modes: thermal water moderator/cryogenic moderator. The low-temperature moderator affects the neutron distribution so that in the region of short neutron wavelengths ( $\sim 1.5$  Å), the intensity decreases by a factor of  $\sim 3$ , while in the region of  $\sim 4-10$  Å, the intensity increases by a factor of  $\sim 10$ . The need for one or another moderator is determined by the requirements of a specific experiment.

Figure 30 shows the functional diagram of the spectrometer. Fast neutrons generated in the core (AZ) of the IBR-2 pulsed reactor are thermalized in the water moderator (WM) surrounding the reactor core. The double-disk chopper (Ch) passes the main pulse of thermal neutrons and cuts off satellite pulses that occur during reactor reactivity modulation. Next, the neutron beam is formed by a complex system of collimators and a polarizer. Variable cadmium diaphragms (D1 and D2) determine the divergence of the neutron beam incident on the sample (S) placed between the poles of the electromagnet (EM). The sample plane is vertical, and neutron scattering occurs horizontally. Spin flippers (F1 and F2) change the direction of polarization of the neutron beam relative to the guiding magnetic field. The fan analyzer (A) with vertical supermirrors is used to analyze the polarization of the scattered neutron beam. Neutrons passing through the analyzer are detected by a gaseous <sup>3</sup>He two-dimensional position-sensitive detector.



**Fig. 30.** Schematic diagram of the REMUR reflectometer. AZ – reactor core, WM – water moderator, Dch – chopper, Col, C1(2,3,4) – collimators, D1(2) – diaphragms, PR – polarizer, SF1(2) – spin-flipper, S – sample position, APF – polarization analyzer, PSD – position-sensitive detector.

During its operation within the User Program, the REMUR reflectometer has proven to be an effective instrument for studying superconducting and magnetic heterostructures. This efficiency is due to the relatively high intensity of neutrons on the sample, the possibility of conducting a complete polarization analysis and effective detection of scattered neutrons. The minimum reflectivity value is  $10^{-5}$ . The REMUR reflectometer provides for additional options, including the possibility to detect secondary radiation.

#### 4.2. Detailed description of proposals for modernization of the instrument

The spectrometer was last upgraded in 2003. During this time, some of the equipment became obsolete, and its performance deteriorated. The modernization is aimed at improving the quality of neutron scattering experiments. The development of the REMUR spectrometer provides for further expansion of possibilities in terms of detecting scattered neutrons, increasing the luminosity and reducing the background level of fast neutrons and gamma-radiation, increasing the polarizing efficiency, and developing sample environment systems, in particular low-temperature equipment and a mode with an oscillating magnetic field.

##### 4.2.1. Development of a new polarization module and a system of collimators

The polarization efficiency of the polarizer has significantly decreased over approximately 18 years of operation. Furthermore, after starting up the cold moderator and replacing the two-disk chopper with a drum chopper, the beam intensity at  $\lambda > 7$  Å will increase by an order of magnitude. It is proposed to develop a polarizer consisting of two mirrors with sequential reflection. Due to double reflection, this solution will make it possible to increase the polarization of the neutron beam for longer wavelengths. Double reflection will also allow better separation of the working and direct beams, thereby reducing the background of fast neutrons and  $\gamma$ -radiation. The dimensions of the mirrors are  $800 \times 100$  mm $^2$  and  $1300 \times 110$  mm $^2$ . The mirrors are assembled from sections 250-300 mm long. For mounting and aligning the mirrors, it is proposed to use the existing alignment platforms in the vacuum tubes without any modifications.

It is also proposed to develop a new system of collimators, which will make it possible to reduce not only the background of fast neutrons, but also the  $\gamma$ -radiation background. For this purpose, the collimator systems are proposed to be installed at flight paths of 10.2 m, 15.6 m, and 20.6 m, with a variable collimator placed at 9.8 m or 16.7 m. It is proposed to install a collimator at the exit from the vacuum tube in front of diaphragm No. 2, and after aligning the mirrors, install an additional collimator inside the tube. To further reduce the  $\gamma$ -radiation background, after each

collimator system it is necessary to install 100-mm-thick lead collimators with fixed windows, which are aligned across the beam.

#### **4.2.2. Development of a new two-drum neutron beam chopper**

The existing disk chopper has low transmission, which reduces the luminosity of the spectrometer. Therefore, it is necessary to replace the disk chopper with a drum chopper. This will increase the luminosity of the instrument and reduce  $\gamma$ -radiation background. It is proposed to replace the beam chopper with a two-drum chopper with a narrow window. The chopper will be based on a lead core with an outer layer of borated polyethylene. This replacement will reduce the background of fast neutrons and  $\gamma$ -rays, and increase the intensity of cold neutrons in the beam by approximately 15% by removing 20-25 mm of duralumin in the current construction.

#### **4.2.3. Development of new low-temperature equipment**

At present, the use of the  $\gamma$ -ray detection mode with the existing cryostat is difficult due to its large diameter, which complicates the positioning of the gamma-detector close to the structure under study. The development of a new cryostat (dilution refrigerator) will increase the luminosity of the mode by a factor of 20-30. The new cryostat will require the manufacture of a vector cryomagnet, which generates a magnetic field directed either in the plane of the structure or perpendicular to it. Also, technical specifications are being developed for a new closed-cycle cryostat with a cryomagnet and a  $^3\text{He}$  insert, as well as for a closed-cycle cryostat with an electromagnet for studying the properties of superconductors and magnetic structures under high-frequency dynamics conditions. This is important for the development of the concept of the suite of spectrometers in the framework of the project of the new neutron source at FLNP.

#### **4.2.4. Development of a scattered neutron detector**

When studying layered structures at a neutron reflectometer, specular neutron reflection is accompanied by scattering from roughness, clusters, and crystal structures. For example, in studies of ferromagnetic-superconducting heterostructures, a correlation in the behavior of structures of different scales was observed due to superconductivity. Therefore, two detectors are required on the spectrometer to detect scattering from clusters (linear size of 1-20 nm) and crystal lattices (interplanar spacing in the range of 0.2-1 nm), respectively.

#### **4.2.5. Modernization of the magnetic system**

An electromagnet is used for reflectometry measurements in an external magnetic field. Over time, the current decreased while the source voltage remained the same, which indicates an increase in the resistance of the coils, a result of short circuits. It is necessary to wind new coils with a square-section wire, using a compound between the turns for insulation.

#### **4.2.6. Development of a stand with oscillating magnetic fields**

A stand for conducting neutron reflectometry studies in oscillating magnetic fields has been developed. The field frequency is  $\sim 30$  MHz, and the amplitude is 1-10 Oe. The stand requires modernization, specifically the purchase of a controlled matching device, the development of a new housing, and the design of a Faraday cage for the sample.

**4.3. Main parameters of the REMUR reflectometer before and after modernization**

Parameters	Before modernization	After modernization
<b>Total flux at sample position</b>	$2 \cdot 10^5 \text{ n/(s}\cdot\text{cm}^2)$	$4 \cdot 10^5 \text{ n/(s}\cdot\text{cm}^2)$
<b>Wavelength range</b>	1 - 10 Å	1 - 15 Å
<b>Q-range</b>	$2 \cdot 10^{-3} - 1 \text{ \AA}^{-1}$	$1 \cdot 10^{-3} - 1 \text{ \AA}^{-1}$
<b>Size range of objects under study</b>	1 nm - 100 μm	1 Å - 100 μm
<b>Flux at 10 Å</b>		An increase by a factor of 20 due to a new chopper
<b>Polarization efficiency</b>	0.8 at 2 Å	An increase up to 0.95 at 2 Å
<b>Background count by gamma-ray detector</b>		A decrease by a factor of 5
<b>Detector system for neutrons</b>	Directly scattered neutron PSD	Directly scattered neutron PSD, backscattering PSD
<b>Distance between gamma-ray detector and structure under study in cryostat</b>	18 cm	3 cm
<b>Temperature range</b>	1.5 - 300 K	1.5 - 300 K
<b>Q-resolution</b>	$2 \cdot 10^{-3}$ at 10 Å	$1.3 \cdot 10^{-3}$ at 15 Å
<b>Magnetic field range</b>	20 Oe - 3 T	20 Oe - 3 T

**4.4. Relevance of the instrument development for the concept of a suite of spectrometers within the project of a new neutron source at JINR**

The development of reflectometry at IBR-2 towards the study of magnetic and superconducting heterostructures is fully in line with current global trends. Nearly all neutron sources, both pulsed and steady-state, are equipped with neutron reflectometers with a vertical sample plane, with a focus on their use in the study of solid heterostructures. At the same time, two-thirds of the world's reflectometers with vertical sample geometry are based on the time-of-flight technique.

**5. Expected scientific results, comparison with the world level**

Data will be obtained on new phenomena caused by the interaction of various order parameters, in particular, the magnetization and density of superconducting pairs. Plans include studying the behavior of helical structures in layered structures with rare earth elements in a temperature-magnetic field diagram.

The studies will be focused on inhomogeneities in bulk and thin-film magnetic superconductors, layered superconducting (S)/ferromagnetic (F) structures, and individual magnetic and superconducting layers. They are related to different areas, including the structure of metal clusters, super-spin glass state, superparamagnetism, the structure and ordering of Abrikosov vortices, vortices at the BKT-transition near  $T_c$ , the vortex state in type-1.5 superconductivity, magnetic domains, cryptoferromagnetism, etc. Correlation lengths of induced superconductivity will be determined for a new type of magnets, including noncollinear magnets and magnetic superconductors. Using neutron methods, non-equilibrium phenomena will be investigated, including the relaxation of the magnetic and superconducting states under pulsed excitation by a magnetic field. The study aims at investigating the coexistence of superconductivity and non-collinear magnetism in heterostructures with rare-earth antiferromagnets exhibiting

helical magnetization. The practical potential of this research is related to the development of layered structures with novel properties, used, in particular, in superconducting nano- and microelectronics and spintronics.

The coexistence of superconductivity and noncollinear magnetism in heterostructures with rare-earth antiferromagnets exhibiting helical magnetization will be the subject of detailed studies. For the first time, it is proposed to investigate the Josephson  $\pi$ -junction and spin injection in systems with noncollinear magnets. A wide range of phenomena will be studied in these systems, including non-equilibrium processes, the behavior of inhomogeneities (clusters, domains, vortices), electromagnetic proximity effects, and the determination of characteristic correlation lengths. For the first time, it is also projected to study superconductivity and magnetism in quasi-periodic Fibonacci structures. The plans include the exploration of submicron hybrid nanostructures based on superconductors, normal metals, ferromagnets, and other modern strongly correlated and topological electron systems. Superconducting nanostructures with quantum phase slip and submicron phase inverters (Josephson  $\pi$ -junctions) in planar geometry will be fabricated and studied, as well as non-equilibrium and coherent phenomena in hybrid bilayers and Josephson nanostructures. A comprehensive experimental approach will be used to characterize the layered structures. Nuclear physics methods will be applied, including polarized neutron reflectometry, synchrotron radiation reflection, charged particle scattering (Rutherford backscattering), resonance methods (NMR, Mössbauer spectroscopy, EPR), and complementary methods (SQUID magnetometry, transport resistivity measurements, atomic force microscopy, and structure decoration techniques).

The influence of superconductivity on helical magnetic ordering will be studied in details. For the first time, the correlation length of superconductivity in a helical magnet will be measured. This type of phenomena is characterized by two correlation lengths, including the correlation length of superconductivity in the magnet and the helicoid period. The determination of their values is crucial for understanding a correlation between superconducting and magnetic properties. Substrates with different crystallographic orientations will be used for structure growth, resulting in different directions of the magnetic helicoid axes. X-ray structural analysis will determine the crystallographic orientation of the Dy and Ho layers, which will allow us to determine the direction of the magnetic helicoid axis. This will also make it possible to determine the correlation between the magnetic and superconducting properties of the structures depending on the direction of the helicoid axis. By measuring the magnetic moment of the structures as a function of temperature and applied magnetic field in various cooling modes, one can determine phase transition temperatures and detect cluster formation. Using polarized neutron reflectometry, the magnetic profile of the structure will be determined in the direction perpendicular to its plane. This will include determining the helicoid period in superlattices over a wide range of temperatures (1.5–300 K) and magnetic fields (0–15 kOe). The influence of superconductivity on the magnetic properties of the structure, in particular, on the helicoid period, will also be determined.

The properties of various strongly correlated phenomena in one-dimensional layered quasicrystals are of current interest. Key stages of the planned research include calculations of the optical properties of quasiperiodic layered systems for neutron and X-ray optics, experimental studies of non-magnetic and non-superconducting structures for optical properties, investigation of the superconducting properties of layered quasicrystals, investigation of the magnetic properties of layered quasicrystals, investigation of the coexistence of superconductivity and magnetism in layered quasicrystals. For the first time, the neutron-optical and X-ray-optical properties of quasiperiodic layered Fibonacci structures will be studied. The expected results are relevant for the development of optics. Furthermore, the project proposes to investigate for the first time the properties of superconducting and magnetic properties in layered quasiperiodic systems, as well as the coexistence of superconductivity and magnetism.

The project involves calculating the optimal parameters of periodic and quasi-periodic niobium/palladium systems, including layer thicknesses, the number of pairs in the superlattice,

substrate and buffer types, a top layer for protecting the structure and implementing standing neutron waves in the structure, and the selection of a support layer for neutron reflectometry. A series of experimental samples will be fabricated using magnetron sputtering. The project will also explore the features of the dynamics of protium and deuterium in a low-dimensional layered Fibonacci system.

The project will result in the development of a new method for studying layered structures by neutrons. This method will make it possible to determine the spatial distribution of isotopes near layer interfaces, thereby linking physical properties to structure. In particular, by determining the correlation between the induction profile and the elemental profiles in the ferromagnetic and superconducting layers during the superconducting transition in the bilayer, it could be possible to propose explanations for the inverse and direct proximity effects.

After the upgrade, the REMUR reflectometer is expected to be among the advanced reflectometers with polarized neutrons in the world. It will also be the only reflectometer equipped with additional modes such as secondary radiation detection, a stand with an oscillating magnetic field, and a low-temperature cryostat with a vector magnet. Key parameters to be improved include luminosity, background conditions, and polarization efficiency.

## 6. Requested resources, costs and time frames of instrument modernization

The terms and cost of upgrading the REMUR reflectometer are shown in **Table 12 (Section 4)**.

### Plan-scheme of activities for the modernization of the REMUR reflectometer

№	Description of activities	2025	2026	2027	2028	2029
1.	Drum-type neutron chopper					
2.	Closed-cycle helium cryostat					
3.	Pumping system					
4.	1. Ring forward-scattering PSD					
	2. Backscattering detector					
5.	Neutron polarizer and system of collimators					
6.	Modernization of electromagnet					
7.	Development of a stand with an oscillating magnetic field					

### References

1. V.L. Aksenov, K.N. Jernenkov, Yu.N. Khaidukov, Yu.V. Nikitenko, A.V. Petrenko, V.V. Proglyado, G. Andersson, R. Wappling, Interplay between superconductivity and ferromagnetism in Fe/V multilayered structure studied by polarized neutron reflectometry, *Physica B* 356 (2005) 9-13.
2. V.L. Aksenov, Yu.V. Nikitenko, A.V. Petrenko, V.M. Uzdin, Yu.N. Khaidukov, H. Zabel, Features of the magnetic state of the layered Fe-V nanostructure of the superconductor-ferromagnet type. *Cryst. Rep.* 2007, 52, 381-386.
3. V.L. Aksenov, Yu.V. Nikitenko, Yu.N. Khaidukov, S.N. Vdovichev, M.M. Borisov, A.N. Morkovin, E.Kh. Mukhamedzhanov, Coexistence of superconductivity and ferromagnetism in the Nb(500 Å)/Fe(39 Å)/[Si(34 Å)/Mo(34 Å)]<sub>40</sub>/Si nanostructure. *J. Surf. Invest.* 3 495, 2009.

4. V.L. Aksenov, Yu.N. Khaidukov, Yu.V. Nikitenko. Peculiarities of magnetic states in Ferromagnet/Superconductor heterostructures due to the proximity effects. *Journal of Physics: Conference Series* 211, 012022 (2010).
5. Khaydukov Yu.N., Aksenov V.L., Nikitenko Yu.V. *et al.* Proximity Effects in S/F Single Bilayer, *J. Supercond. Novel Magnetic Magn.*, Vol. 24 (2011), p. 961-964;
6. Yu.N. Khaidukov, N.S. Perov, M.M. Borisov, E.Kh. Mukhamedzhanov, A. Csik, K.N. Zhernenkov, Yu.V. Nikitenko, V.L. Aksenov, Structural and magnetic properties of the periodic  $10 \times \text{Fe}(5\text{nm})/\text{V}(5\text{nm})$  and  $20 \times \text{Fe}(3\text{nm})/\text{V}(3\text{nm})$  systems, *Solid State Phenomena* Vol. 190 (2012) pp. 396-400.
7. Yu.N. Khaidukov, D. Nagy, J.-H. Kim, T. Keller, A. Ruhm, Yu.V. Nikitenko, K.N. Zhernenkov, J. Stahn, L.F. Kiss, A. Csik, L. Bottyan, V.L. Aksenov, On the Feasibility to Study Inverse Proximity Effect in a Single S/F Bilayer by Polarized Neutron Reflectometry, *JETP Letters*, 2013, Vol. 98, No.2, pp. 107-110.
8. V.D. Zhaketov, Yu.V. Nikitenko, F. Radu, A.V. Petrenko, A. Chik, M.M. Borisov, E.Kh. Mukhamedzhanov, V.L. Aksyonov, Magnetism in structures with ferromagnetic and superconducting layers. *JETP* (2017) 124, 114–130.
9. V.D. Zhaketov, Yu.V. Nikitenko, A.V. Petrenko, F. Radu, A. Chik, V.L. Aksenov, Relaxation of the magnetic state of a ferromagnetic–superconducting layered structure. *JETP* (2017) 125, 480–494.
10. V.D. Zhaketov, Yu.V. Nikitenko, A.V. Petrenko, S.N. Vdovichev, A.V. Churakov, A. Chik, Reflexivity and Correlation of Magnetic States of Nanostructures in the  $\text{Nb}(70 \text{ nm})/\text{Ni}_{0.65}\text{Cu}_{0.35}(6.5 \text{ nm})/\text{Si}$  Ferromagnet–Superconductor Heterostructure. *JETP* (2018) 127, 508–515.
11. V.D. Zhaketov, Yu.V. Nikitenko, Yu.N. Khaidukov, O.V. Scriabin, A. Chik, M.M. Borisov, E.Kh. Mukhamedzhanov, S.N. Vdovichev, E.I. Litvinenko, A.V. Petrenko, A.V. Churakov, Magnetic and Superconducting Properties of the Heterogeneous Layered Structures  $\text{V}/\text{Fe}_{0.7}\text{V}_{0.3}/\text{V}/\text{Fe}_{0.7}\text{V}_{0.3}/\text{Nb}$  and  $\text{Nb}/\text{Ni}_{0.65(0.81)}\text{Cu}_{0.35(0.19)}$ . *JETP* (2019) 129, 258-276.
12. Yu.N. Khaydukov, A.S. Vasenko, E.A. Kravtsov, V.V. Progliado, V.D. Zhaketov, A. Csik, Yu.V. Nikitenko, A.V. Petrenko, T. Keller, A.A. Golubov, M.Yu. Kupriyanov, V.V. Ustinov, V.L. Aksenov, B. Keimer. Magnetic and superconducting phase diagram of Nb/Gd/Nb trilayers . *Physical review B* 97, 144511 (2018)
13. Yu.N. Khaydukov, E.A. Kravtsov, V.D. Zhaketov, V.V. Progliado, G.Kim, Yu.V. Nikitenko, T. Keller, V.V. Ustinov, V.L. Aksenov, B. Keimer. Magnetic proximity effect in Nb/Gd superlattices seen by neutron reflectometry // *Physical Review B*, vol.99, No.14, 2019.
14. Yu. Khaydukov, V. Zhaketov, Yu. Nikitenko, V. Aksenov *et al.*, 2019 *J. Phys.: Conf. Ser.* 1389 012060, Neutron reflectometry studies of Gd/Nb and Cu30Ni70/Nb superlattices.
15. A.A. Gurgel, F.G. Queiroz, E.S.N. Silva *et al.* Thermal hysteresis in Ho thin films– experimental results and modeling. // *Journal of Magnetism and Magnetic Materials*, Vol. 610, pp. 172579, 2024.
16. Zaky A. Zaky, V.D. Zhaketov, Mohammed Sallah, Arafa H. Aly. Using periodic multilayers of ferromagnetic and paramagnetic layers as neutron filter: Simulation study. // *Plasmonics*, pages 1-11, 2024.
17. A.V. Altynov, A.P. Buzdavin, V.I. Bodnarchuk *et al.* Development of a Vector Magnet Based on High-Temperature Superconductors for Working with Polarized Neutrons // *Journal of Surface Investigation: X-ray, Synchrotron and Neutron Techniques*, Vol. 18, No. 4, pp. 900-907, 2024.
18. D.I. Devyaterikov, E.A. Kravtsova, V.V. Proglyado *et al.* On the Prospects of Using Polarized-Neutron Reflectometry to Study Helimagnetism in Rare-Earth Thin Films and Nanostructures on the DARIA Compact Neutron Source // *Journal of Surface Investigation: X-ray, Synchrotron and Neutron Techniques*, Vol. 17, No. 4, pp. 832-836, 2023.
19. V.D. Zhaketov, D.I. Devyaterikov, M.M. Avdeev *et al.* Structural Properties of Nb/Dy and Nb/Ho Superlattices. // *Fizika Tverdogo Tela*, 2023, Vol. 65, No. 7, pp. 1123-1128.
20. E.S. Nikova, Yu.A. Salamatov, E.A. Kravtsov *et al.* Gd-Reference-Layer Method in Polarized Neutron Reflectometry. // *Journal of Surface Investigation: X-ray, Synchrotron and Neutron Techniques*, Vol. 16, No. 6, pp. 939-942, 2022.
21. Yu.V. Nikitenko, V.D. Zhaketov. Magnetism in ferromagnetic-superconducting layered structures. // *Physics of Particles and Nuclei*, Vol. 53, No. 6, pp. 1089-1125, 2022.
22. D.I. Devyaterikov, E.A. Kravtsov, V.V. Proglyado *et al.* Study of Helimagnetism in Dy/Ho Superlattice by Neutron Reflectometry. // *Journal of Surface Investigation: X-ray, Synchrotron and Neutron Techniques*, Vol. 16, No. 5, pp. 839-842, 2022.

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23. D.I. Devyaterikov, V.V. Proglyado, V.D. Zhaketov et al. Influence of Dimensional Effects on the Curie Temperature of Dy and Ho Thin Films // Physics of Metals and Metallography, Vol. 122, pp. 465-471, 2021.
24. D.I. Devyaterikov, E.A. Kravtsov, V.V. Proglyado et al. Investigation of helimagnetism in Dy and Ho thin films by neutron reflectometry // Journal of Surface Investigation: X-ray, Synchrotron and Neutron Techniques, Vol. 15, No. 3, pp. 542-548, 2021.
25. D.I. Devyaterikov, E.A. Kravtsov, V.V. Proglyado et al. Investigation of helimagnetism in Dy and Ho thin films by neutron reflectometry. // Journal of Surface Investigation: X-ray, Synchrotron and Neutron Techniques, Vol. 15, No. 3, pp. 542-548, 2021.
26. Yu.V. Nikitenko, A.V. Petrenko, N.A. Gundorin, Yu.M. Gledenov, V.L. Aksenov. Isotope-Identifying Neutron Reflectometry. Cryst. Rep. (2015), 60, 466-479.
27. V.D. Zhaketov, A.V. Petrenko, S.N. Vdovichev et al. Grazing-Incidence Neutron Spectrometer Detecting Neutrons and Charged Particles. // Journal of Surface Investigation: X-ray, Synchrotron and Neutron Techniques, vol.13, No. 3, pp. 478-487, 2019.
28. Yu. N. Khaydukov, V.D. Zhaketov, D.S. Korolkov et al. Gamma Spectroscopy of Nanometer Layers of Gadolinium in the Mode of Resonantly Enhanced Standing Neutron Waves. // Physics of Particles and Nuclei Letters, Vol. 21, No. 5, pp. 1065-1068, 2024.
29. V.L. Aksenov, V.D. Zhaketov, Yu. V. Nikitenko. Neutron Reflectometry with Detection of the Secondary Radiation: Particle-Wave Method of Determining the Nanoscale Isotope Density Distributions // Physics of Particles and Nuclei, Vol. 54, No. 4, pp. 756-775, 2023.
30. V.D. Zhaketov, K. Hramco, A.V. Petrenko et al. Polarized Neutron Reflectometer with the Recording of Neutrons and Gamma Quanta // Journal of Surface Investigation: X-ray, Synchrotron and Neutron Techniques, Vol.15, No.3, pp. 549-562, 2021.

## NRT — Neutron Radiography and Tomography Station

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### 1. Abstract

Neutron radiography is an imaging technique based on varying degrees of attenuation of a neutron beam passing through sample components with different chemical compositions, densities, and thicknesses. It is used to obtain information on the internal structure of the materials being studied with a spatial resolution on the micrometer scale. The functional elaboration of neutron radiography is neutron tomography. In this method, a three-dimensional reconstruction of the internal structure of the object under study is performed from a set of individual single-plane radiographic projections obtained for various angular positions of the sample relative to the direction of the neutron beam. In 2015, a specialized neutron radiography and tomography (NRT) station was put into operation at the IBR-2 high-flux pulsed reactor. The significant penetration depth of neutrons makes it possible to obtain fundamentally new information using neutron radiography and tomography techniques compared to X-ray methods. In particular, they are used to solve a wide range of interdisciplinary scientific problems, especially in archeology and paleontology, which is dictated by the uniqueness of the objects being studied, which often exist in a single copy and are of great value. In order to ensure reliable operation of the instrument and improve its efficiency, it is planned to modernize the detector system and develop an automated sample-changing system.

### 2. Scientific program, relevance and comparison with the world level

Neutron radiography allows one to obtain images of the internal structure of various objects. The resulting neutron image is a set of finite-size dots, or pixels, reflecting the degree of attenuation of the neutron beam intensity at a specific point on the object being studied. Neutron radiography and tomography is an applied non-destructive testing technique in modern technologies. The penetration of neutrons into the thickness of a material provides information about the spatial distribution of internal components, cracks and internal defects, as well as the locations and routes of corrosion penetration in industrial and engineering objects. The creation of the neutron radiography and tomography station at FLNP has enabled the development of a new applied research area related to the non-destructive analysis of the internal structure of three-dimensional objects, products, and materials, including paleontological objects of natural heritage, engineering products, objects of extraterrestrial origin—meteorites—and archaeological objects of cultural heritage. A comprehensive study of objects of cultural heritage, allowing us to peer

deep into the centuries and trace the formation and development of civilizations and ethnic groups, is one of the most important tasks of interdisciplinary research at the intersection of the natural sciences, archaeology, and other humanitarian sciences.

### **3. Scientific and methodological groundwork laid in FLNP JINR**

In recent years, there has been a global trend toward the widespread application of natural science methods, including nuclear physics, to the analysis of the internal structure and various characteristics of large objects [1-4]. This allows us to obtain the most comprehensive information on the chemical composition and structural features, the presence of internal defects and hidden structures, the phase composition, and the spatial distribution of components using non-destructive research techniques. For these applied studies, new approaches to the analysis of 3D neutron tomography data [5-9] have been developed, and certain structural markers of ancient technological processes of ceramic production [10, 11], coin minting [12], or metal casting methods [13-15] have been identified.

The method of neutron radiography and tomography belongs to the family of methods used for non-destructive testing of technological and engineering objects. High radiographic contrast between zirconium and steel made it possible to investigate the internal volume of a bimetallic adapter for reactor technologies [1]. An interesting application of neutron radiography is the study of the melting kinetics of a mixture of ice and granular quartz. It was found that the melting point of the mixture depends on the size of the quartz granules [5]. The method of neutron radiography and tomography is successfully used to study cement materials. Specific types of building materials used in the construction of specialized repositories or disposal facilities for various types of radioactive waste were investigated. Particular attention was paid to the study of defects in cement and concrete structures, such as cracks and cavities, which could lead to radionuclide leakage from the repositories into the environment.

Neutron tomography was used to reconstruct models of the internal volume of objects of extraterrestrial origin – the Seymchan [6] and Marjalahti meteorites. The features of the interaction of neutrons with matter made it possible to detect and separate the internal mineral components of these meteorites, obtain a three-dimensional spatial distribution of nickel in the metallic component of these pallasite meteorites, construct distributions of occupied volumes and average sizes of the internal constituent minerals, and determine their morphological features.

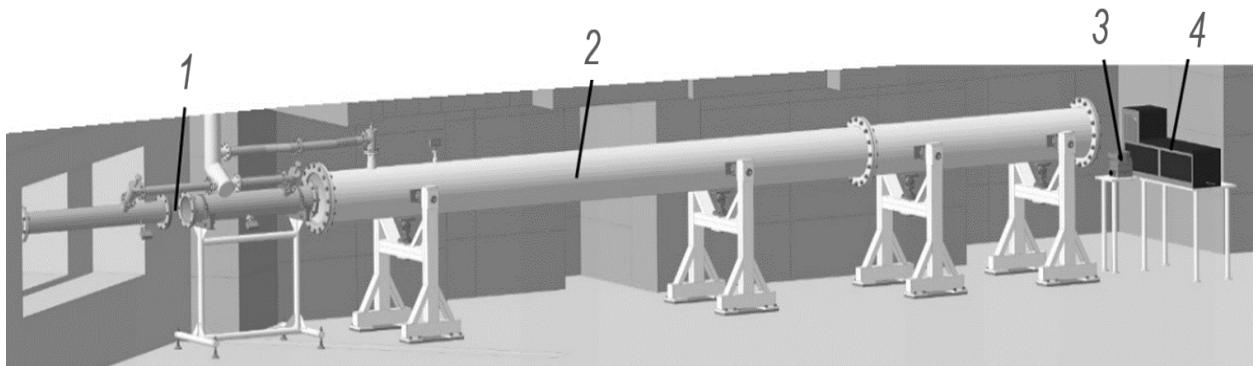
The new possibilities offered by neutron non-destructive analysis of applied materials have proven to be in high demand among the scientific community of a number of JINR Member States, and have played a key role in solving important scientific problems that were difficult to resolve using other alternative approaches.

### **4. Current status of the instrument and proposals for its modernization**

#### ***4.1. Information about the current status of the instrument***

The schematic of the main components of the NRT experimental instrument for neutron radiography and tomography on beamline 14 of the IBR-2 pulsed high-flux reactor is shown in Fig. 31. The neutron beam is formed using a collimator system consisting of four ring-shaped cylindrical inserts made of borated polyethylene, alternating with additional steel rings for structural rigidity. The internal diameters of the ring collimators increase from 5 cm (at the entrance) to 23.7 cm (at the exit of the neutron beam from the collimator system). The collimator system provides space for installing single-crystal filters: sapphire or bismuth. These filters are designed to suppress fast neutrons and gamma-radiation in the spectrum of the neutron beam of the experimental station. The integrated thermal neutron flux at the sample location was measured using the gold-foil activation method and is  $\Phi \sim 5.5(2) \times 10^6 \text{ n/cm}^2/\text{s}$ . The length  $L$  is 10 m, and the diameter of the collimator entrance aperture  $D$  is 5 cm, which corresponds to a value of  $L/D = 200$ . The design of the collimator system allows for a reduction in the diameter of the entrance aperture

to 0.5 cm, which makes it possible to achieve a value of  $L/D = 2000$ . The collimator system is placed in an evacuated tube to reduce intensity losses due to neutron scattering in the air.

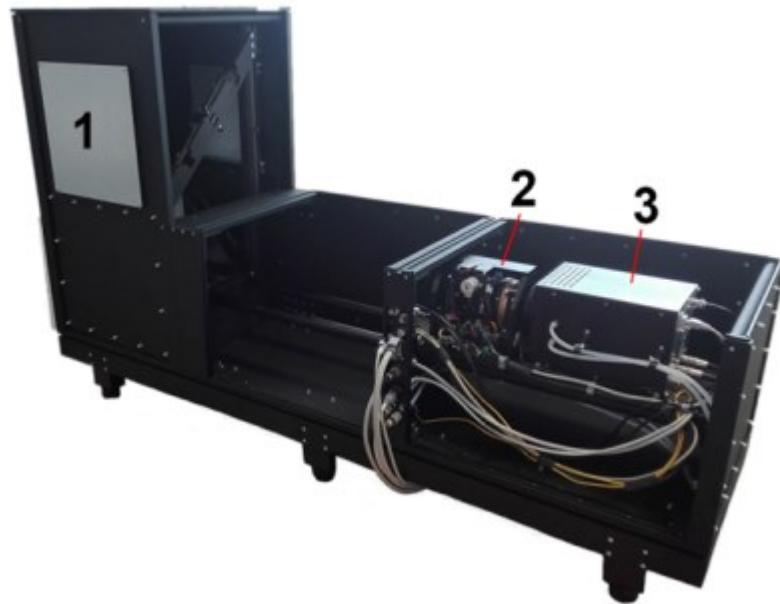


**Fig. 31.** Schematic diagram of the main units of the NRT instrument at the IBR-2 reactor: 1 – location of neutron beam filters; 2 – evacuated tube of the collimator system; 3 – system of rotary and tilt goniometers for positioning the sample under study; 4 – detector system.

A photograph of the detector system based on a CCD video camera, used in the experimental station for neutron radiography and tomography, is shown in Fig. 32. The conversion of neutrons into light photons, recorded by the HAMAMATSU CCD-camera is performed using a 0.2-mm thick  ${}^6\text{LiF}/\text{ZnS}(\text{Ag})$  scintillator plate manufactured by RC TRITEC Ltd (Switzerland). The CCD matrix has a resolution of  $2048 \times 2048$  pixels, with each pixel measuring  $12 \times 12 \mu\text{m}$ . To suppress the background of scattered neutrons from the scintillator and structural elements of the detector, a two-mirror scheme is used in the detector module. The light image is focused onto a CCD-matrix by a TAMRON objective lens with a variable focal length of 70-300 mm. All detector optical systems are housed in a light-protective casing. The size of one pixel of a neutron radiographic image is  $52 \times 52 \mu\text{m}$ , and the spatial resolution of images recorded for a neutron beam size of  $20 \times 20 \text{ cm}$  is  $134 \mu\text{m}$ .

Tomography experiments are performed using the system of HUBER goniometers with a minimum rotation angle of down to  $0.02^\circ$  and a remote control system. The high neutron flux at the sample makes it possible to reduce the exposure time down to 10 s per one image.

The obtained neutron images are corrected for the background noise of the detector system and normalized to the incident neutron beam using the ImageJ software package. Tomographic reconstruction of the studied objects from a set of angular projections is performed using the SYRMEP Tomo Project software package. The VGStudio MAX 2.2 software package is used for visualization and analysis of obtained 3D data.



**Fig. 32.** Photograph of the detector system: 1 – neutron scintillator and module of rotating mirrors; 2 – lens with variable focal length; 3 – CCD-camera.

**Main technical parameters of the NRT experimental station for neutron radiography and tomography**

<b>Characteristic collimation parameter <math>L/D</math>:</b>	200 - 2000
Diameter of inlet aperture of collimation system, $D$	5 - 50 mm
Distance between the collimation system aperture and sample position, $L$	10 m
Size of neutron beam (field-of-view, $FOV$ of station)	$20 \times 20 \text{ cm}^2$
<b>Characteristic parameters of detecting video camera</b>	
Type of high-sensitivity video camera	HAMAMATSU
<b>Size of CCD sensor (pixels)</b>	$4008 \times 2672$
<b>Size of CCD sensor (mm)</b>	$36 \times 24$
<b>Size of one pixel in sensor (<math>\mu\text{m}</math>)</b>	$9 \times 9$
<b>Cooling</b>	Based on Peltier element, down to $-25^\circ\text{C}$
<b>Parameters of optical system of video camera</b>	
	Based on Nikon lens with a focal length of 50 mm and an aperture width of 1:1.4D

**4.2. Detailed description of proposals for modernization of the instrument**

1. To expand the capabilities of the NRT station for neutron experiments in real time, it is necessary to develop and manufacture a compact detector system based on a high-speed camera with an sCMOS sensor. The new detector system requires the purchase of optical, mechanical and protective components: a video camera, mirrors, fasteners, borated polyethylene plates, software. The development of such a system will make it possible to

conduct neutron radiographic test experiments on other neutron beamlines of the IBR-2 reactor.

2. Due to long-term operation of the detector system at the NRT station, the HAMAMATSU CCD-camera needs to be replaced with a new one or similar one.
3. To improve the optical parameters of the detector system, it is necessary to purchase optical components: objective lens, extension rings, new types of scintillators, etc.
4. Work is planned on the design and implementation of an automated system for changing samples during long-term experiments. This will require the purchase of stepper motors, structural components, and control electronics.

#### ***4.3. Current technical parameters of the instrument in thermal and cold modes of the moderator***

The NRT experimental station is positioned on beamline 14 of the IBR-2 reactor, where the installation of a cold moderator is not planned.

#### ***4.4. Expected technical parameters after modernization of the instrument***

The efficiency of the detector system and automation of the sample-changing procedure is expected to improve.

#### ***4.5. Relevance of the instrument development for the concept of a suite of spectrometers within the project of a new neutron source at JINR***

For the new neutron source at FLNP, it is planned to build several stations for neutron radiography and tomography, including a station for energy-selective neutron imaging. Thus, the methodological and scientific development of the research direction of neutron radiography and tomography is one of the most promising and relevant research areas for the future FLNP scientific program.

#### ***5. Expected scientific results, comparison with the world level***

X-ray radiography and tomography techniques, particularly in the field of medical diagnostics, have given impetus to the development of neutron tomography. In the 1980s, the first neutron tomography experiments were conducted, allowing for the reconstruction of three-dimensional images of the distribution of internal components within massive samples. The development of research neutron sources, including those based on charged particle accelerators (spallation neutron sources), the evolution and optimization of the parameters of extracted neutron beams, neutron optics and elements of neutron detector systems have also influenced the development of neutron radiography and tomography methods. These methods, as representatives of the family of non-destructive testing methods, have become widely used in applied scientific research of technological and engineering objects; in paleontology and geophysics; in the study of electric current sources and batteries; processes associated with the penetration of water into the thickness of various materials. In recent years, there has been a rapid increase in the number of publications devoted to non-destructive methods of studying cultural heritage objects. Due to these broad application possibilities, as well as the advent of modern neutron imaging detectors based on CCD- (charge-coupled device) and CMOS- (complementary metal-oxide-semiconductor) cameras and the development of high-flux neutron sources, there is currently a high level of activity in the development of neutron radiography and tomography techniques, as well as in the construction of specialized experimental facilities at neutron centers around the world. Work is currently underway on the design and construction of new experimental neutron-imaging instruments: ODIN at the future European pulsed neutron source ESS (Lund, Sweden) and VENUS at the spallation neutron source SNS (Oak Ridge, USA).

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It should be noted that the NRT experimental station allows measurements with samples up to 200 mm in size, which is competitive with the main neutron radiography facilities at the world's neutron centers. The configuration of the neutron beamline provides a thermal neutron flux at a level of  $1.5 \times 10^6 \text{ n/s} \cdot \text{mm}^2$ , which is an order of magnitude lower than at other neutron centers. This circumstance imposes objective limitations on the possibilities of further increasing the resolution of the NRT station. The problem can be solved by optimizing the detector system and developing software for tomographic data analysis.

The high level of scientific research in the field of neutron radiography and tomography has enabled the development of a new area of applied research at FLNP, related to the non-destructive analysis of the internal structure of three-dimensional objects, products, and materials, including objects of paleontological natural heritage, engineering products, objects of extraterrestrial origin (meteorites), and archaeological objects of cultural heritage.

### **6. Requested resources, costs and time frames of instrument modernization**

The costs of the modernization stages are given in **Table 13 (Section 4)**.

<b>№</b>	<b>Description of activities</b>	<b>2026</b>	<b>2027</b>	<b>2028</b>	<b>2029</b>	<b>2030</b>
1	New high-sensitivity camera for detector system					
2	Compact detector system for neutron radiography					
3	Additional optical components: objective lens, extension rings, new types of scintillators					
4	Design and realization of an automated sample-changing system for long-term experiments					
5	Purchase of stepper motors, structural components, control electronics for automated sample-changing system					

### **References**

1. D.P. Kozlenko, S.E. Kichanov, E.V. Lukin, A.V. Rutkauskas, G.D. Bokuchava, B.N. Savenko, A.V. Pakhnevich, A.Yu. Rozanov "Neutron Radiography Facility at IBR-2 High Flux Pulsed Reactor: First Results" Physics Procedia, 69, 87-91 (2015).
2. D.P. Kozlenko, S.E. Kichanov, E.V. Lukin, A.V. Rutkauskas, A.V. Belushkin, G.D. Bokuchava, B.N. Savenko "Neutron radiography and tomography facility at IBR-2 reactor", Physics of Particles and Nuclei Letters, 13, 3, 346-351 (2016).
3. A.V. Rutkauskas, D.P. Kozlenko, S.E. Kichanov, G.D. Bokuchava, E.V. Lukin, B.N. Savenko. "Investigation of the Neutron Transmission Spectra of Materials Promising for the Manufacturing of Crystalline and Polycrystalline Filters", Journal of Surface Investigation X-ray Synchrotron and Neutron Techniques, 9(2):317-319 (2015).
4. E.V. Lukin, D.P. Kozlenko, S.E. Kichanov, A.V. Rutkauskas, G.D. Bokuchava, B.N. Savenko. "First attempts on energy-selective neutron imaging at IBR-2", Physics Procedia, 69, 271-274 (2015).
5. S.E. Kichanov, D.P. Kozlenko, E.V. Lukin, A.V. Rutkauskas, E.A. Krasavin, A.Y. Rozanov, B.N. Savenko. "A neutron tomography study of the Seymchan pallasite", Meteoritics & Planetary Science, 53, 10, 2155-2164 (2018).

6. M.G. Abramson, I.A. Saprykina, S.E. Kichanov, D.P. Kozlenko, K.M. Nazarov. "A Study of the Chemical Composition of the 3rd Century AD Bosporan Billon Staters by XRF-Analysis, Neutron Tomography and Diffraction", *Journal of Surface Investigation: X-ray, Synchrotron and Neutron Techniques*, 12, 1, 114-117 (2018).
7. S.E. Kichanov, K.M. Nazarov, D.P. Kozlenko, I.A. Saprykina, E.V. Lukin, B.N. Savenko. "Analysis of the internal structure of ancient copper coins by neutron tomography", *Journal of Surface Investigation: X-ray, Synchrotron and Neutron Techniques*, 11, 3, 585-589 (2017).
8. S.E. Kichanov, I.A. Saprykina, D.P. Kozlenko, K. Nazarov, E.V. Lukin, A.V. Rutkauskas, B.N. Savenko. Studies of Ancient Russian Cultural Objects Using the Neutron Tomography Method, *J. Imaging*, 4(2), 25 (2018).
9. I.A. Saprykina, S.E. Kichanov, D.P. Kozlenko, E.V. Lukin. "The capabilities of neutron tomography in archaeology on an example of the study of old Russian jewelry from the Tver hoard of 2014", *Rossijskaja Arheologija*, №3 (2018) 36-42 (In Russian).
10. I.A. Saprykina, S.E. Kichanov, D.P. Kozlenko. «Possibilities, Limitations, and Prospects of Using Neutron Tomography and Radiography for Preservation of Archaeological Heritage Objects», *Cryst. Rep.* 64(1), 177-180 (2019).

## **NERA – Inverse Geometry Spectrometer for simultaneous investigations of atomic structure and dynamics of condensed matter**

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### **1. Abstract**

The inverse geometry method, in which the sample under study is irradiated with a “white” neutron beam from a pulsed source, provides a unique possibility for simultaneous investigation of diffraction and inelastic neutron scattering spectra, i.e., the structure and dynamics of matter depending on external conditions on the sample. This technique is particularly useful for studying polymorphism and phase transitions in crystalline materials depending on temperature and pressure. At the same time, the inelastic neutron scattering technique is an *indispensable* tool in the study of biologically active materials, which, in combination with quantum chemistry methods, makes it possible to gain insight into the processes occurring in them. The NERA spectrometer positioned at a distance of 109 m from the moderator allows one to obtain high-resolution spectra. The principle of operation of the inelastic neutron scattering spectrometer in inverse geometry is that the final energy of scattered neutrons is registered by an analyzer made of highly oriented pyrolytic graphite (HOPG) and a filter made of beryllium cooled to 77 K, which suppresses high-order reflections. Liquid nitrogen is used to cool the beryllium filters at a rate of approximately 60 l per day. Replacing the existing nitrogen tank, which is nearing the end of its service life, would significantly improve the operating conditions of the NERA. For example, the maximum permissible pressure in the tank currently cannot exceed 0.7 atm, and there is a high probability that its further operation will be prohibited upon the next technical inspection. Also, the creation of an automatic system for maintaining the level of liquid nitrogen in the filters would be an important element in improving the operating conditions of the spectrometer.

### **2. Scientific program, relevance and comparison with the world level**

The NERA spectrometer [1,2] was developed and built at the IBR-2 in 1986-92 in collaboration between the H. Niewodniczański Institute of Nuclear Physics in Krakow and the Frank Laboratory of Neutron Physics, JINR, Dubna. Since the beginning of its operation in 1993,

the NERA spectrometer has been used within the FLNP User Program for studies of molecular dynamics and phase transitions using inelastic neutron scattering and neutron diffraction.

The main topics in recent years have been the research into molecular-ionic crystals, inclusive systems and pharmacologically active compounds. Neutron spectroscopy provides a highly accurate verification of the validity of quantum-chemical calculations of the structure, dynamics, and electronic properties of the molecules of these materials. Urea [3] and methanol [4] have also been studied for this purpose, as examples of compounds with hydrogen bonds. It is also worth noting the studies of phase transitions and the dynamics of methyl groups in methylbenzene compounds [5,6], which led to the choice of mesitylene-based solutions as a neutron moderator for the cold source at the IBR-2 reactor. Also noteworthy is the study of the structure, phase transitions, and dynamics in a number of inorganic materials.

The spectrometers NERA at IBR-2, TOSCA at ISIS, VISION at ORNL, and LAGRANGE at ILL are currently the best world-class instruments for neutron spectroscopy of hydrogen-containing materials.

### **3. Scientific and methodological groundwork laid in FLNP JINR**

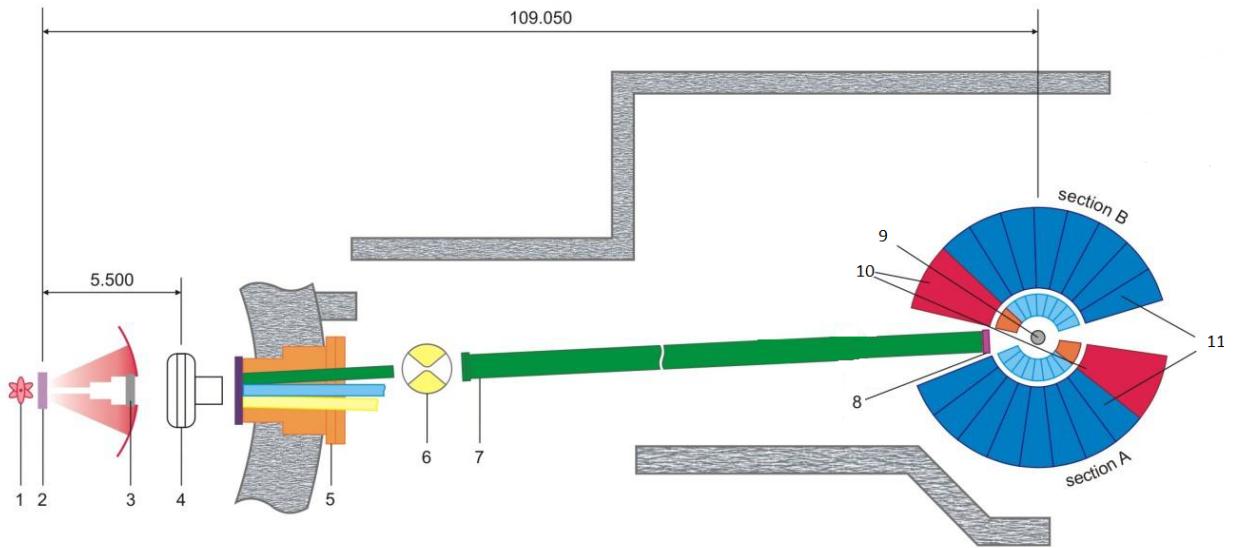
Inelastic neutron scattering (INS) is the main universal method for studying the atomic and magnetic dynamics of a wide variety of materials. The method was proposed in the middle of the last century and continues to be improved to this day. Its competitiveness is based on a set of properties inherent to thermal neutrons, which are important from the point of view of their application to problems of condensed matter physics. FLNP has been developing the INS method from the very beginning: inverse geometry spectrometers on the IBR-30; on the IBR-2 – KDSOG and NERA, DIN-2PI direct geometry spectrometer.

### **4. Current status of the instrument and proposals for its modernization**

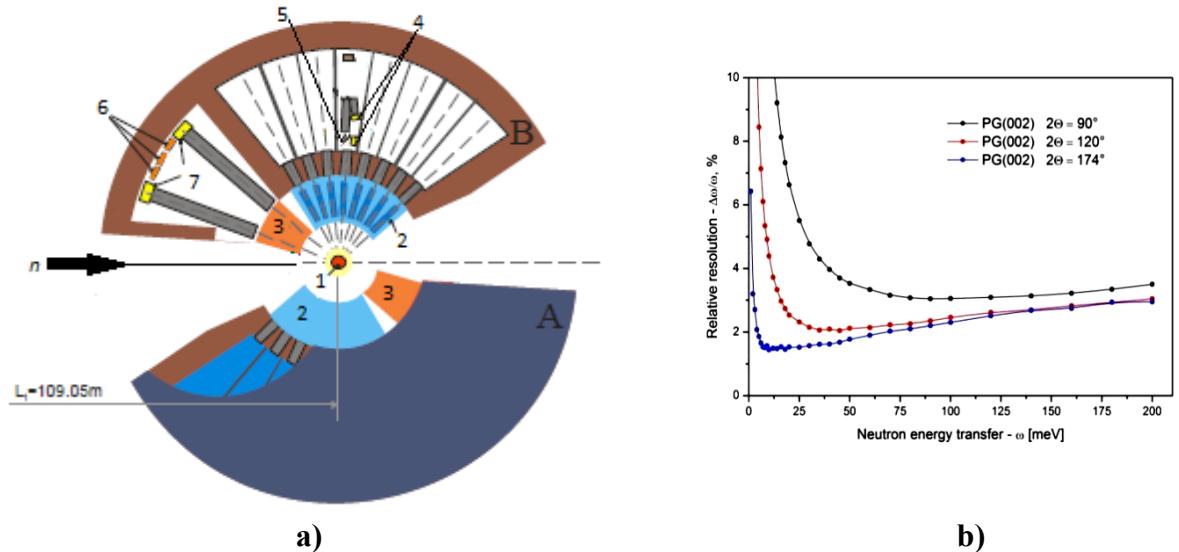
#### ***4.1. Information about the current status of the instrument***

The layout of the NERA spectrometer and its main components are shown in Fig. 33. The beginning of the mirror neutron guide (7) is located at a distance of 6 m from the moderator immediately behind a disk chopper ( $\varnothing$  134 cm) of delayed neutrons (4). The thickness of the  $\text{TiH}_2$  layer is 6.2 cm, which is twice as much as before modernization. A drum  $\lambda$ -chopper (6) with a radius of 48 cm is located at a distance of 26.95 m from the moderator and rotates at a rate of 2.5 Hz.

The neutron guide consists of two parts: the first part is straight, 75 m long, with a constant beam cross-section of  $16 \times 5$  cm, and the second part is vertically converging, 25 m long, with an inlet cross-section of  $16 \times 5$  cm and an outlet cross-section of  $5 \times 5$  cm. After scattering from a sample, usually in a cryostat (this allows the sample temperature to be varied over a wide range from 5 K to 300 K), the neutrons pass through an energy analyzer consisting of a cooled beryllium filter and highly oriented pyrolytic graphite plates that only allow neutrons with a fixed final energy of 4.65 meV to pass through. The NERA spectrometer consists of two mirror-symmetric sections. The main units of the spectrometer and one of the sections are illustrated in Fig. 34 a), and the resolution function is shown in Fig. 34 b).



**Fig. 33.** 1 – IBR-2 reactor core, 2 – thermal and cold moderators, 3 – beam shutter, 4 – delayed neutron chopper, 5 – vacuum splitter of three neutron channels, 6 –  $\lambda$ -chopper, 7 – vacuum mirror neutron guide, 8 – incident beam monitor, 9 – sample, 10 – neutron diffraction sections, 13 – INS sections.



**Fig. 34.** a) 1 – sample, 2 – beryllium filters, 3 – collimators, 4 – He-3 detectors, 5 – pyrolytic graphite analyzers (INS), 6 – detectors for high-intensity diffraction, 7 – detectors for high-resolution diffraction; b) Relative resolution of INS spectra obtained using a pyrolytic graphite (PG) (002) analyzer at different scattering angles  $2\lambda = 9^\circ, 120^\circ$  and  $174^\circ$ .

On the NERA spectrometer, the sample is irradiated with a white neutron beam. Therefore, the installation of detectors without an energy analyzer allows for neutron diffraction measurements to be carried out in parallel with inelastic neutron scattering measurements.

***Main technical parameters of the NERA inverse geometry spectrometer***

<b>Thermal neutron flux on the sample</b>	$4.6 \times 10^6 \text{ n/cm}^2/\text{s}$
<b>Energy transfer range</b>	1 - 200 meV
<b>Energy resolution at the elastic line</b>	FWHM = 0.8 meV
<b>Scattering angle range</b>	10° – 170°

***5. Expected scientific results, comparison with the world level***

As already noted, the spectrometers NERA at IBR-2, TOSCA at ISIS, VISION at ORNL, and LAGRANGE at ILL are among the best world-class instruments for neutron spectroscopy of hydrogen-containing materials.

The role of beryllium filters in the operation of the NERA inelastic neutron scattering spectrometer is undoubtedly significant. Maintaining them at nitrogen temperatures is key to conducting experiments. In recent years, first developments have been made to automate the process of filling beryllium filters with liquid nitrogen and maintaining low temperatures. To complete this work, it is necessary to purchase a new nitrogen storage tank.

***6. Requested resources, costs and time frames of instrument modernization***

The costs of the stages of modernization of the spectrometer are presented in **Table 14 (Section 4)**.

**References**

1. I. Natkaniec, S.I. Bragin, J. Brankowski and J. Mayer, in Proc. ICANS XII Meeting, Abington 1993, RAL Report 94-025, vol. I., p.89- (1994).
2. I. Natkaniec, D.M. Chudoba et al. „Parameters of the NERA spectrometer for cold and thermal moderators of the IBR-2 pulsed reactor” J. Phys.: Conf. Ser. 554 012002 (2014); DOI 10.1088/1742-6596/554/1/012002 .
3. M.R. Johnson, K. Parlinski, I. Natkaniec, S. Hudson. „Ab initio calculations and INS measurements of phonons and molecular vibrations in a model peptide compound – urea”, Chemical Physics, Vol. 291, No. 1, 2003, 53-60.
4. I. Natkaniec, K. Holderna-Natkaniec, I. Majerz, K. Parlinski. Neutron spectroscopy of deuterated substitutes and DFT modeling vibrational spectra of methanol clusters. Chemical Physics 317 (2005) 171-177.
5. I. Natkaniec, K. Holderna-Natkaniec, J. Kalus. Neutron scattering studies of methyl derivatives of benzene selected as potential materials for cold neutron moderators. Physica B 350 (2004) 651-653.
6. I. Natkaniec, K. Holderna-Natkaniec. Structural phase transitions and dynamics of solid mesitylene investigated by diffraction and inelastic incoherent neutron scattering methods. In Proceedings of 6th International Workshop on Advanced cold moderators. Ed. H. Conrad, Matter and Materials. Vol. 20, p.103-111, Forschungszentrum Julich, 2004.

### 3. PROPOSALS OF NEW INSTRUMENTS

#### BJN — Inverse-Geometry Inelastic Neutron Scattering Spectrometer

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**Main participants:** D.M. Chudoba, E.A. Goremychkin, A.V. Belushkin, A.A. Kruglov, V.I. Bodnarchuk, A.V. Churakov, A.N. Chernikov, A.B. Eresko, V.I. Sukhanov

**Collaborating organizations:** Petersburg Nuclear Physics Institute, NRC "Kurchatov Institute", Gatchina, Russia;

##### 1. Abstract

At present, the Frank Laboratory of Neutron Physics operates one inelastic neutron scattering (INS) spectrometer with inverse geometry—NERA.

The parameters of the NERA spectrometer, which has been upgraded in recent years, make it possible to carry out INS experiments at a good level, comparable to that provided by instruments of the same type operating in advanced neutron research centers. Nevertheless, all possible options for upgrading NERA have essentially been exhausted, and since it was built at the end of the last century, many of its units are outdated. So, the development of a new inverse-geometry spectrometer based on advanced technologies will make it possible to create an instrument significantly superior to similar setups in other neutron centers.

Thus, although the available INS spectrometer provides certain possibilities for conducting research into the dynamics of condensed matter, it does not fully correspond to the level of instruments of this class in the world's leading neutron centers and does not meet the needs of the user community. In order to make FLNP competitive in this area of research, to match or surpass the parameters of similar instruments in other world's neutron centers, it is necessary to build new INS spectrometers using advanced neutron optics and new design solutions to provide high resolution, low background and wide range of energy transfer. Nowadays, advanced technologies employed in neutron guides and optical devices make it possible to use the brightness of IBR-2 with maximum efficiency, which, combined with the necessary design solutions, will enable the creation of new neutron instruments with record-breaking parameters. The first phase of the project for the development of new INS instruments involves the construction of a new spectrometer with inverse geometry on beamline 2 of the IBR-2 reactor.

##### 2. Scientific program, relevance and comparison with the world level

Spectroscopic studies of condensed matter dynamics encompass a variety of experimental methods aimed at obtaining information about the structure, chemical bonding, and intermolecular interactions in materials under study. Infrared (IR) absorption spectroscopy and Raman spectroscopy are probably the best-known examples and most popular methods used for this purpose. Inelastic neutron scattering (INS) is also a spectroscopic method for studying atomic and molecular dynamics, which, in combination with advanced quantum-chemical calculations and complementary experimental methods, provides unique insight into specific vibrational properties of materials. There is an ever-growing demand for inelastic neutron scattering instruments, which is driven by the advantages of INS over optical vibrational spectroscopy:

1. No selection rules,
2. Isotopic sensitivity (isotopic substitution to hide or highlight selected molecular fragments),
3. High penetrating power of neutrons,
4. Minimum energy release in the sample,
5. Possibility to study chemical processes *in situ*,
6. Possibility to study magnetic excitations using polarized neutrons,
7. Experimental information as a function of both energy transfer and momentum transfer,
8. Relatively simple theoretical description and modeling.

The possibility of neutron spectroscopy to see overtones and their combinations is unique, and, in this respect, INS is clearly superior to IR and Raman spectroscopy (in optical methods such vibrations contribute about 1% of the spectral intensity, whereas in INS spectra they can be up to 75% of the intensity).

The planned research program for the new INS inverse geometry spectrometer includes studying the dynamics of:

1. methyl groups in molecular crystals;
2. molecular crystals and glass-forming agents at low temperatures in combination with the studies by complementary techniques and *ab initio* quantum-chemical calculations;
3. pharmaceutical substances in bulk (native) state and in the form of “micronized” or amorphized powders;
4. materials in spatial confinement: “hard” nanomatrices (for example, membranes) and “soft” confinement (for example, microfibre);
5. materials for energy storage, for example, solid polymer electrolytes with plasticizers for lithium batteries;
6. catalysts;
7. photonic materials for industrial applications.

Every advanced neutron research center in the world operates one (e.g., ISIS, ORNL) or even more (e.g., ILL) highly efficient optimized INS spectrometers. The development of a high-quality INS spectrometer optimized for low wavenumbers has good prospects for realization at the IBR-2 pulsed reactor.

### **3. Scientific and methodological groundwork laid in FLNP JINR**

Considerable experience has been accumulated in FLNP JINR in the application of neutron spectroscopy on the basis of operation of the KDSOG, NERA, and DIN-2PI spectrometers [1,2], which over many years of service have been used to conduct numerous experiments on the dynamics of condensed matter.

### **4. Proposals for design of the instrument**

#### ***4.1. Detailed description of proposals for design of the instrument***

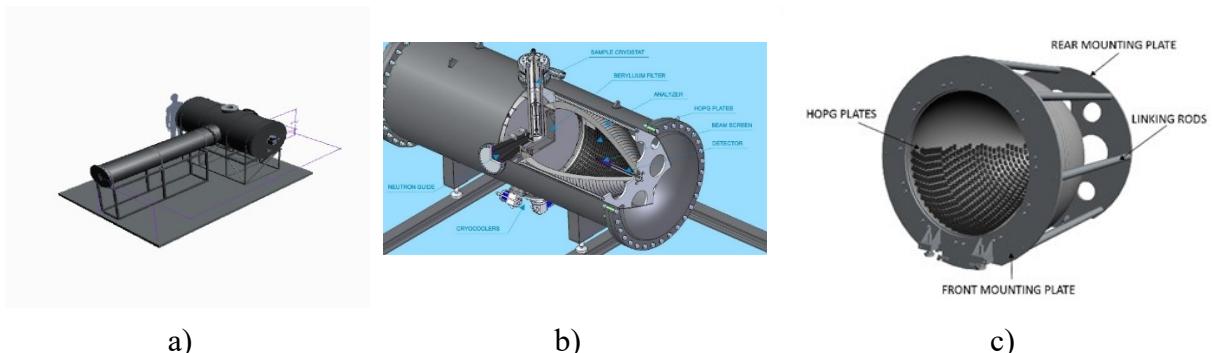
The new BZN inverse-geometry inelastic neutron scattering spectrometer [3] will be located on beamline 2 of the IBR-2 reactor (building 117/1). The main units of the spectrometer will include a mirror neutron guide, a cascade of choppers, a sample assembly, an analyzer made of highly-oriented pyrolytic graphite and a beryllium filter, and detectors. The proposed layout of the new spectrometer is shown in Fig. 35.



**Fig. 35.** Layout of the new inelastic neutron scattering spectrometer with inverse geometry on beamline 2 of the IBR-2 reactor.

#### Geometry of the secondary spectrometer

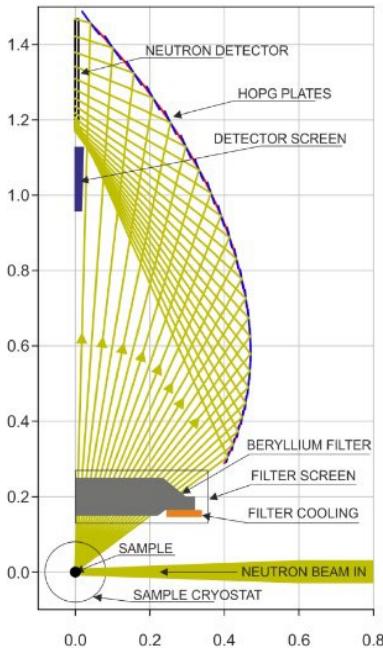
The schematic of the secondary inelastic neutron scattering spectrometer with inverse geometry is shown in Fig. 36.



**Fig. 36.** Geometry of the new INS spectrometer with inverse geometry: a) general view; b) view of the part with pyrolytic graphite analyzers in a vacuum tube; c) part of the spectrometer with pyrolytic graphite analyzers.

The new BZN inelastic neutron scattering spectrometer will consist of two symmetrical parts. Each section will be shaped like a paraboloid of revolution and lined with plates of highly-oriented pyrolytic graphite. It is proposed to install approximately 1000 plates measuring 40x40 mm on each section.

The schematic diagram of neutron scattering from a sample is shown in Fig. 37.



**Fig. 37.** Schematic diagram of neutron scattering and detection on the new INS inverse-geometry spectrometer.

It is planned to install one detector in each arm of the spectrometer. The detectors will consist of several cylindrical counters filled with  ${}^3\text{He}$ , inscribed within a circle with a diameter of 50 mm.

#### 4.2. Current technical parameters of the instrument in thermal and cold modes of the moderator

Since the spectrometer will be placed in the existing building 117/1, the optimal distance from the moderator surface to the sample was chosen to be 105 m, which provides optimal time resolution over the entire range of energy transfer.

The neutron-optical elements of the mirror neutron guide will be optimized for the wavelength range of 0.5 - 1 Å (energy transfer range of 80 – 300 meV). The final section of the neutron guide ( $\sim 25$  m) will converge to the beam size on the sample ( $3 \times 3 \text{ cm}^2$ ).

The INS inverse-geometry spectrometer on beamline 2 will operate with the existing thermal grooved moderator.

#### 4.3. Expected technical parameters

Parameter	NERA	BJN	Notes
Analyzer area	$15 \times 3 \times 25$ $1125 \text{ cm}^2$	$4 \times 4 \times 1962$ $31392 \text{ cm}^2$	The analyzer area is 28 times larger
Energy transfer	up to 160 meV	up to 300 meV	
Neutron guide inlet/outlet ratio	$16 \times 5 \text{ cm}^2 / 5 \times 5 \text{ cm}^2$ 3.2	$20 \times 20 \text{ cm}^2 / 3 \times 3 \text{ cm}^2$ 44.44	increase in flux density (without taking into account the higher quality of the neutron guide) $44.44/3.2 = 14$
Solid angle	$\sim 0.2 \text{ sr}$	$\sim 5.64 \text{ sr}$	Increase in solid angle 28 $28 \times 14 = 392$
Ratio of luminosity of BJJ and NERA			times greater, i.e. it will be possible to measure a sample weighing 10-20 mg

## 5. Expected scientific results, comparison with the world level

One of the advantages of the new spectrometer at the IBR-2 reactor will be its high luminosity. This feature will allow for more efficient and virtually lossless use of the neutron beam, significantly reducing the duration of experiments and enabling the use of low-mass samples.

Overall, the proposed instrument will be one of the best in terms of neutron efficiency. Results obtained with the new spectrometer will be comparable to those obtained with leading European inelastic neutron scattering instruments, such as TOSCA (ISIS, UK) and VESPA (ESS, Sweden).

## 6. Requested resources, costs and time frames of the instrument construction

The time frames and costs for the stages of spectrometer construction are shown in **Table 15 (Section 4)**.

## References

1. I. Natkaniec, S.I. Bragin, J. Brankowski and J. Mayer, in Proc. ICANS XII Meeting, Abington 1993, RAL Report 94-025, vol. I, p.89- (1994).
2. I. Natkaniec, D.M. Chudoba et al. „Parameters of the NERA spectrometer for cold and thermal moderators of the IBR-2 pulsed reactor” *J. Phys.: Conf. Ser.* 554 012002 (2014); DOI 10.1088/1742-6596/554/1/012002
3. M. Klepacka, A. Belushkin, E. Geremychkin, D. Chudoba “High luminosity inverse geometry inelastic neutron scattering spectrometer at pulsed fast reactor” *Rev. Sci. Instrum.* 96, 033902 (2025); <https://doi.org/10.1063/5.0244264>

## SANSARA — Small-Angle Neutron Scattering Instrument

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**Main participants:** Yu.E. Gorshkova, M. Yerdauletov, F.S. Napolksy

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Moscow Institute of Physics and Technology, Russia;

### 1. Abstract

The SANSARA (Small-Angle Neutron Scattering And RAdiography) instrument is a small-angle neutron scattering (SANS) diffractometer combined with a neutron radiography (NRG) station located at beamline 10a with cold neutrons. The construction of a new advanced small-angle neutron scattering instrument at IBR-2 with the most efficient realization of the capabilities of neutron scattering, is an important part in the development of structural methods for studying nanosystems at the reactor. Following the current trends in the development of neutron centers, the instrument is a general-purpose small-angle diffractometer aimed at providing a wide range of possibilities for conducting SANS experiments. The optimization of the setup follows the conventional configuration of a SANS instrument at a cold neutron source. To enhance the efficiency of using cold neutrons, it is proposed to combine a SANS diffractometer with a neutron radiography station.

### 2. Scientific program, topicality and comparison with the world level

The SANSARA (Small-Angle Neutron Scattering And RAdiography) setup is a small-angle neutron scattering (SANS) diffractometer combined with a neutron radiography (NRG) station located at beamline 10a with cold neutrons.

Small-angle neutron scattering is one of the widely used methods of structural studies of nano-objects – systems whose properties are determined by structural features at a level of 1-100 nm. The scientific program of small-angle instruments includes a variety of research areas:

- Complex fluids, including magnetic fluids, surfactant solutions, anisotropic fluids, liquid crystals, etc.
- Magnetic nanocomposites
- Polymers, including magnetic polymers
- Biological macromolecules, membranes, vesicles
- Liposomes, including magnetosomes
- Dispersions of carbon materials
- Inhomogeneities in structural materials

Today, every advanced neutron center runs at least one such instrument. Moreover, there is a tendency to create several small-angle instruments in one center, optimized for different types of tasks. This concept provides the most effective use of this method. The small-angle scattering

instrument is a ‘fast-payback’ project in the scientific sense, since the average time per one experiment is only a few hours. The latter factor determines the possibility of conducting a complete study using neutrons in a relatively short time. The small-angle scattering instruments operating today at IBR-2 cannot satisfy the growing demand for research using this method. Thus, analyzing the statistics of the use of neutron instruments at the IBR-2 reactor within the user policy over the past 10 years, one can conclude that there is a stable excess of experimental proposals over the capacity of the YuMO setup, operating at the level of world standards. In view of the current boom in nanosciences, one can only expect an increase in demand for SANS experiments in the nearest future. The use of neutron scattering in the study of nanosystems is determined by two factors: (1) wide possibilities of contrast variation based on the isotopic substitution of atoms in the systems under study; (2) magnetic neutron scattering, which allows obtaining information on magnetic correlations in magnetic systems. Thus, the construction of a new advanced small-angle neutron scattering instrument at IBR-2 with the most efficient realization of the capabilities of neutron scattering, is an important part in the development of structural methods for studying nanosystems at the reactor.

### 3. Scientific and methodological groundwork laid in FLNP JINR

The YuMO time-of-flight small-angle neutron scattering instrument [1], which is oriented to work with the thermal moderator ( $T = 300$  K) and uses collimation with the direct view of the moderator [2], is successfully operating at the IBR-2 pulsed reactor. To obtain a scattering curve in the range  $q = 0.05 - 5 \text{ nm}^{-1}$ , a special procedure is used with a continuous calibration to the vanadium standard placed in front of the detector. The time spent on the calibration is compensated by a high peak intensity when using the thermal moderator, which reduces the characteristic measurement time per one curve to an interval of 10 - 90 min (depending on the cross section of the sample).

The main advantage of YuMO is a two-detector system with large-area ring detectors for detecting isotropic scattering and central openings in the detectors allowing the direct beam to pass. This feature makes it possible to realize a record ( $\sim 100$ ) dynamic  $q$ -range (scanning range in one measurement run). Thus, the setup can be effectively used to study changes in the nanoscale range (10-100 nm) in real time (time resolution down to 1 min).

This feature, however, imposes some limitations. Since the instrument is optimized for the thermal moderator, there is a limitation in resolution at the minimum  $q$ -value, which narrows the sensitivity in the size range (submicron region). Also, because of the direct view of the source, the background level is comparatively high. The detector openings complicate the use of conventional designs of position-sensitive detectors (PSD), as well as the use of direct measurement of the transmission and corresponding calibration.

The design of the new SANS instrument is aimed at eliminating the above limitations, which is possible due to the availability of a cold moderator. The natural compensation will be a decrease in the average beam intensity at the sample. The availability of a large-area 2D PSD with the conventional procedure for obtaining and calibrating scattering curves at two detector positions (short and long flight paths) will allow experiments for a large number of equilibrium systems, including systems with scattering anisotropy (oriented systems, magnetized magnetic systems). In turn, this will make it possible to focus the YuMO scientific program on the study of kinetic phenomena.

#### 4. Proposals for design of the instrument

##### 4.1. Information about the current status of the project

The concept, simulation and selection of the optimal configuration and moderator temperature, as well as estimates of the instrument parameters were published in [3].

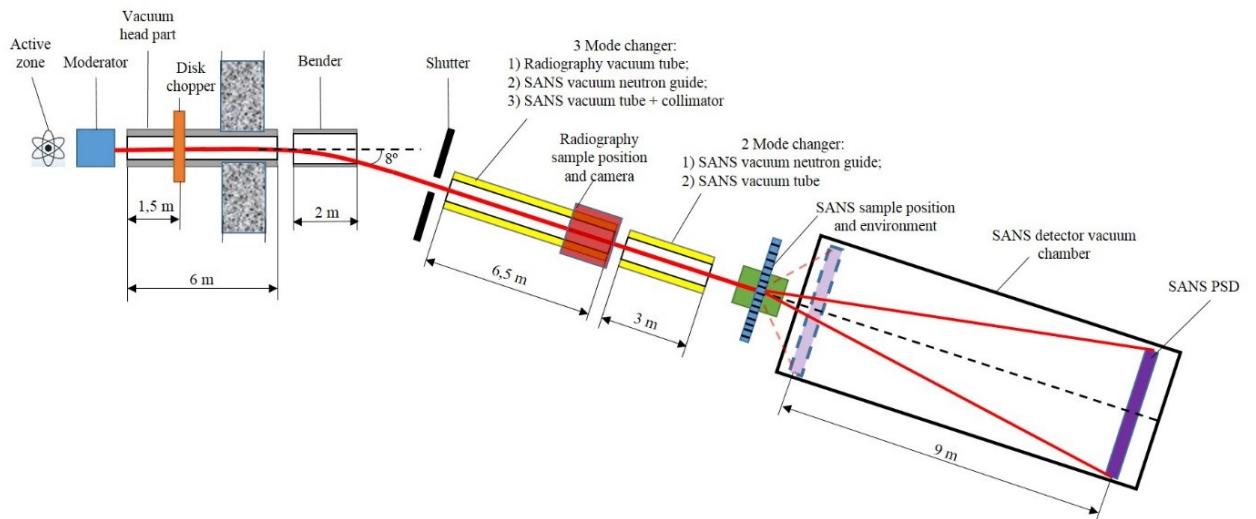
The schematic layout of the proposed instrument is presented in Fig. 29. For the implementation of the project, it is proposed to use channel 10 of the IBR-2 reactor, which is currently split into two beamlines: 10a and 10b. At present, the GRAINS reflectometer (reflectometer with a horizontal sample plane) is operating at beamline 10b [4]. Beamline 10a is equipped with a head part with a neutron guide (supermirror  $m = 2$ ) and a background chopper in the ring corridor. Also, the head part is followed by the previously installed multi-slit optical beam deflector (bender), a supermirror  $m = 2$ , bending angle of  $8^\circ$  at a length of 2 m.

##### 4.2. Detailed description of proposals for design of the instrument

The main factors determining the parameters of SANSARA are: (1) minimization of fast neutron background; (2) measurement in a wide range of momentum transfer (minimum  $q \sim 0.01 \text{ nm}^{-1}$ ).

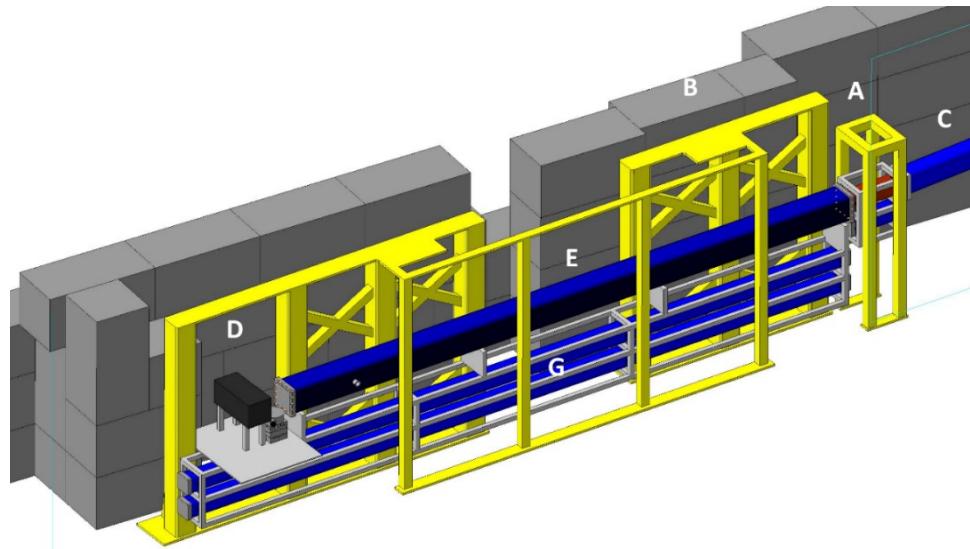
The implementation of (1) is provided by the tangential nature of beamline 10 relative to the reactor core and the use of a neutron bender with the beam axis diverted from the direct view of the neutron moderator.

The implementation of (2) is provided by the availability of a cold moderator at beamline 10.



**Fig. 29.** Schematic layout of SANSARA.

A replaceable beam-forming system together with an additional shutter are installed behind the bender (Fig. 30). The system provides the following options: *NRT-COLD* (radiography) and SANS (small-angle neutron scattering). Switching between the options is provided by three separate beam-forming units in the vertical direction (one unit for *NRT-COLD* and two units for SANS) via remote control.



**Fig. 30.** Conceptual view of the beam-forming system of the SANSARA instrument in the experimental hall of IBR-2. Main units: (A) movable shutter with a vacuum tube; (B) mechanical part for changing the vertical position of neutron guides to form a beam; (C) bender; (D) NRT platform; (E) NRT vacuum tube; (G) two SANS beam-forming units (neutron guide and vacuum tube).

#### 4.3. Current technical parameters of the instrument in thermal and cold modes of the moderator

For the most efficient use of neutron scattering in terms of sensitivity to the size range, the SANS instrument with the conventional configuration requires a cold moderator with a temperature of 30 K. The availability of cold neutrons additionally allows the use of neutron-optical devices to separate fast and cold neutrons and significantly reduce the background level at the sample. Also, to enhance the efficiency of using cold neutrons, this research technique can be relatively easily combined with other methods, such as neutron radiography and tomography. As a result of the use of a bender (beam deflector) optimized for a temperature of 30 K, the instrument is intended only for operation in the cold mode of the moderator.

#### 4.4. Expected technical parameters

<b>Beam size</b>	$50 \times 50 \text{ mm}^2$
<b>Neutron wavelength range:</b>	$0.5 - 15 \text{ \AA}$
<b>q-range</b>	$0.001 - 1 \text{ \AA}^{-1}$
<b>Angular resolution</b>	$5 - 20 \text{ %}$
<b>Sample dimensions</b>	$5 \times 5 \times 1 - 20 \times 50 \times 50 \text{ mm}$
<b>Neutron flux at sample position</b>	$1.0 \times 10^6 \text{ cm}^{-2} \text{ s}^{-1}$
<b>Detector</b>	2D PSD, efficiency $> 50\%$ (0.2 nm) $64 \times 64 - 80 \times 80 \text{ cm}^2$ , resolution $5 \times 5 - 10 \times 10 \text{ mm}^2$ count rate $10^5 - 10^6 \text{ s}^{-1}$

#### **4.5. Relevance of the instrument construction for the concept of a suite of spectrometers within the project of a new neutron source at JINR**

The construction of the instrument is fully consistent with the current trends in methodological development. Over the past five years, general-purpose small-angle diffractometers have been designed and are successfully operating at almost all neutron sources, both pulsed and steady-state. Their main task is to meet a huge demand for this technique. Among the examples are GP-SANS - General Purposes SANS (SNS), Sans2d (ISIS), KWS-2 (FRM-II), Bilby (ANSTO).

#### **5. Expected scientific results, comparison with the world level**

The SANS technique, which is used in solving a wide range of fundamental and applied problems within a broad research scope related to the nanoscale structure of matter, remains one of the most popular methods among neutron scattering techniques. Such kind of problems are very important and of great current interest in various sciences, including condensed matter physics, physics and chemistry of complex liquids and dispersed systems, including solutions of surface-active agents and polymers, biophysics and molecular biology, materials science. The most important area of application of small-angle scattering is the analysis of the structure of disordered systems using non-destructive testing with an emphasis on obtaining direct structural information about systems with chaotic and partially ordered arrangement of density inhomogeneities with sizes of the order of 1 - 100 nm. This includes dispersed structures of alloys, powders, and glasses (phase separation mechanisms, particle size and degree of polydispersity), structural features of polymers in various states of aggregation, weight and geometric characteristics of biological macromolecules and their complexes, biological supramolecular structures, such as biological membranes and viruses.

In terms of its characteristics, including the available range of momentum transfer, resolution, and measurement time of a typical scattering curve, the SANSARA is comparable to instruments at other international neutron centers—the Loq instrument at the ISIS pulsed source, as well as small-angle instruments at medium-flux steady-state reactors such as the YS (BNC, 10 MW). An important improvement of the SANSARA instrument compared to these setups is the expected reduction of the minimum  $q$  (to 0.01 nm), which meets modern user requirements for studying large-scale nanosystems, when a large number of measurements with varying parameters are carried out with achieving, at the same time, high-quality scattering curves in a reasonable time.

#### **6. Requested resources, costs and time frames of the instrument construction**

The construction period is 3 years. The requested resources are listed in **Table 10 (Section 4)**.

#### **References**

1. A.I.Kuklin, A.V.Rogachev, D.V.Soloviov, O.I.Ivankov, Y.S.Kovalev, P.K.Utrobin, S.A.Kutuzov, A.G.Soloviev, M.I.Rulev, V.I.Gordeliy. Neutronographic investigations of supramolecular structures on upgraded small-angle spectrometer YuMO, *J. Phys.: Conf. Ser.* 848 (2017) 012010.
2. Yu.M.Ostanevich, Time-of-flight small-angle scattering spectrometers on pulsed neutron sources, *J. Macromol. Chem.*, 15, 91-103 (1988).
3. Avdeev M.V., Eremin R.A., Bodnarchuk V.I., Gapon I.V., Petrenko V.I., Erkhan R.V., Churakov A.V., Kozlenko D.P. Concept of small-angle diffractometer in classical configuration at the cold moderator of the IBR-2 reactor. *J. Surf. Investigation* 12 (4) (2018) 638-644.
4. M.V.Avdeev, V.I.Bodnarchuk, V.I.Petrenko, I.V.Gapon, O.V.Tomchuk, A.V.Nagorny, V.A.Ulyanov, L.A.Bulavin, V.L.Aksenov. Neutron time-of-flight reflectometer GRAINS with horizontal sample plane at the IBR-2 reactor: possibilities and prospects. *Crystallography Reports* 62(6) (2017) 1002-1008.

## NRT – Neutron Radiography and Tomography Station with Cold Neutrons

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### 1. Abstract

Neutron radiography is an imaging technique based on various degrees of attenuation of the neutron beam passing through components of the sample under study with different chemical composition, density, and thickness. It is used to obtain information on the internal structure of the studied materials with a micrometer-scale spatial resolution. To radically improve the neutron radiographic contrast in neutron radiography experiments, cold neutrons are required. In addition, the spectrum of cold neutrons is preferred for realization of the energy-selective neutron imaging method. Therefore, it is planned to develop and implement a neutron radiography mode on beamline 10a of the IBR-2 reactor, where the source of cold neutrons is a curved multi-module neutron guide in combination with a cryogenic moderator.

### 2. Scientific program, topicality and comparison with the world level

Neutron radiography allows obtaining images of the internal structure of various objects [1, 2]. The resulting image is a set of finite-size dots, or pixels, reflecting the degree of attenuation of the neutron beam intensity at a specific point of the object under study. Neutron radiography and tomography are non-destructive testing methods in modern technologies. The penetration of neutrons into the thickness of a material provides information on the spatial distribution of internal components, cracks and internal defects, as well as the locations and pathways of corrosion in industrial and engineering objects. One of the possibilities for varying contrast in neutron radiography is the dependence of the elastic scattering cross-section on the neutron wavelength. Considering the development of the method, the pulsed nature of the IBR-2 reactor operation opens up broad prospects for implementing energy-dispersive neutron radiography [3]: the total neutron scattering cross-section in crystalline materials exhibits strong jumps at certain wavelengths (the so-called Bragg absorption edges). Therefore, by selecting the optimal neutron energy range in an experiment, it is possible to enhance the contrast of the constituent parts of an object made of a certain material for more detailed analysis. The energy range of incident neutrons is selected using the time-of-flight technique, which significantly expands the research capabilities of the experimental neutron radiography and tomography station.

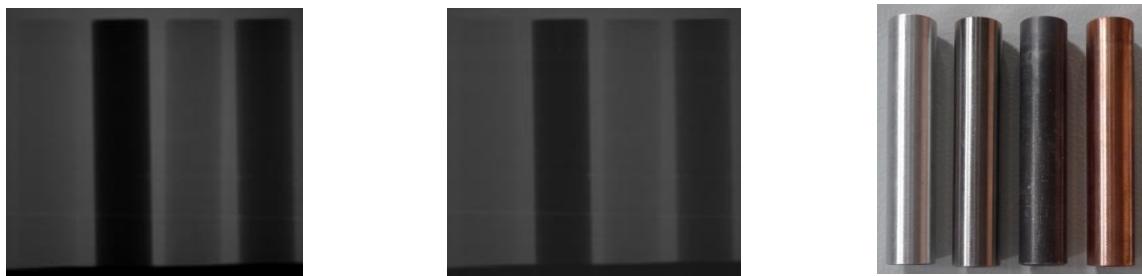
### 3. Scientific and methodological groundwork laid in FLNP JINR

Progress in modern applied experimental physics is inextricably linked to the development of new methods for the structural diagnostics of materials possessing various functional properties. A fairly wide range of physical properties and phenomena observed in various materials can be caused by certain local structural inhomogeneities at the micron level: non-uniform spatial distribution of chemical components, presence of cracks or cavities within the final product, non-

uniform chemical composition, etc. Detailed studies of the structural aspect of the formation of a particular physical phenomenon at the micron level provide the possibility to control a specific functional property of a material through the understanding of the mechanisms at the level of structural organization. In this connection, FLNP specialists have been successfully implementing and developing the experimental and methodological basis for neutron radiography and tomography [1-3] at the IBR-2 pulsed high-flux reactor (NRT) and the steady-state neutron source—the WWR-K research reactor. As a result, significant experience has been accumulated in FLNP regarding the application of this neutron method and radiographic data analysis algorithms [4-6], including experimental approaches and interpretation of the obtained results, in applied research of cement materials, objects of cultural heritage, rocks and meteorites.

Energy-dispersive neutron radiography provides additional information on structural features such as mosaicity, crystallite orientation, and internal stresses in bulk samples. A possible way to implement energy-dispersive radiography is to conduct neutron radiography experiments using the time-of-flight technique. The pulsed nature of the IBR-2 reactor makes it possible to implement such a design of the experiment: high-speed video cameras or special detectors should be used, for which the exposure start time is synchronized with the reactor pulses. Certain energy ranges can be selected from the ‘white’ neutron spectrum. For a total neutron pulse time width of 340  $\mu$ s and a flight path of about 30 m, a time-of-flight range of up to 10 ms can be covered, which corresponds to the minimum wavelength range of  $\Delta\lambda \sim 0.3$  Å.

The first results [3] on varying neutron wavelengths using a fast video camera have been published. A series of frames were obtained with a minimum time channel width of 10 ms: the time component of the video camera allows the neutron spectrum to be divided into three parts. In test experiments, the original neutron spectrum was divided into three regions: 0.2  $\div$  2 Å, 2  $\div$  3.7 Å, 3.8  $\div$  8 Å (Fig. 40). Neutron radiographic images obtained for various neutron wavelength ranges are presented in Fig. 40.



**Fig. 40.** Photograph of model objects made of aluminum, steel, lead and copper and their neutron images for the neutron wavelength ranges of 0.2  $\div$  2 Å and 3.8  $\div$  8 Å.

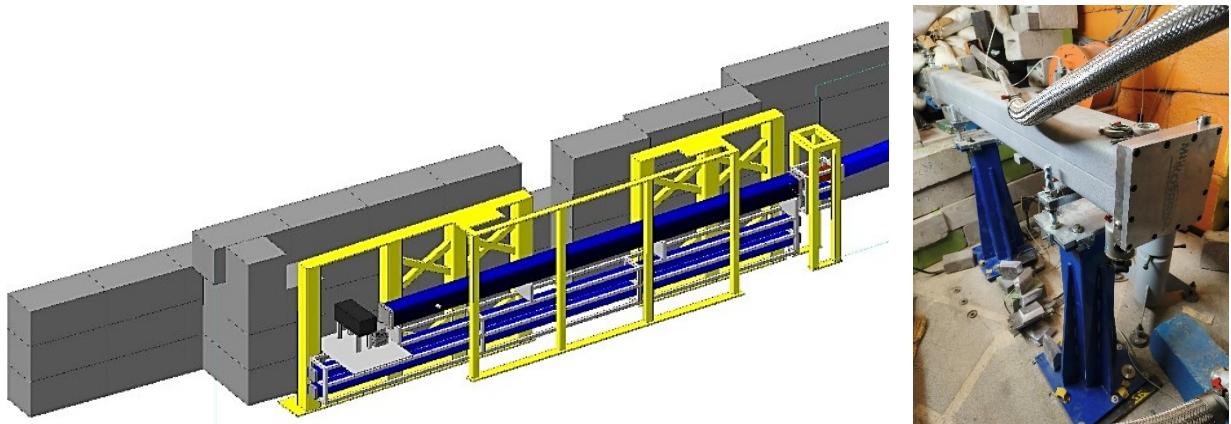
One of the possibilities for varying contrast in neutron radiography is the dependence of the elastic scattering cross-section on the neutron wavelength. For crystalline materials, due to diffraction at some wavelength values, jumps in the total cross-section are observed, which are typically strongest at the maximum interplanar spacing for a given substance. Such jumps typically occur at wavelengths from 4 to 5 Å. It should be noted that for fast neutrons with energies of the order of eVs, the energy dependence exhibits narrow absorption resonances, which can be used to achieve contrast in neutron images.

To improve contrast in neutron radiographic experiments, a neutron beam is required that is characterized by a spectral redistribution of intensity in the region of cold neutrons, which is a feature of beamline 10a of the IBR-2 reactor.

#### 4. Current status of the project and proposals for the construction of the instrument

#### 4.1. Information about the current status of the project

In the framework of the project of a new neutron radiography station with the “cold” neutron spectrum, it is planned to place the equipment on beamline 10 of the IBR-2 reactor. At present, beamline 10a is equipped with a special optical device—a neutron bender, at the exit of which a squared neutron beam with dimensions of  $60 \times 60$  mm is formed with a spectral distribution shifted towards the region of cold neutrons. Along with it, it is planned to construct a new small-angle scattering instrument at the same beamline 10a. To divide experimental time between several techniques, it is proposed to install a multi-module system with a mechanical equipment changer (Fig. 41).



**Fig. 41.** Schematic representation of a multi-module setup for research using small-angle neutron scattering and neutron radiography. A mechanical system for changing the corresponding set of experimental and methodological equipment is shown. One of the modules is a system for neutron radiography and tomography at the cold neutron source.

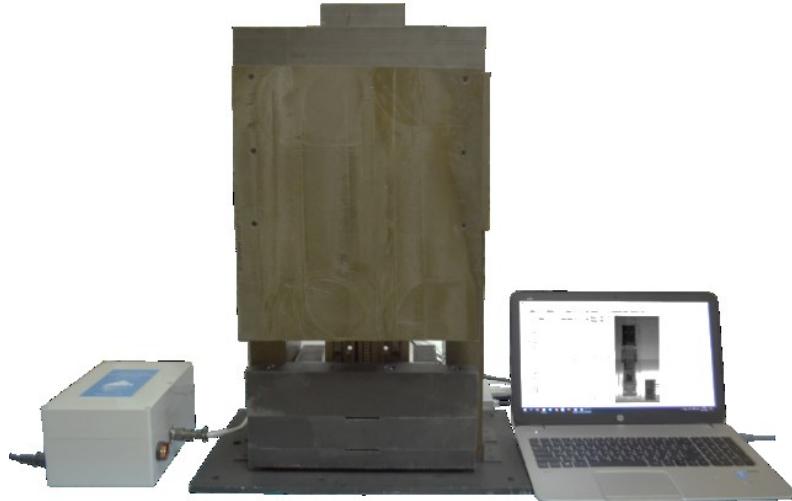
The implementation of the project of a new experimental neutron radiography and tomography station on beamline 10a requires design work, purchase of equipment and its installation on a movable module.

It is suggested that the neutron beam is formed by a neutron bender with initial dimensions of  $60 \times 60$  mm, and then, using a 10-m long collimation system consisting of circular cylindrical inserts made of borated polyethylene, it expands up to 120 mm at the exit of the neutron beam from the collimation system. The collimation system will be placed in a vacuum casing to reduce losses in intensity due to neutron scattering in the air.

As a detection system, it is intended to use a detector module with replaceable CCD video cameras of various types. The conversion of neutrons into photons will be performed using scintillator plates of various types (from standard  $^6\text{LiF}/\text{ZnS}$  to gadox  $\text{Gd}_2\text{O}_2\text{S}$ ). The light from the scintillator is reflected from rotating mirrors tilted at an angle of  $45^\circ$  with respect to the axis of the incident neutron beam, and hits the optical system of the video camera. All optical systems of the detector will be in a light-shielding casing. Tomography experiments will be performed using a system of goniometers with a minimum rotation angle of down to  $0.01^\circ$  and a remote control system.

A further development of the energy-dispersive neutron radiography method involves the use of a specialized neutron detector based on a scanning scintillation array made of  $^6\text{LiF}/\text{ZnS}$  (Fig. 42). The array size is  $5 \times 100 \text{ mm}^2$ . To form a neutron image, two independent scintillation arrays vertically scan the space in 1-mm increments.

The obtained neutron images will be corrected for background noise of the detector system and calibrated to the incident neutron beam using the ImageJ software package. Tomographic reconstruction of the studied object from a set of angular projections will be performed using the H-PITRE software. For visualization and analysis of the obtained 3D data, it is planned to use the VGStudio MAX 2.2 software package from Volume Graphics (Heidelberg, Germany).



**Fig. 42.** Photograph of the detector system for implementing energy-dispersive neutron radiography.

#### **4.2. Detailed description of proposals for construction of the instrument**

- To implement the energy-dispersive neutron radiography mode on beamline 10a, it is planned to install a three-stage multi-module setup for small-angle neutron scattering and neutron radiography.
- Alignment of the vacuum collimator system and the detector system based on the Andor Neo high-speed camera.
- Conducting a series of test experiments to determine the basic parameters of the new setup.
- Development of the necessary software for automation of neutron energy-dispersive experiments.
- Purchase and installation of a vacuum pumping system for the collimator system for the neutron energy-dispersive radiography mode.

#### **4.3. Current technical parameters of the instrument in thermal and cold modes of the moderator**

A new mode for experiments on neutron radiography and tomography will be realized on beamline 10, where a cold moderator operates. This will increase the incident flux of cold neutrons in the wavelength range of 4-13 Å by a factor of  $\sim 7$ -10.

**4.4. Relevance of the instrument construction for the concept of a suite of spectrometers  
within the project of a new neutron source at JINR**

At the new neutron source of FLNP, it is planned to construct several stations for neutron radiography and tomography investigations, including a station for energy-selective neutron imaging. Thus, the methodological and scientific development of the research direction of neutron radiography and tomography is one of the promising and relevant research areas for the future FLNP scientific program. The construction of a neutron radiography station at a cold neutron source will allow conducting standard classical experiments on neutron radiography and tomography with high radiographic contrast. The redistribution of the spectrum towards the region of slower neutrons, as well as improved background conditions for the sensitive optical components of cameras will make it possible to use a wider range of technical solutions in the field of recording neutron images: from simple CCD cameras to highly sensitive cameras based on sCMOS sensors. Experiments with detectors based on Timepix and Medipix chips will also be possible. In this case, the method of energy-selective neutron imaging with low-background conditions will be implemented.

**5. Expected scientific results, comparison with the world level**

The instrument on radiography of cold neutrons equipped with high-speed recording systems will enable research into fast processes: water absorption by materials, solidification and melting, and dissolution in liquids. Furthermore, opportunities are opening up for varying radiographic contrast by segmenting the energy of detected neutrons. This will enable the study of alloys and welds, multi-component structures, and metal products. The development of energy-dispersive neutron tomography methods and the creation of the corresponding experimental facilities is one of the top-priority tasks at leading pulsed neutron sources. The proposed instrument will make it possible to carry out similar experiments comparable to those conducted at other neutron centers. This instrument will complement the operating NRT setup on radiography and tomography with thermal neutrons.

**6. Requested resources, costs and time frames of the instrument construction**

The costs for the design and development stages of the experimental station are presented in **Table 17 (Section 4)**.

**Plan-scheme of activities for the modernization of the DN-6 diffractometer**

<b>№</b>	<b>Description of activities</b>	<b>2026</b>	<b>2027</b>	<b>2028</b>	<b>2029</b>	<b>2030</b>
1	Design and manufacture of individual components and their arrangement on the platform of the module change mechanism					
2	Purchase of tools and special fastening devices for fixing and aligning the three-section module					
3	Purchase of neutron scintillators					
4	Development and purchase of specialized software for energy-dispersive neutron radiography					
5	Purchase of concrete blocks for biological shielding between beamlines 10a and 10b					
6	Purchase of forevacuum pumps and accessories					

## References

1. D.P. Kozlenko, S.E. Kichanov, E.V. Lukin, A.V. Rutkauskas, G.D. Bokuchava, B.N. Savenko, A.V. Pakhnevich, A.Yu. Rozanov “Neutron Radiography Facility at IBR-2 High Flux Pulsed Reactor: First Results” Physics Procedia, 69, 87-91 (2015).
2. D.P. Kozlenko, S.E. Kichanov, E.V. Lukin, A.V. Rutkauskas, A.V. Belushkin, G.D. Bokuchava, B.N. Savenko “Neutron radiography and tomography facility at IBR-2 reactor”, Physics of Particles and Nuclei Letters, 13, 3, 346-351 (2016).
3. E.V. Lukin, D.P. Kozlenko, S.E. Kichanov, A.V. Rutkauskas, G.D. Bokuchava, B.N. Savenko “First attempts on energy-selective neutron imaging at IBR-2”, Physics Procedia, 69, 271-274 (2015).
4. S. E. Kichanov, D. P. Kozlenko, E. V. Lukin, A. V. Rutkauskas, E. A. Krasavin, A. Y. Rozanov, B. N. Savenko “A neutron tomography study of the Seymchan pallasite”, Meteoritics & Planetary Science, 53, 10, 2155-2164 (2018).
5. M.G. Abramson, I.A. Saprykina, S.E. Kichanov, D.P. Kozlenko, K.M. Nazarov “A Study of the Chemical Composition of the 3rd Century AD Bosporan Billon Staters by XRF-Analysis, Neutron Tomography and Diffraction”, Journal of Surface Investigation: X-ray, Synchrotron and Neutron Techniques, 12, 1, 114-117 (2018).
6. S.E. Kichanov, K.M. Nazarov, D.P. Kozlenko, I.A. Saprykina, E.V. Lukin, B.N. Savenko, “Analysis of the internal structure of ancient copper coins by neutron tomography”, Journal of Surface Investigation: X-ray, Synchrotron and Neutron Techniques, 11, 3, 585-589 (2017).

#### 4. REQUESTED FINANCIAL RESOURCES FOR PROPOSALS

The tables below summarize the cost (in k\$) and desired periods of manufacturing (purchase) of units and equipment necessary for the implementation of the projects presented in Sections 2 - 3.

**Table 1. HRFD - High-Resolution Fourier Diffractometer**

Description	Cost, k\$	Realization period	Notes
Shaft cryostat	500	2026-2028	Manufacturing in FLNP
Fourier chopper	500	2026-2028	Purchase
Infrastructure	50	2026-2030	Purchase and installation
<b>TOTAL: 1 050 k\$</b>			

**Table 2. FSD - Fourier Stress Diffractometer**

Description	Cost, k\$	Realization period	Notes
Backscattering scintillation detectors	350	2026-2030	Manufacturing in FLNP
Mirror neutron guide	240	2026-2030	Order
<b>TOTAL: 590 k\$</b>			

**Table 3. FSS - Fourier Diffractometer**

Description	Cost, k\$	Realization period	Notes
ZnS-based 90°-detectors	265	2026-2030	Manufacturing in FLNP
Manufacturing of new Fourier chopper	370	2026-2030	Order
Radial collimators	150	2026-2030	Order
Device for moving radial collimators	10	2026-2030	Order
Goniometer	25	2026-2030	Order
CAEN digitizer for list-mode data acquisition	30	2026-2030	Order
<b>TOTAL: 990 k\$</b>			

**Table 4. RTD - Neutron Diffractometer (Real-Time Diffraction)**

Description	Cost, k\$	Realization period	Notes
Supermirror neutron guide ( $m \approx 2$ ) with two-plane focusing	400	until 2026	Purchase
Backscattering detector system ( $\Omega_d \approx 2 \text{ sr}$ ) with electronic components and slit collimators	350	until 2029	Purchase and order in FLNP
90° ZnS scintillation detector system and electronic components	300	until 2030	Purchase and order in FLNP
High-temperature furnace (1800°C)	70	2029	Purchase
Electrochemical cell with possibility of passing high current (up to 10 A) through the sample for a temperature range of 20-900°C	20	2030	Order in FLNP
Huber three-circle goniometer with refrigerator (down to 4 K)	120	2029	Purchase
Mini-furnace for Huber three-circle goniometer	30	2030	Purchase
Refrigerator with shaft sample loading for a temperature range of 8 - 290 K	70	2030	Purchase and manufacturing in FLNP
Cryostat with possibility of producing a magnetic field on the sample	100	2029	Purchase
Add-on device for generating a magnetic field from 0 to 2 T	30	2029	Purchase and order in FLNP
Modernization of electronics and software	30	2028	Order in FLNP
Radiation shielding screen with Pb glass "TISSA-RP" for beamlines 5-6	3	2029	Purchase
Infrastructure	30	2027-2029	Purchase and installation
<b>TOTAL: 1553 k\$</b>			

**Table 5. DN-6 - Neutron Diffractometer for Ultrahigh-Pressure Research**

Description	Cost, k\$	Realization period	Notes
Set of high-pressure diamond-anvil cells	130	2026-2030	Purchase
Equipment for preparing high-pressure diamond-anvil cells for experiments: gasket drilling machine, gas charging machine for cells, sets of diamond anvils, gaskets, and carbide supports	50	2026-2028	Purchase
Development of drawings for a new type of diamond-anvil cells	10	2026	Development
Purchase of vacuum pumping stations based on spiral or forevacuum pumps and special accessories for their operation	80	2026-2027	Purchase
Sample-positioning system based on an endoscope and a video monitoring system	5	2026	Purchase
Purchase of materials for background shielding and structural components	60	2026-2028	Purchase
<b>TOTAL: 335 k\$</b>			

**Table 6. DN-12 - Neutron Diffractometer for Investigations of Microsamples at High Pressures**

Description	Cost, k\$	Realization period	Notes
Commissioning of cryogenic system for cooling simultaneously permanent magnet of up to 5 T and high-pressure cell (cryocooler system)	32	2026-2030	Development and purchase
Purchase of accessories for the cryocooler system: mechanical fasteners, system positioning device, radiation shielding, vacuum equipment and accessories, vacuum sensors	51	2026-2028	Purchase
Modernization of the detector system to increase its solid angle and install a cryocooler system. Purchase of neutron counters and DAQ electronics.	350	2026-2029	Purchase
Purchase of high-pressure chambers and accessories: sapphire anvils, carbide supports, gaskets, and tooling	135	2026-2030	Purchase
Protective casing of borated polyethylene for the upgraded detector ring	60	2026-2027	Purchase
Long working distance monocular microscope for adjusting anvils in high-pressure cells and its accessories	120	2026	Purchase
<b>TOTAL:</b>	<b>748 k\$</b>		

**Table 7. EPSILON - Neutron Diffractometer**

Description	Cost, k\$	Realization period	Notes
Development of a second detector ring for a scattering angle of 20=65°	180	2026-2028	Development and purchase
Purchase of electronics and high-voltage unit	140	2026-2027	Purchase
Design and manufacture of collimators for detector modules	40	2026-2027	Development and purchase
Design, manufacture and purchase of a furnace	91	2026-2029	Development and purchase
Design and manufacture of high-temperature press	80	2026-2030	Development and purchase
Design and manufacture of an automated sample-changing system	74	2026-2030	Development and purchase
<b>TOTAL:</b>	<b>605 k\$</b>		

**Table 8. SKAT - Texture Diffractometer**

Description	Cost, k\$	Realization period	Notes
Helium neutron detectors, 20 pcs	80	until 2027	Purchase
Manufacturing of Soller collimators	80	until 2028	Manufacturing
CAEN high-voltage units	60	until 2027	Purchase
Electronics and computing equipment	20	until 2027	Purchase
<b>TOTAL:</b>	<b>240 k\$</b>		

**Table 9. YuMO - Small-Angle Neutron Scattering Instrument**

Description	Cost, k\$	Realization period	Notes
Development and construction of 2D direct-beam detector based on solid-state converter	35	2025-2030	Development and purchase
Putting into service of a large-area PSD, including infrastructure components	75	2025-2028	Development and purchase
Development of algorithms and writing code for primary processing of data and processing of anisometric patterns from PSD	15	2025-2028	Development and purchase
Development and construction of a new PSD	250	2025-2030	Development and purchase
Optimization of collimation and detector systems	90	2025-2030	Development and purchase
Modernization of spectrometer units and sample environment system	185	2025-2030	Development and purchase
<b>TOTAL: 650 k\$</b>			

**Table 10. GRAINS - Neutron Reflectometer with Horizontal Sample Plane**

Description	Cost, k\$	Realization period	Notes
Drum-type chopper	100	2026-2028	Purchase
Analyzer unit	100	2026-2028	Purchase
Detector system	50	2026-2028	Purchase
Magnetic system	50	2026-2029	Purchase
Sample environment system	50	2026-2029	Development and purchase
<b>TOTAL: 350 k\$</b>			

**Table 11. REFLEX - Reflectometer with Polarized Neutrons**

Description	Cost, k\$	Realization period	Notes
Background shielding for the neutron guide	30	2026-2027	Development and purchase
Mechanical chopper	25	2026-2027	Purchase
Automation system	15	2026-2027	Purchase
Magnetic system for the sample	40	2028-2029	Development and purchase
Cryogenic system for the sample	100	2029-2030	Purchase
Fan polarization analyzer	120	2028-2029	Purchase
Spin-echo diffractometer	300	2026-2030	Development and purchase
<b>TOTAL: 630 k\$</b>			

**Table 12. REMUR - Reflectometer with Polarized Neutrons**

Description	Cost, k\$	Realization period	Notes
Drum-type neutron chopper	120	2027	Purchase
Closed-cycle helium cryostat	200	2027	Purchase
Pumping system	5	2026	Purchase
1. Ring forward-scattering PSD	300	2027	Manufacturing in FLNP
2. Backscattering detector	5	2027	Manufacturing in FLNP
Neutron polarizer	110	2027	Purchase
Modernization of electromagnet	110	2026	
Development of a stand with an oscillating magnetic field	110	2026	
<b>TOTAL: 960 k\$</b>			

**Table 13. NRT - Neutron Radiography and Tomography Station**

Description	Cost, k\$	Realization period	Notes
New high-sensitivity camera for detector system	75	2026	Purchase
Compact detector system for neutron radiography	85	2026	Purchase
Additional optical components: objective lens, extension rings, new types of scintillators	90	2026-2030	Purchase
Design and realization of an automated sample-changing system for long-term experiments	10	2026	Development and purchase
Purchase of stepper motors, structural components, control electronics for automated sample-changing system	60	2027-2030	Purchase
<b>TOTAL:</b>	<b>320 k\$</b>		

**Table 14. NERA – Inverse Geometry Spectrometer**

Description	Cost, k\$	Realization period	Notes
Nitrogen storage tank	~50	2027-2028	Purchase
Automatic liquid nitrogen filling system for beryllium filters (consumables)	~5	2028-2029	Development and purchase
<b>TOTAL:</b>	<b>55 k\$</b>		

**Table 15. BJJ - Inverse-Geometry Inelastic Neutron Scattering Spectrometer**

Description	Cost, k\$	Realization period	Notes
Neutron guide with casings	2500	2026-2028	Purchase
Analyzer (graphite plates)	200	2026-2027	Purchase
Vacuum tank of secondary spectrometer	600	2027-2027	Development and purchase
Sample environment system	200	2029-2030	Development and purchase
<b>TOTAL:</b>	<b>3500 k\$</b>		

**Table 16. SANSARA - Small-Angle Neutron Scattering Instrument**

Description	Cost, k\$	Realization period	Notes
Detector system	200	2026-2028	
Sample assembly (SANS mode), including thermostating	200	2026-2028	Purchase
Electromagnet (SANS mode)	150	2026-2028	
Control system	100	2026-2028	Development and purchase
<b>TOTAL:</b>	<b>650 k\$</b>		

**Table 17. NRT\_COLD - Neutron Radiography and Tomography Station with Cold Neutrons on beamline 10a**

Description	Cost, k\$	Realization period	Notes
Design and manufacture of individual components and their arrangement on the platform of the module change mechanism	8	2026-2027	Design
Purchase of tools and special fastening devices for fixing and aligning the three-section module	7	2026-2027	Purchase
Purchase of neutron scintillators	30	2026-2028	Purchase
Development and purchase of specialized software for energy-dispersive neutron radiography	18	2026-2027	Development and purchase
Purchase of concrete blocks for biological shielding between beamlines 10a and 10b	30	2026-2027	Purchase
Purchase of forevacuum pumps and accessories	42	2026-2028	Purchase
<b>TOTAL:</b>	<b>135 k\$</b>		

## Summary Table of Requested Funding for Proposals

Nº	Instrument	Project title	Requested funding, k\$
1.	HRFD	High-Resolution Fourier Diffractometer	1 050
2.	FSD	Fourier Stress Diffractometer	590
3.	FSS	Fourier Diffractometer	990
4.	RTD	Neutron Diffractometer (Real-Time Diffraction)	1 553
5.	DN-6	Neutron Diffractometer for Ultrahigh-Pressure Research	335
6.	DN-12	Neutron Diffractometer for Investigations of Microsamples at High Pressures	748
7.	EPSILON	Neutron Diffractometer for Measuring Crystallographic Texture and Internal Strains	605
8.	SKAT	Neutron Diffractometer	240
9.	YuMO	Small-Angle Neutron Scattering Instrument	650
10.	GRAINS	Neutron Reflectometer with Horizontal Sample Plane	350
11.	REFLEX	Reflectometer with Polarized Neutrons	630
12.	REMUR	Reflectometer with Polarized Neutrons	960
13.	NRT	Neutron Radiography and Tomography Station	320
14.	NERA	Inverse Geometry Spectrometer for Simultaneous Investigations of Atomic Structure and Dynamics of Condensed Matter	55
15.	BJN	Inverse-Geometry Inelastic Neutron Scattering Spectrometer	3 500
16.	SANSARA	Small-Angle Neutron Scattering Instrument	650
17.	NRT_COLD	Neutron Radiography and Tomography Station with Cold Neutrons	135

**TOTAL: 13 361 k\$**