INTEREST

Wave 5 (27 September - 05 November, 2021)

Report

about the course

«Introduction to neutron scattering experiments at large scale facilities»

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Abstract

It is a report of the participation in the lecture courses in the framework of INTEREST University Centre JINR course program. The course was devoted to the introduction and basics of neutron and x-rays scattering experiments at large scale facilities. The international neutron sources with different types of neutron capabilities and working laboratories of experimental facilities were reviewed. The experimental tasks were using VITESS software package. VITESS is a tool for simulation of neutron scattering instruments and experiments at pulsed and continuous sources. Using VITESS, you can simulate a large variety of instruments at all major current and future neutron sources [1].

1.Theoretical basis

1.1. Small angle neutron scattering

The interaction of incident neutrons with the surface of the sample results to scattering on three main channels: specular reflection, non-specular scattering (off-specular/diffuse scattering) and small-angle neutron scattering during sliding incidence = GISANS (grazing incident small angle neutron scattering).[2] Scattering is shown only in the corresponding central panes to simplify the drawing.

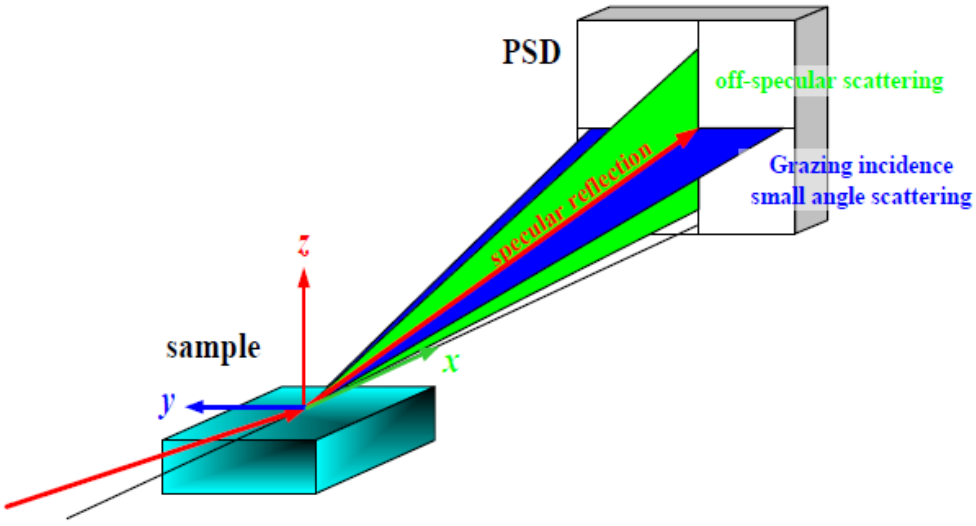


Fig.1. Tree channels of scattering [3]

If we base only from the name «grazing incident small angle neutron scattering», that by this term we can understand all scattering channels. The term GISANS is used for scattering, additional to specular and non-specular scattering. The more intense channels of specular and off-specular scattering are often simply blocked with an absorber to unload the detector and reduce electronic noise. The intensity of the specular reflection is mostly determined by the effective potential V(z) averaged by the lateral coordinates (x and y). The cross-sections of off-specular and GISANS are also determined by the features of the lateral structure mainly. Full measurements are made using a position-sensitive detector (PSD).

1.2. Neutron scattering – GISANS experimental method

SANS is an effective method for studying fundamental problems and for solving the most important technological problems is widely used in studies of the supratomic structure of matter. The most important feature of SANS is the ability to analyze the structure of disordered systems. This method is often the only one way to obtain structural information about systems with chaotic and partially ordered arrangement of density inhomogeneities. It makes possible to study the dispersed structure of alloys, powders, glasses, structural features of polymers in various aggregate states, weight and geometric characteristics of biological macromolecules and their complexes, biological supramolecular structures such as biological membranes and viruses.[4] The significant difference in the neutron scattering lengths on hydrogen and deuterium, as well as the possibility of selective deuteration of macromolecules and supramolecular structures, makes small-angle neutron scattering an indispensable tool for the study of biological, colloidal objects, as well as polymers and liquid crystals. Small-angle neutron scattering has a number of important features that distinguish it from small-angle X-ray scattering. This is primarily determined by the general features of the interaction of thermal neutrons with matter: a large penetration depth into the substance, the dependence of scattering on the isotopic composition of the substance and on its magnetic properties, which makes SANS an indispensable method for studying the structure of matter.

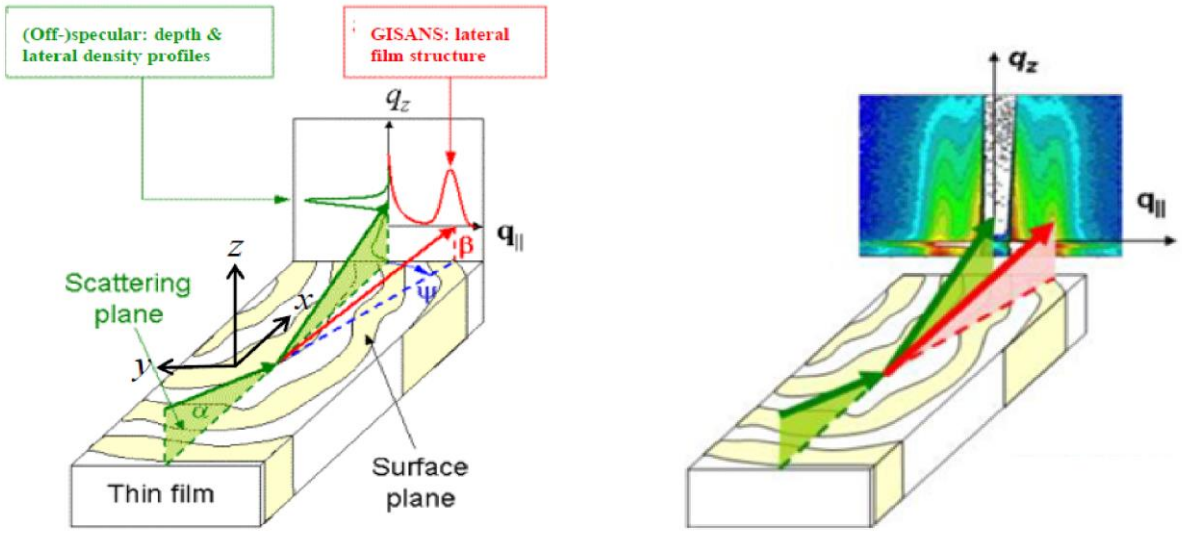


Fig.2 Schematic representation of GISANS method [3]

1.3 Neutron reflectometry introduction

The method of neutron reflectometry consists in measuring the neutron reflection coefficient as a function of the transmitted neutron pulse. The structure of the sample is determined by fitting an experimental curve.

$$\mathbf{k}_1 = (k \cdot cos\alpha, 0, -k \cdot sin\alpha)$$

$$\mathbf{k}_2 = (k \cdot cos\beta \cdot cos\psi, k \cdot cos\beta \cdot sin\psi, k \cdot sin\beta)$$

$$\boldsymbol{q} = \mathbf{k}_2 - \mathbf{k}_1 = (q_x, q_y, q_z) = k(cos\beta \cdot cos\psi - cos\alpha, cos\beta \cdot sin\psi, sin\beta + sin\alpha)$$

$$= k\left(-\frac{1}{2}(\beta^2 - \alpha^2 + \psi^2), \psi, \beta + \alpha\right)$$

$$= (-\frac{1}{2}\,(\beta - \alpha)q_z - \frac{1}{2}\psi\frac{\psi}{\beta + \alpha}q_z, \frac{\psi}{\beta + \alpha}q_z, q_z)$$

α - the angle between $\mathbf{k}_1$ and the reflecting surface

β - the angle between $\mathbf{k}_2$ and the reflecting surface

ψ - the angle between $\mathbf{k}_2$ and the surface of mirror reflection, the x and y axes are on the surface of the sample, and $\mathbf{k}_{1,y}$ =0, the z axis is normal to the surface of the sample.

q – the wave vector transfer or "scattering vector", ħ**q** – momentum transfer.

$q_z \cong k(\alpha + \beta)$ - approximation of small angles. [5],[6]

1.4. Neutron reflectometry experimental facilities

Neutron reflectometry needs reflection of neutrons with a momentum transfer in the range from 0.001-1 $Å^{-1}$. There are two ways to obtain the necessary values of the momentum transfer: the first is to carry out measurements by very cold neutrons at large angles of incidence, the second is to use neutrons with wavelengths in the few angstroms range at small angles of incidence of the neutron beam (~ 0.1° -2°) with low divergence.

Neutron reflectometers can be divided into three classes:

1.Time-of-flight

2.Monochromatic

3.Combined. Facility works in time-of-flight and monochromatic mode at the same time.

Monochromatic reflectometers operate on continuous neutron sources. They provide a higher resolution on the momentum transfer than time-of-flight reflectometers. Monochromators usually made from pyrographite. It is necessary to scan the angle of incidence and scatter (θ-2θ-geometry) to measure R(Q). The typical value of the operating wavelength is 4-5 Å, i.e. close to the maximum of the reactor spectrum. Time-of-flight reflectometers work both on pulsed neutron sources and on continuous ones. In pulsed neutron sources the neutron beam is modulated by choppers. Usually scanning is carried out by wavelength, i.e. by the time of neutron flight with a stationary sample and detector.

All reflectometers have a similar structure. Their main parts:

1. Neutron guide.

Mirror neutron guides have been installed between the source and the installations for increasing the flux density of thermal and especially cold neutrons. Curved neutron guides or straight ones with a curved section are also widely used. The simplest mirror neutron guide is a pipe with polished walls made from highly scattering material. The guides were evacuated to prevent the neutrons from scattering on the air molecules, and the low divergent neutrons hitting the neutron guide walls at low grazing angles were reflected and transported more efficiently. Most materials have a refractive index that is slightly below 1 for cold and thermal neutrons. To reflect neutrons at still higher angles than that corresponding to m=1, one could use the 58Ni isotope. This has a higher neutron scattering length density compared to Ni, equivalent to m=1.18. The material with the highest neutron scattering length density in the periodic table is carbon in the form of diamond. Thereafter come Ni and Be, which have nearly the same scattering length density.

Both C and Be have a very low absorption cross section compared to Ni, and when a neutron is absorbed, both emit gamma radiation at lower photon energies than Ni. [7]

2. Collimating devices (slits).

Slits provide the necessary divergence of the beam and the dimensions corresponding to the sample. Usually, collimating devices are made with a changeable slit size.

3. Deflecting mirror.

It is used in time-of-flight reflectometers with a vertical scattering plane. Allows you to set the incidence angle of the neutron beam leaving the sample plane horizontal. The position of the mirror is set using a goniometer that moves the mirror in a vertical plane. Super mirrors or polarizing mirrors (in reflectometers with a polarized beam) are used as a deflecting mirror.

4. Polarizer and spin-flipper.

Magnetic mirrors made from ferromagnets (for example, Co) or magnetic super mirrors are used as polarizers. Magnetic super mirrors are becoming increasingly popular because they work in a wider range of momentum transfer. The spin-flipper is used to reverse the polarization of neutrons. Neutron radio frequency (RF) spin-flippers with mutually opposing currents are also used in neutron reflectometers. [8] These spin flippers provide a high degree of polarization in a wide range of neutron wavelengths.

5. Sample part.

It includes a displacement device that moves the sample in three mutually perpendicular directions and a goniometer that rotates or swings the sample in the scattering plane within a few degrees and with a precision of at least 1'.

6. Polarization analyzer.

Allows you to separate neutrons reflected with a spin flip from neutrons reflected without a spin flip. Similar to item 4. by design. Polarized 3He has been used as polarizers and analyzers. These devices provide a wide aperture, high polarization efficiency in a wide range of wavelengths and can work in beams with high divergence. However, this type of polarizers requires periodic laser pumping. Laser pumping systems are very bulky and expensive.

The mirror analyzer allows you to register neutrons reflected with a spin flip and without a flip at the same time. The detector registers neutrons reflected from the analyzer mirror and those that have passed through it. This scheme allows you to

decrease the measurement time if you need to analyze the polarization after reflection by the sample. [9]

7. Detector.

The detector is necessary for measuring the reflected neutrons and flux that determines the neutron reflection coefficient. It is necessary to measure the angular distribution of the scattered neutrons in the scattering plane to study non-mirror reflections caused by the irregular surface and axial geometry relative to the axis of the incident beam for studies of (GISANS). Two-coordinate detector is required for this case. The coordinate resolution of the detectors is 1-3 mm for each coordinate, typical efficiency of registration is about 30%.[10] Usually, the detectors are placed in a vacuum tube, inside which it can move, or the detector rotates with the tube, thus changing the viewing angle. The use of a vacuum tube helps to avoid the neutron losses due to scattering and absorption in air.

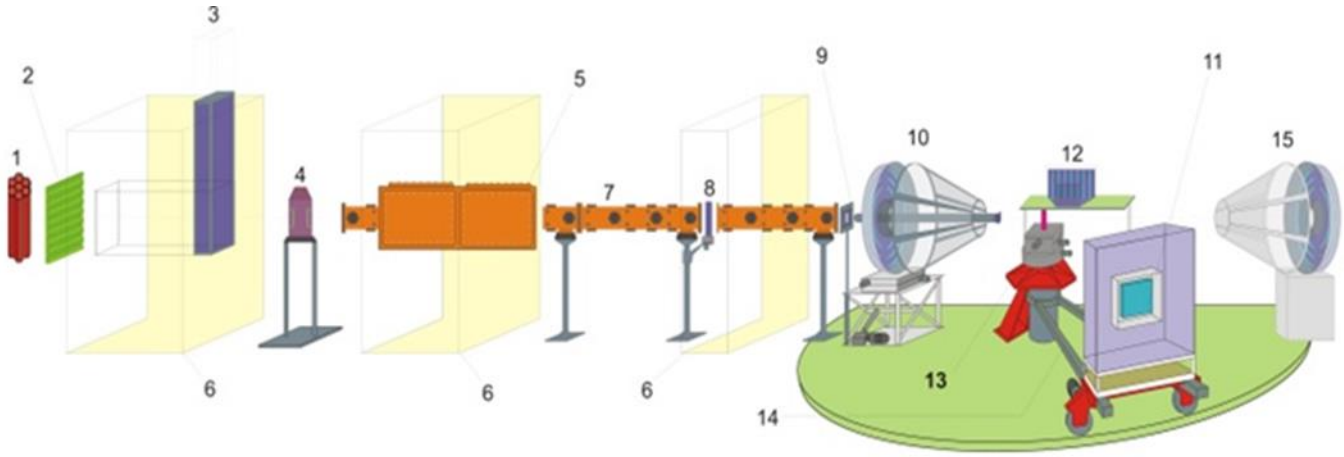


Fig.3 Typical neutron facility. Here we have: 1 – reactor core, 2 – moderator, 3 – beam shutter, 4 – chopper, 5 – beam splitter, 6 – biological shielding, 7 – mirror neutron guide, 8 – secondary beam shutter, 9 – adjustable diaphragm, 10 – backscattering 8-rings detector, 11 – position-sensitive detector, 12 – 3 banks * 8 3He “point” detectors, 13 – goniometer and sample position,14 – PSD movable arm,15 – small-angle ring detector.[11]

2. Practical task

My practical task in the course of INTEREST was to get start work in the VITESS program. VITESS is a software package for Monte Carlo stimulation of neutron experimental facilities. The virtual neutron instrument comprises of several modules, which represent instrument components like guides or detectors, various

helper modules, e.g. frames modifying the coordinate system between two instrument components, and modules for data visualization and evaluation. All modules can be selected within the GUI. When you run a simulation, such modules are co-working sequentially embedded in a pipe structure:

Each module processes and then passes neutron data to the following one.

The first module must be either a neutron source module, or it must read information about the neutron trajectories from a file typically created in an earlier simulation of the preceding part of your instrument, which includes the source module.

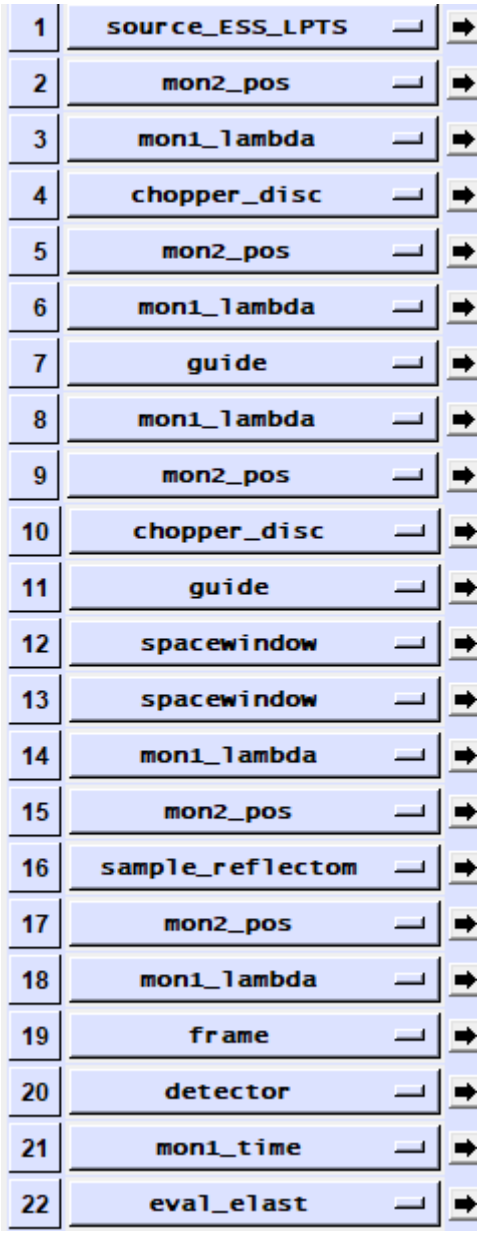
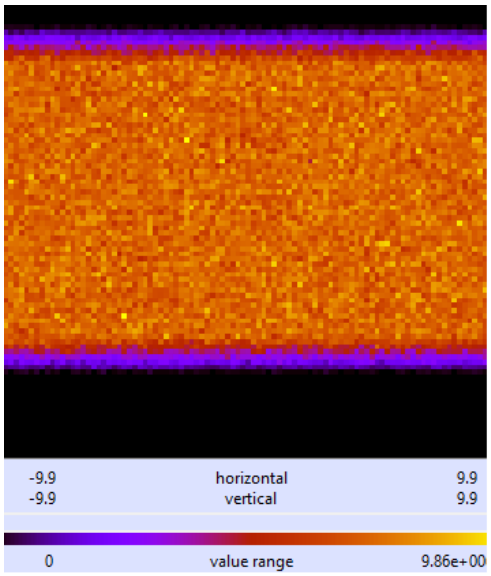


Fig.4 Modules used to simulate a neutron reflectometer in VITESS

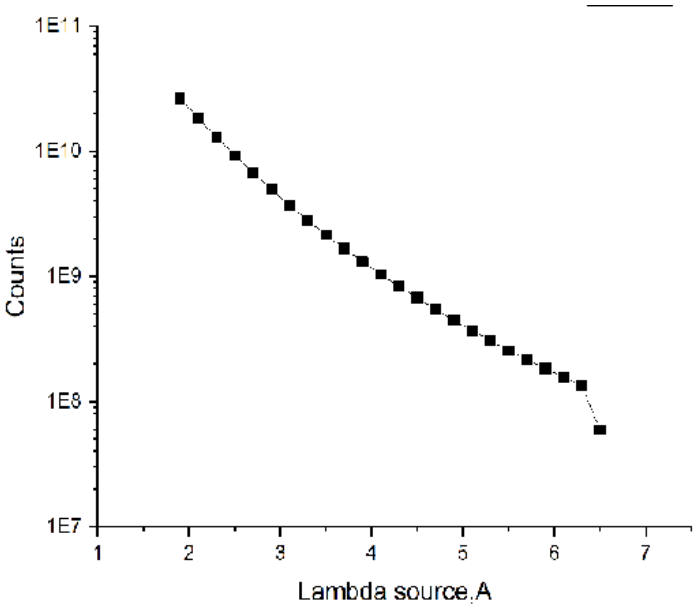
The last module may be set up to write a file of neutron trajectories. This makes sense if you wish to use these trajectories in further simulations as input, e.g., for future studies of parameter distributions of these neutrons.

My task was to simulate neutron reflectometry experimental facility. View the evolution of the beam characteristics during the transmission in different parts of the facility. As previously described, it is necessary to measure the neutron reflection coefficient as a function of the momentum transfer. **Fig. 4** shows the main parts of the neutron reflectometer. There I have position sensitive monitor and wavelength monitor after each module for studying the changes of the beam.

Fig. 5 shows the results of "measurements" of the position and wavelength monitors after the source. **Fig.5a** shows that the source has no highlighted directions and the illumination occurs almost uniformly. **Fig. 5b** shows that the maximum of flux density (10^{12} n/(cm2 s)) for the source neutrons is ~ 2 A. This value of waveleght corresponds to the energy of a thermal neutron. In general, this flux density from the moderator is normal for spallation sources, but as far as I know ESS writes about 10^{13} everywhere.

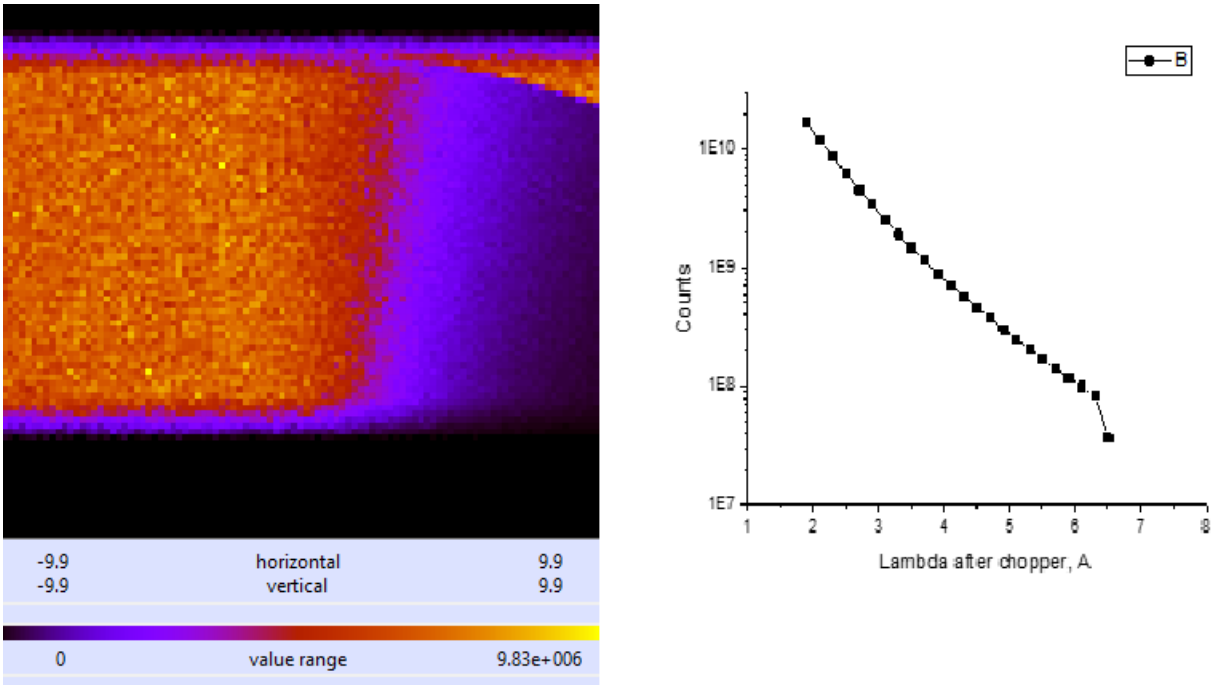


a



b

Fig.5 PS and lambda monitors after source



a b

Fig.6 PS and lambda monitors after chopper

Fig. 6 shows PS and wavelength monitors after chopper. It can be seen that part of the beam was "cut off", flux density decreased, but not so much.

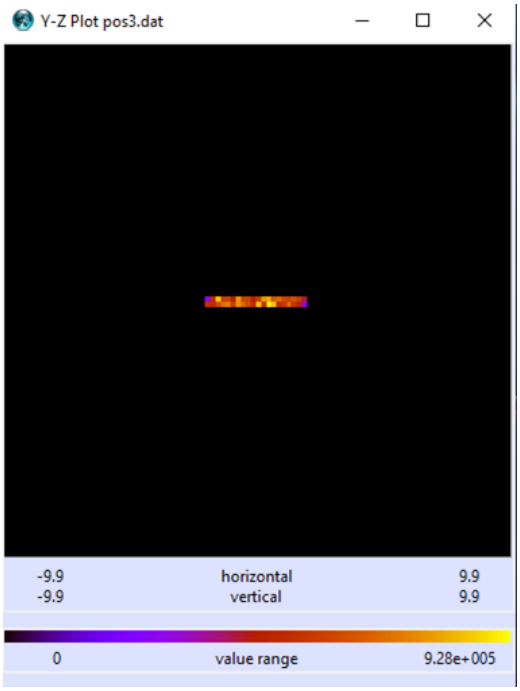


Fig.7 Profile of the beam before sample (after slit)

The measured value is the reflection coefficient as a function of the momentum transfer, which is used to determine the structure of the sample. There are databases of the reflection coefficient with which you can determine the materials of the layers in sample. **Fig. 7** shows the dependence obtained for the Si-50 Au sample in VITESS.

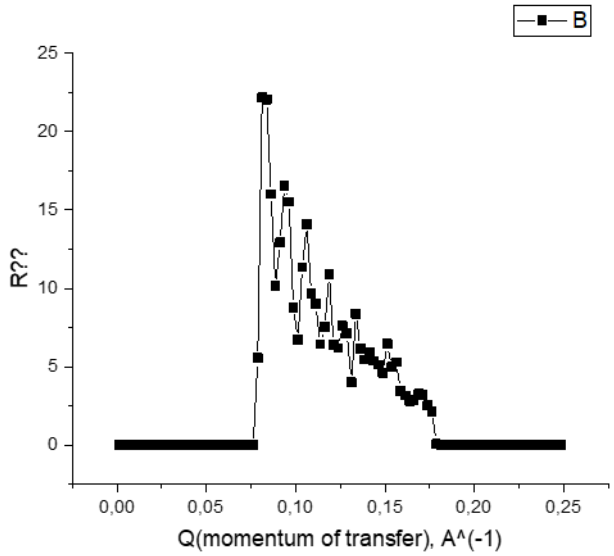


Fig. 7 Neutron reflection coefficient as a function of the transmitted neutron pulse for Si-50 Au sample

3. Proposal for instrumental modernization of GRAINS facility.

3.1. Aim of the proposed experiment and description of the scientific background

The aim of this experiment is to test a new thermal neutron detector based on RPC-detector with B_4C converter for GRAINS reflectometer. Multilayers PRC-strips detectors can have spatial resolution better than 100 μm. Betterment of spatial resolution of detectors provide to increase resolutions for momentum transfer. Also, it has efficiency of registration ~50 %. This number can be increased by adding new layers or by incline the detector. Increasing of efficiency provide to decrease data set time [12].

3.2. Results of previous own work, especially experiments at IBR (if applicable)

It will be working like modernization of GRAINS facility because B^{10} RPC-detector has a large number of advantages over the current He^3 wire-detector. He^3 is so expensive, wire-detectors have spatial resolution is about few centimeters. Also, it's

so hard to make gas-detector with large square. The requirements for the detector in this facility are to betterment the spatial resolution and increase the sensitive area.[13],[14],[15]

3.3. Details of the experiment at IBR-2

a. Measurement of the induced charge. The induced charge on the strips will be caused by the movement of the ionization charge under the action of an electric field.

b. This is new type of detector for thermal neutrons experimental facilities. Because for determinations of characteristics it is needs neutrons.

c. One measuring on this facility needs a few hours. It takes me at least 24 hours to make a series of measurements with different detector positions and different samples

d. The circulation of a standard ArCO2 gas is required for the correct operation of this detector. The gas cylinder is under high pressure. A high voltage (~12kV) supply to the detector is needed. [16]

e. The detector has no bulky parts.

3.4. Description of the results expected and their scientific relevance

It is expected to obtain a spatial resolution of better than 1 mm. Decreasing of "dead time" due to the short pulse duration provided by high voltage. Decreasing the data set time. Sensitivity to the gamma background will be obtained.[9] If the obtained characteristics are better than those of the detector existing at the Facility (He^3 2D chamber detector), then it can be used to upgrade the GRAINS.

3.5. Related own publications during the last 3 years

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3.6. Other related references

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