

Neutron time-of-flight reflectometer GRAINS at the IBR-2 reactor: status and prospects

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DOI: 10.63907/ansa.v2i3.96
Received: 10 September 2026

Abstract

The GRAINS (GRAzing Incidence Neutron Scattering) time-of-flight neutron reflectometer with a horizontal sample plane is operating 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. Here, the current state of the GRAINS reflectometer is presented together with experimental examples for solid and liquid interfaces. The main focuses of the development of the instrument are emphasized.

1 Introduction

The development of neutron reflectometry (NR) at the pulsed IBR-2 reactor [1, 2] of the Joint Institute for Nuclear Research (Dubna, Russia) in the area of interfaces in soft matter is fully consistent with modern world trends [3]. 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.

The GRAINS (GRAzing Incidence Neutron Scattering) time-of-flight neutron reflectometer with a horizontal sample plane is operating at beamline 10-B of the IBR-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 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 [2] 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 covers various areas related to the physics of soft matter including 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, coatings including coatings for neutron optics.

The GRAINS setup includes a wide configuration of modern capabilities of neutron reflectometry in horizontal geometry. First of all, the research is focused on 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 user program of the IBR-2 reactor and is available for interdisciplinary research [4–17].

Here, we report the current state of the GRAINS reflectometer following its restart in 2025 after the interruption of IBR-2 operation for technical modernization in 2021–2024. Characteristics of the instrument are presented together with experimental examples for solid and liquid interfaces. The main focuses of the development of the instrument are emphasized. The accumulated experience at GRAINS is planned to be used in the development of the ARMAN Neutron Reflectometer at the WWR-K Reactor in the Institute of Nuclear Physics (Almaty, Kazakhstan) [18].

2 Current state of the instrument

2.1 Instrument configuration and technical characteristics

The design of GRAINS drew on the experience gained from developing two other successfully operating reflectometers at the IBR-2 reactor (REMUR, REFLEX) with a vertical sample plane [19]. The GRAINS reflectometer implements geometry with a horizontal sample plane (vertical scattering plane), which opens up possibilities for studying interfaces with liquid media.

Schematic layout of GRAINS is given in Fig. 1. 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, which is a principal element that separates the thermal neutron beam from fast background neutrons and directs it onto the horizontally placed interface at a selected 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 $+15^\circ$ to $+180^\circ$. The reflectometer is equipped with a platform for remote change of sample positions (up to 10 positions depending on sample dimensions).

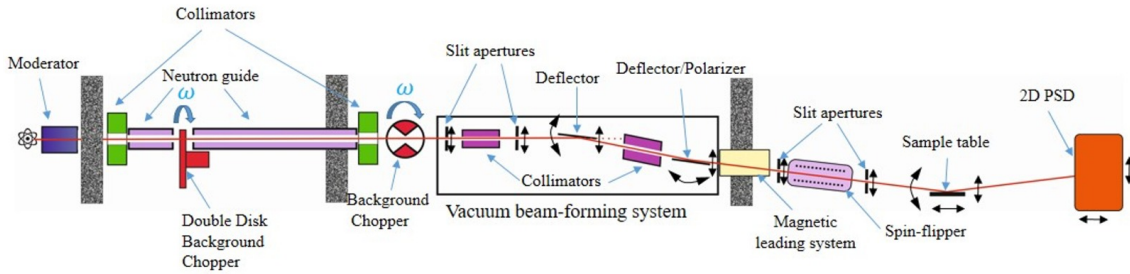


Figure 1: Schematic layout of the GRAINS reflectometer, current configuration

Table 1: Current technical parameters of GRAINS for the thermal and cold modes of the moderator

Parameter	Value
Neutron wavelength range, nm	0.05–1.0 (cold); 0.05–0.7 (thermal)
Grazing angle, mrad	0–25
q_z interval covered, nm^{-1}	0.05–2
Angle resolution, %	2–10
Neutron flux at sample position, $\text{cm}^{-2}\text{s}^{-1}$	$(1 \text{ (cold)} - 2 \text{ (thermal)}) \times 10^6$
Sample dimensions, cm	$(2 \times 2) - (7 \times 15)$
Deflecting mirror	Supermirror NiTi, $m = 2$, $L = 1$ m
Detectors	2D PSD, ^3He , 20×20 cm, spatial resolution 2×2 mm; 1D cylindrical counter, ^3He , $d = 18$ mm, $L = 190$ mm

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 [20, 21]). The detectors are mounted on

a movable platform placed behind the sample at a certain distance (Fig. 1); 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 [22–24]).

2.2 Data acquisition and reduction

The neutron reflectivity measurements were performed using the time-of-flight (TOF) technique. The data acquisition system was synchronized with the reactor pulse repetition frequency of 5 Hz. Neutron events were registered within a predefined time window relative to the source pulse and distributed among time-of-flight channels according to their arrival times. The spectra accumulated over successive reactor pulses were converted into neutron wavelength distributions.

Measurements were performed at a fixed grazing incidence angle for each sample, selected according to the reflectivity characteristics of the investigated structure. The incidence angle was determined from the spatial separation between the direct and specularly reflected beam positions on the detector, taking into account the experimental geometry.

The specularly reflected intensity was obtained by integrating the two-dimensional detector data over the region defined by the full width at half maximum (FWHM) of the reflected beam profile. The incident neutron spectrum and the background were measured separately at the beginning of each reactor operating cycle, with control measurements performed at the end of the cycle. The background was recorded with the incident neutron beam intercepted by a cadmium shutter. Background subtraction was performed using the corresponding detector region, taking into account the acquisition times of the respective measurements.

The neutron reflectivity was calculated by normalizing the background-corrected reflected intensity to the incident neutron intensity, accounting for differences in acquisition time. The resulting reflectivity curves were expressed as functions of the normal component of the momentum transfer

$$Q_z = \frac{4\pi \sin \theta}{\lambda}, \quad (1)$$

where θ is the grazing incidence angle and λ is the neutron wavelength.

3 Experimental results

3.1 Multilayers for imitating roughness modulation in neutron supermirrors

Aperiodic neutron supermirrors are nowadays used at many neutron scattering facilities. They consist of multilayer structures composed of bilayers of two materials with high (e.g. Ni) and low (e.g. Ti) optical potentials. Control of the interface roughness is of particular importance in the fabrication of supermirrors on extended substrates by magnetron deposition using an aperiodic multilayer design [25]. There is a general problem how to characterize and control the roughness modulation in a multiparameter system with a large number of inner interfaces. The samples were

produced (NRC KI – PNPI), by dc magnetron sputtering on float glass (FG). Several series of multilayered structures based on $\text{Ni}_{0.9}\text{Mo}_{0.1}/\text{Ti}$ bilayers (hereafter, the $\text{Ni}_{0.9}\text{Mo}_{0.1}$ alloy is denoted as NiMo.) were prepared for NR experiments in the specular mode. The NiMo alloy was used to avoid magnetic scattering of neutrons.

At the first stage, the series of periodic and aperiodic structures NiMo/Ti_N were compared. Periodic structures were a series of samples $\text{FG}/[\text{Ti}_5\text{nmNiMo}_5\text{nm}]_N$ with $N = 1 - 5$. Aperiodic structures comprised a series of samples

$$\text{FG}/[\text{Ti}(h_1)/\text{NiMo}(h_1)/\text{Ti}(h_2)/\text{NiMo}(h_2)/\dots/\text{Ti}(h_N)/\text{NiMo}(h_N)], \quad (2)$$

with $N = 1 - 6$ and $h_m = (4 + m)$ nm. The typical evolution of the neutron reflectivity curves obtained at GRAINS for the two series is shown in Fig. 2. In both cases, the first peak is well described by the nominal structure using the mean values of the structural parameters. At higher q_z values, the reflectivity becomes increasingly sensitive to the distributions of these parameters. At $N = 5$, the reflectivity also becomes sensitive to the interlayer correlation function. The experiment was aimed at testing the applicability of specular neutron reflectometry to the step-by-step characterization of model supermirror structures during successive deposition stages.

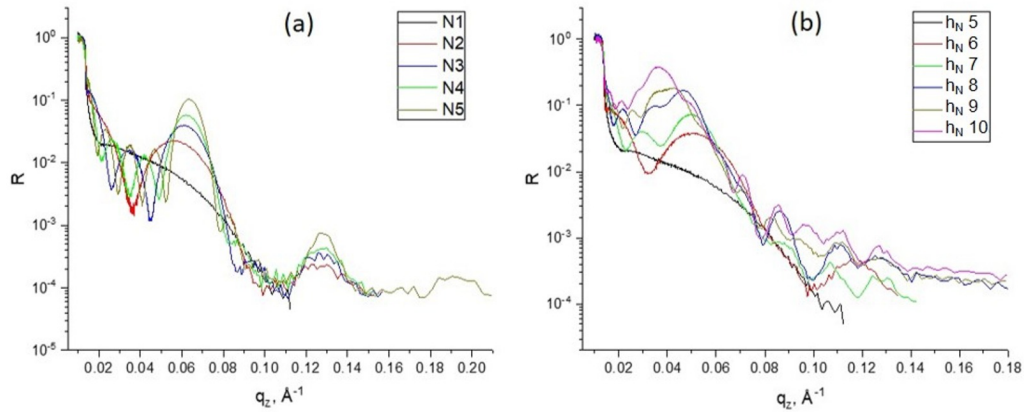


Figure 2: Evolution of experimental neutron reflectivity curves for periodic (a) and aperiodic (b) multilayers with varied number of bilayers, N , indicated in the legends

3.2 Adsorption of phospholipid transport vesicles on lipid membranes at planar solid interface

A phospholipid transport nanosystem (PhTNS) represents comparatively small aggregates (size of about 50 nm) and consists of soy phospholipids [26]. The use of PhTNS as a delivery system leads to an increase in the effectiveness of medicinal compounds. For improving PhTNS-based drugs, the study of the process of the penetration of these systems into membranes is of great importance. Studies of the structure of a planar phospholipid bilayer deposited on a solid-water interface using neutron reflectometry have shown high efficiency of this method both for studying the structure of the phospholipid lipid bilayer and for studying the kinetics of its formation in situ [27]. Time-of-flight neutron reflectometry is most effective for such studies. A specialized liquid cell was used in the NR experiments at the GRAINS

reflectometer. The washing function allowed the removal of particles that are not settled on the substrate, thus fixing the settled bilayer. A crystal with a nickel ‘support’ layer was used as a substrate (Si//Ni 50 nm/Si 10 nm). A bilayer of soybean phospholipid S100 was first considered as a model membrane deposited from the 0.1% phospholipid solution in heavy water. To study the permeation of the drug incorporated into the PhTNS, solutions of 0.5 and 5% doxolip were prepared, where doxolip is a PhTNS system containing doxorubicin. The PhTNS was prepared using soy phospholipid S100 and maltose in a 1:4 ratio. Experiments were also conducted to deposit a doxolip bilayer onto the substrate.

In the first kind of experiments, the surface was in contact with the solution of S100 for 0.3 h. Then, the cell was refilled three times with D_2O . The formation of a bilayer of soy phospholipid (S100) on the substrate was detected after such kind of washing (Fig. 3 a). The same procedure was repeated for the 0.5% PhTNS solution above the formed bilayer of S100. The deposition of the additional bilayer from PhTNS above the formed S100 bilayer after 0.3 h was not detected. So, the cell was again refilled with 0.5% PhTNS solution above the formed bilayer of S100, and measurements of reflectivity were made within one day after the refilling. Slight changes in the initial curve from the S100 bilayer were detectable after 24 h only (Fig. 3 a). In the second kind of experiments, the deposition of the bilayer from 5% PhTNS in heavy water in the direct contact (no S100 bilayer) with the same substrate was followed during 24 h (Fig. 3 b). A bilayer is formed during the first 12 h and then changes slightly in time (Fig. 3 b). A slight difference between S100 and doxolip bilayers is observed. A possible explanation of the effects observed is that the interaction of doxolip and the bilayer was hindered by the presence of maltose in the solution. Further steps will include analyzing the interaction of pure doxorubicin with the bilayer, using a DMPC system as the bilayer. An important result is the formation of a stable bilayer that remained intact after several washings.

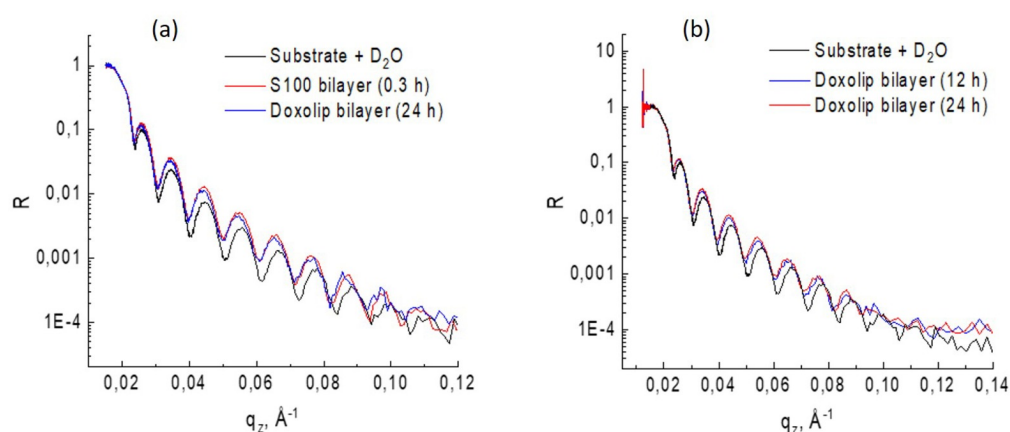


Figure 3: (a) Reflectometry curves from the substrate in contact with pure D_2O , after deposition of S100 bilayer (for 0.3 h) and after deposition of doxolip bilayer (for 24 h) above S100 bilayer. (b) Reflectometry curves from the substrate in contact with pure D_2O and direct contact with doxolip solution after 12 and 24 h.

4 Conclusions

GRAINS reflectometer has proved to be effective in studies of applied systems such as components of neutron optics (solid samples) or phospholipid transport nanosystems (liquid samples). For solid samples, 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. For solid/liquid interfaces, the research is focused on in situ experiments. 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 q_z -range using several grazing angles are limited only by strongly scattering systems due to a relatively high background. Under the current experimental conditions, the minimum experimentally accessible reflectivity is approximately 10^{-5} .

The current modernization of GRAINS is aimed at improving the quality of NR experiments. The current modernization of GRAINS is aimed at improving the quality of NR experiments, with particular emphasis on expanding the capabilities for interdisciplinary studies. The development of the 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.). The plan is 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 $10 - 200^\circ\text{C}$ 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 organizing the grinding and redeposition of planar structures on the crystalline substrate blocks after the experiment.

Within the development of the scientific program, the research is planned in various areas of solid/soft interfaces including layered phospholipid structures, polymer brushes and surface coatings, composite catalyst films, phase separation in mixed liquids, multilayered structures of a special design.

Acknowledgments

This work was supported by a grant from the Plenipotentiary Representative of the Republic of Kazakhstan at JINR, Russia (p.11, No.111 from 03.02.2026).

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