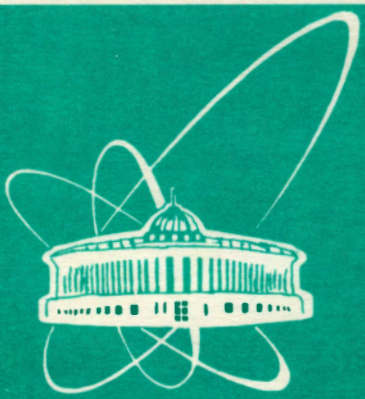

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MULTI-BEAM NEUTRON REFLECTOMETER FOR A STEADY STATE REACTOR

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1993

1 Introduction

The property of the neutron to be reflected from the interfaces of media has found broad application in experimental neutron physics. For a long time this property has been employed to form neutron beams using mirrors and neutron guides for research reactors [1,2,3]. It has been about ten years since this property was first applied to the investigation of physical properties of surfaces and thin films. Now, the method of neutron reflectometry is widely used in the physics of surfaces (see, for example, the proceedings of recent conferences dedicated to the investigation of surfaces with X-rays and neutrons [4,5,6]). In recent times, almost all leading neutron centers in the world have built or are building neutron reflectometers. Since 1989, a neutron reflectometer (SPN [7]) has been successfully operating in Dubna, and now, the construction of a two-reflectometer complex; with unpolarized (REFLEX-1) and polarized (REFLEX-2) neutrons, [8] is nearing completion.

This paper was stimulated by a discussion of the experimental program for the ET-RR-1 reactor of the Atomic Energy Authority of Egypt held under the auspices of the agreement for cooperation between AEAE and the Joint Institute for Nuclear Research (JINR). Herein is a proposal for the creation of a reflectometer for the ET-RR-1 reactor. Section 2 briefly explains the ideas and possibilities of neutron reflectometry. Section 3 outlines the details for a neutron reflectometer that could suit the particular conditions of the ET-RR-1 operation.

2 Neutron Reflectometry

Reflectometry, in the fullest sense of the word, is a combination of all methods for investigating plane interfaces by the means of analysis of the specular reflection of beams of molecules, atoms, particles or electromagnetic radiation from an investigated boundary. Under neutron reflectometry, one includes the combination of methods for investigating plane interfaces of media which have as a basis the neutron-optical phenomenon of specular reflection of low-energy neutrons ($\leq 10^{-1} eV$) with small grazing angles ($10^{-3} \div 10^{-2} rad$) incident to the interface. Neutron reflectometry (hereafter referred to as "reflectometry") is classified by investigated objects into reflectometry of non-magnetic media

and reflectometry of magnetic media. The first uses beams of unpolarized neutrons (reflectometry in the full sense), the other — beams of polarized neutrons (polarization reflectometry). With the methods of reflectometry one studies the depth profile of the neutron-optical nuclear potential, U_{nucl}, along the normal to a surface, and the objects are liquid, crystal or amorphous samples in the form of a massive plate, thin mono- or multi-layer films on a massive substrate. The polarization reflectometry investigates the behaviour over depth of the vector of local magnetization, in particular, the peculiar magnetic properties of a near-surface ($> 10\mathring{A}$) area of ferromagnetics or ideal diamagnetics — superconductors. Here, as a rule, the object is a massive plate or a thin film on a substrate.

Recently, reflectometry has been developed as one of the methods using neutron sources for investigating condensed matter. Figures 1 and 2 show the principle schemes of these two types of reflectometers. In Fig.1, a beam of thermal neutrons (n) from a source is formed by absorbing diaphragms (collimators) (1,2) and falls on the surface of a sample (3) at a grazing angle, θ, ($\sim 10^{-3}-10^{-2} rad$) with an uncertainty of $\delta\theta/\theta \sim (1\div 5)\times 10^{-2}$. The specularly reflected neutrons are registered by a neutron detector (4). In the polarization reflectometer (Fig.2) the incident beam is polarized in a neutron polarizer (1) prior to reflection. The sample (6) is placed in the gap of an electromagnetic system (5) (it may be a system of Helmholtz coils) that creates a magnetic field in the sample and also permits one to change both the field direction and/or value. The electromagnetic system performs two important functions: a) produces a magnetic field action in a sample; and b) sets up a certain direction of magnetic polarization, $\vec{P}$, of neutrons incident with respect to the surface of a sample. The latter (b) is ensured by the adiabatic behaviour of neutron spin in the guide magnetic field of the system. A special electromagnetic device — a spin-flipper — (4) changes the sign of polarization in an incident beam. A change in the angle θ is accomplished by mechanically rotating the mirror. A movable detector permits measurement of both a reflected and an incident beam. In the latter case the sample is removed from the beam by rotating it. A difference in coordinates of the detector, corresponding to the maximum of direct and reflected beams, enables the angle 2θ to be determined

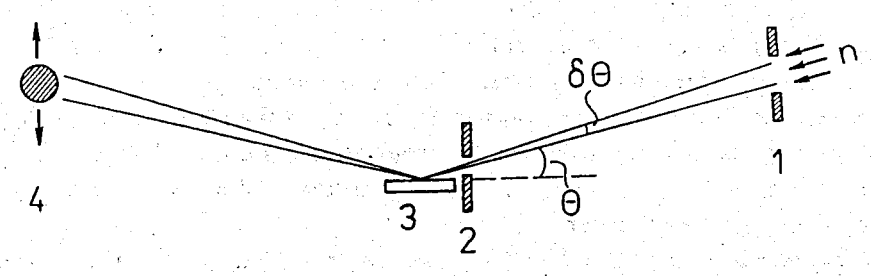

Fig. 1. The principal scheme of a neutron reflectometer. 1,2 — absorbing diaphragms, 3 — a sample, whose surface is irradiated with a narrow-collimated beam of thermal neutrons, 4 — a detector for registration of the neutrons specularly reflected from the surface of a sample. The typical distance from diaphragm 1 to the detector is 3 - 10 m.

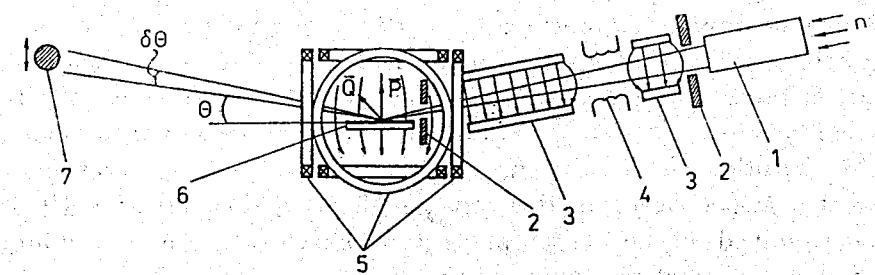

Fig. 2. The principal scheme of a polarization neutron reflectometer. 1 - a polarizer for thermal neutrons, 2 — absorbing diaphragms, 3 — permanent magnets for adiabatic guiding of neutron spin, 4 — a spin-flipper which provides, when on, the reverse of the polarization vector with respect to the leading magnetic field, 5 — a system of Helmholtz coils defining the direction of the P-vector with respect to the sample surface, 6 — a sample, 7 — a detector for registration of a specularly reflected beam.

with high accuracy. In the case of a liquid sample, θ is changed by the vertical displacement of the sample, not by rotating it. Now, reflectometers are upgraded by including one-dimensional position-sensitive neutron detectors of high resolution ($< 1mm$) in their schemes, and by the use of a multi-beam method for irradiating a sample. This method uses two or more narrow beams, having different θ angles, which are registered separately after reflection [8,9].

In reflectometry the measured data are presented in the form of the reflectivity $R(k_z)$ (see Fig.3,4) related to the measured intensity of an incident, $I_o(k_z)$, and a specularly reflected, $I(k_z)$, beam as: $R(k_z) = I(k_z)/I_0(k_z)$. Here k_z is the normal to the surface component of the wave vector of a neutron $k(k_z = ksin\theta)$. In reflectometers operating with a pulsed neutron source, the k_z scanning is accomplished, with the time-of-flight method, over the whole thermal neutron spectrum at a constant θ. For continuous beams, a monochromatic beam of neutrons is used and the k_z scanning is done by means of varying θ. Theoretical interpretation of the function $R(k_z)$ is based on the solution of a stationary quantum-mechanics problem of the reflection of a scalar plane neutron wave, $e^{ik_z z}$, from the boundary of a one-dimensional neutron-nuclear potential $U(z) = 4\pi(\hbar^2/2m)N(z)b(z)$, where $U(\infty) \sim 10^{-7}eV$ is the typical value, $N(z), b(z)$ are the local (averaged over the plane XY) densities of the scattering nuclei and their neutron scattering lengths, respectively. In this way the shape of the potential, $U(z)$, is determined by the spatial (along z) peculiarities of the density and the composition of a medium on a microscopic level (see Fig.3,4).

Reasons for the microscopic inhomogeneity of the $U(z)$ potential (assuming inhomogeneities of the order of $10 - 1000\AA$) can be reduced to surface microroughness, difference in surface and bulk density, and surface admixtures. The same reasons, together with interdiffusion, cause a smoothing of the meandering-like potentials of thin-film multi-layer structures in the vicinity of internal inter-layer boundaries. Actually, the processing of experimental data consists of minimizing the difference, $\Delta R(k_z)$, between the experimental and calculated reflectivities on the basis of a model dependence on $U(z)$ (a direct problem). To calculate a theoretical value for $R(k_z)$, numerical methods are applied to solve the stationary Schroedinger equation with a model potential

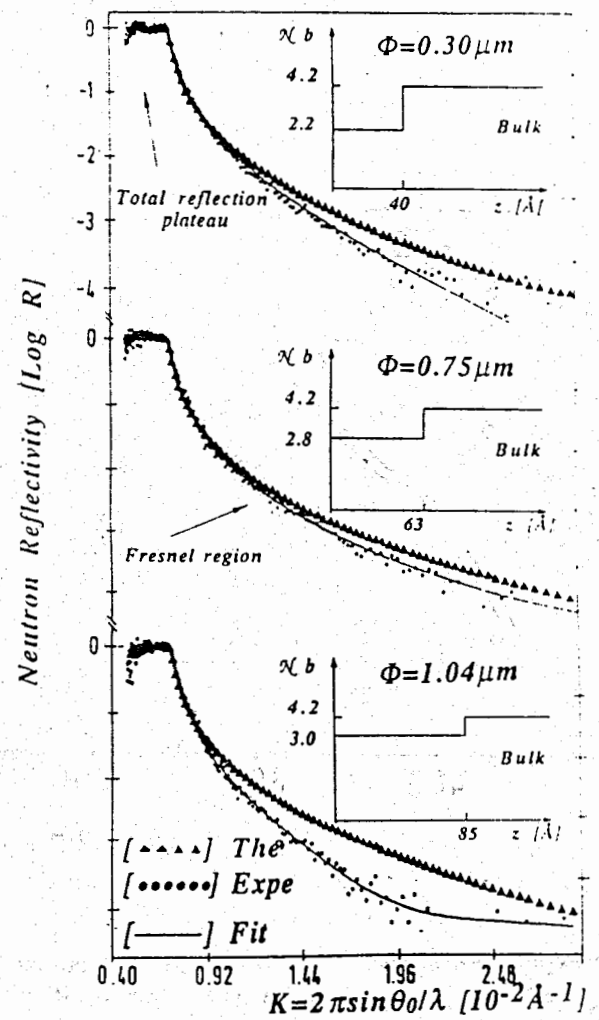

Fig. 3. Neutron reflectivity profile (see [10]) of the float-glass faces, in logarithmic scale versus the normal neutron vector component $k = 2\pi \sin\theta/\lambda$, at a constant polishing tool pressure (P = 0.5 bar) and various mean grain sizes of polishing powder Φ. The models used to simulate the reflectivity profiles are shown in the inserts.

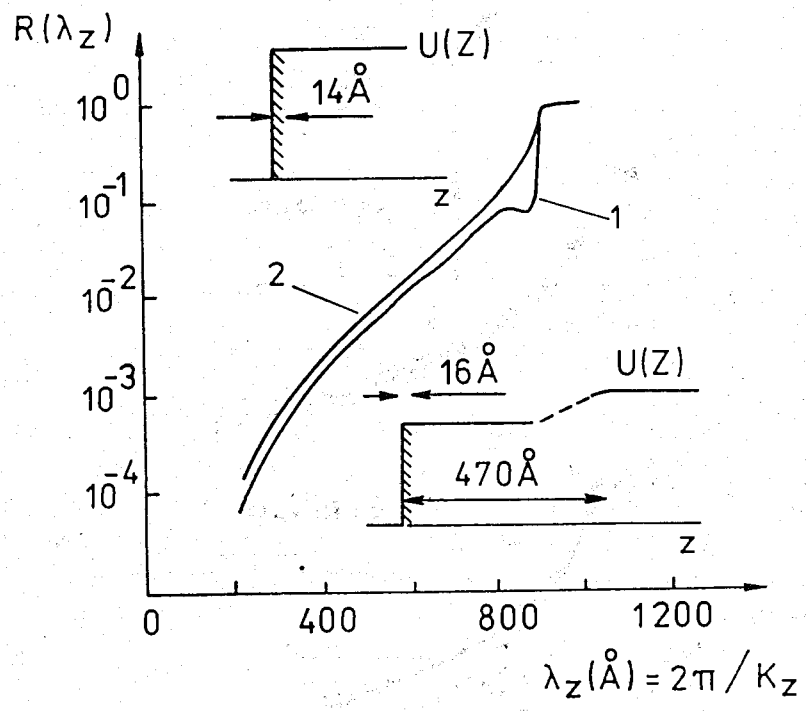

Fig. 4. The experimental (see [11]) wavelength dependence of the reflectivity $R(\lambda_z)$ ($\lambda_z = 2\pi/k_z$) from the surface of a sample (a plate of the "float-glass" type made by pouring melted glass onto liquid tin); 1, 2 — coefficients of reflection from the surfaces contigous to tin and air, respectively, during the manufacturing process. At the inserts: spatial dependences of the $U(z)$ potentials which help fit the experimental $R(\lambda_z)$ curves with the theoretical. The rough areas are shaded.

$U(z)$. In the framework of a model of semi-infinite space in the region

$$R(k_z) \ll 1 \qquad (k_z \gg \sqrt{U(\infty)/(\hbar^2/2m)} = k_b,$$

where the Born approximation is valid, the problem is reduced to an inverse one:

$$R(k_z) = R_0(k_z) \mid \frac{1}{U(\infty)} \frac{dU(z)}{dz} exp(2ik_z)dz \mid^2,$$

where $R_0(k_z)$ is the coefficient of reflection from a potential with an absolutely sharp barrier:

$$R_0(k_z) = |k_z - k_z'|^2/|k_z + k_z'|^2,$$

and where $k_z' = k_z\sqrt{1 - U(\infty)/(\hbar^2/2m)}$ is the component of the wave vector of a neutron in a medium, and m is the neutron mass. Frequently used in this instance is the assumption of a Gaussian distribution for the gradient of the potential:

$$\frac{1}{U(z)} \frac{dU}{dz} = (1/\sqrt{2\pi}\sigma) exp(-z^2/2\sigma^2). \qquad (1)$$

In this case, i.e., in the case of a diffuse boundary, for all k_z, a good approximation is the formula:

$$R(k_z) = R_0(k_z) exp(-4k_z k_z' \sigma^2). \qquad (2)$$

The lower limit value of σ (see formulas (1),(2)), reliably obtained from an experimentally measured value of $R(k_z)$, lies in the area of few angstroms. In the process of the reflection of neutrons from a thin film which has a contrast with a substrate, i.e., where there is a potentials difference of the film and the substrate, the dependence of R on k_z acquires an oscillating character (see Fig.5) caused by the interference of waves reflected from the surface of the film and its hidden boundary with the substrate. As a result, the averaged-area thickness of the film equal to several thousand angstroms can be determined with an accuracy of the order of $1\mathring{A}$.

Ferromagnetic media have the quality to polarize thermal neutrons specularly reflected from their surfaces. The physical reason for this

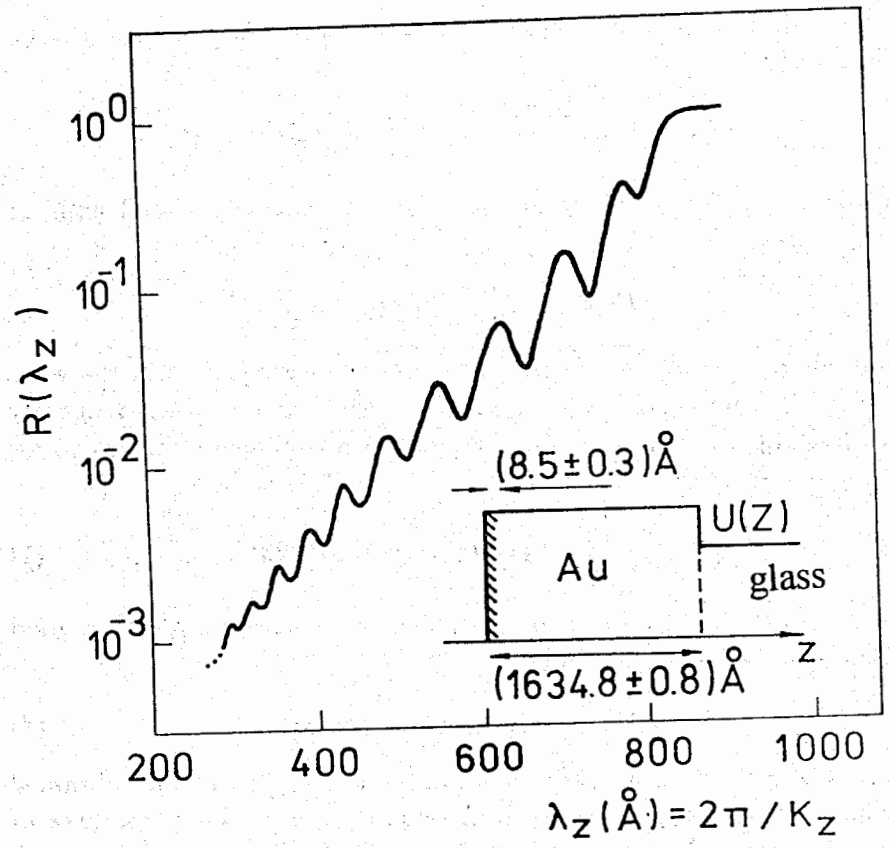

Fig. 5. The experimental dependence of the reflection coefficient $R(\lambda_z)$ from the surface of a thin gold film deposited on a glass surface by thermal spraying. At the insert: the shape of the neutron-optical potential providing the $R(\lambda_z)$ fitting.

phenomenon is connected with the fact that the value of the interaction potential $U_{mag} = -4\pi(\mu_n M)$ of the magnetic moment of a neutron, μ_n, and the vector of local magnetization of a sample, M, is, as a rule, comparable with that of the neutron-nuclear potential, U_{nucl}. Note that $U_{mag} + U_{nucl} = U(z)$. The quantitative caracteristic for the process of polarization following specular reflection is the vector of the polarizability of a medium, which defines the value and direction of polarization in a reflected beam. The vectors $Q(k_z)$ and $M_s(z)$, ($M_s(z)$ is the projection of the $M(z)$ vector onto the XY plane), are unambigously related. This fact forms the basis for deriving information about $M_s(z)$ from data on $Q(k_z)$. The evident consequence of Q being dependent only on the components of M_s lying in the plane of a film is the possibility to separate the $U_{mag}(z)$ and $U_{nucl}(z)$ contributions to specular reflection by total magnetization of a film in a direction perpendicular to its surface. The same procedure is used in diffraction to separate the magnetic and nuclear contribution to neutron scattering.

The unambiguous inter-relationship of Q and M_s allows the application of polarization reflectometry methods to the investigation of magnetization of thin films with an inhomogeneous over depth magnetic structure. This is a unique quality of polarization reflectometry. Other well-developed methods for investigating magnetic films allow either a statement of the existence and type of an inhomogeneous state (the spin-wave resonance), or an estimation of the thickness of an inhomogeneously magnetized near-surface layer (on the level of $0.1 - 0.2\mu m$) and the determination of the type of anisotropy in this layer (the Kerra magneto-optical effect). The well-developed methods of electron microscopy of the Lorentz or raster type cannot, in principle, be applied to the investigation of inhomeneous magnetic film depth profiles along the normal to surface, as a contrast electron image appears, due to the existence of magnetization inhomogeneities along the surface.

Consequently, in polarization reflectometry, one measures the intensities of reflected beams: positively polarized $I_+(k_z)$ (spin-flipper off) and negatively polarized $I_-(k_z)$ (spin-flipper on). The sign of polarization is taken with respect to the vector, H of the guiding magnetic field of the neutron path. The direction of H at the sample position defines the spatial direction of the P polarization vector of an incident beam.

The values $I_+(k_z)$ and $I_-(k_z)$ are related to the scalar product of the vectors P and $Q(k_z)$ via the relation:

$$\vec{P} \cdot \vec{Q} = \frac{I_+(k_z) - I_-(k_z)}{I_+(k_z) + I_-(k_z)}.$$

So, to determine the components $Q_{x,y,z}$ of the vector $Q(k_z)$ for a given sample, it is sufficient to measure the intensities $I_{+,-}(k_z)$ in the P direction along the XYZ-axes, respectively. The procedure of restoration of the $M_s(z)$ function of a film is reduced to the fit of the experimentally measured components $Q_{x,y,z}(k_z)$ of the medium's polarizability vector with their theoretical values calculated for a model distribution $M_s(z)$. The method of calculating $Q(k_z)$ is based on the solution of the quantum-mechanics problem of the reflection of a spinor wave from the surface of a magnetic medium with an inhomogeneous distribution $M_s(z)$. The solution allows the construction of a matrix, R, measuring 2×2 and connecting the spinors of an incident $\binom{\alpha}{\beta} e^{ik_z z}$ and a scattered $\binom{\gamma}{\varepsilon} e^{-ik_z z}$ wave:

$$\begin{pmatrix} \gamma \\ \varepsilon \end{pmatrix} = R \begin{pmatrix} \alpha \\ \beta \end{pmatrix}.$$

Numerical procedures have been developed [12] to determine the $R_{ij}(k_z)$ coefficients of the matrix for the general case of an arbitrary structure, $M_s(z)$. The $Q(k_z)$ vector, which is measured experimentally, is related to the R matrix via a simple relation:

$$Q = \frac{tr(RR^+\sigma)}{tr(RR^+)},$$

where σ is the vector with the components $\sigma_x, \sigma_y, \sigma_z$ (the Pauli matrix), and R^+ is the ermit-conjugated matrix. An important consequence of the non-collinear behavior of the $M_s(z)$ vector is the appearance of the component $Q_z \neq 0$, while for all types of collinear structures, this component is $Q_z = 0$. In addition, polarization reflectometry has been successfully applied to the study of the London penetration depth of a magnetic field in superconductors [13,14].

3 The Scheme for a Neutron Reflectometer at a Steady State Reactor

The main details of the proposed reflectometer (see Fig.6) are:

1. A vertical geometry for a sample.
2. Simultaneous irradiation of a sample with several monochromatic beams at different angles of incidence.
3. Registration of reflected neutron beams with a one-dimensional position-sensitive detector.

Close to the reactor gate, a block of double-crystal monochromators (1) is installed to form a monochromatic beam of neutrons. This double-crystal monochromator has proved to be an effective device providing certain advantages in comparison with a single-crystal one. The main advantage in this case is the possibility to reconstruct the neutron wavelength without changing the direction of the beam, thus preserving the condition of low background on a sample.

Simple removeable cadmium diaphragms (2) with a set of slits allow division of a beam into two, three or more narrower beams of the required collimation, $\delta\theta$. The widths of the slits and the distance between them are chosen to meet the requirements of an actual experiment and ensure the necessary angular resolution of $\delta\theta/\theta$ and the difference, $\Delta\theta$, in the grazing angles on a sample. This arrangement does not exclude the possibility of one-beam irradiation of a sample. It is evident that the multi-beam principle of sample irradiation permits an essential decrease in the measuring time of the angular dependence of the reflectivity.

At a distance of about four meters from the monochromator block is a rotating table (3) for positioning samples that enables transverse displacement of a sample, and scanning over the various neutron grazing angles with respect to an investigated surface.

The table is provided with uniform sample holders that permit the fixing of different sized samples. The construction and size of the table will allow future positioning of additional devices for changing external conditions at the sample, such as temperature, humidity, pressure, gaseous medium composition, etc. The sample holders are aslo equipped with collimating cadmium diaphragms (4,5).

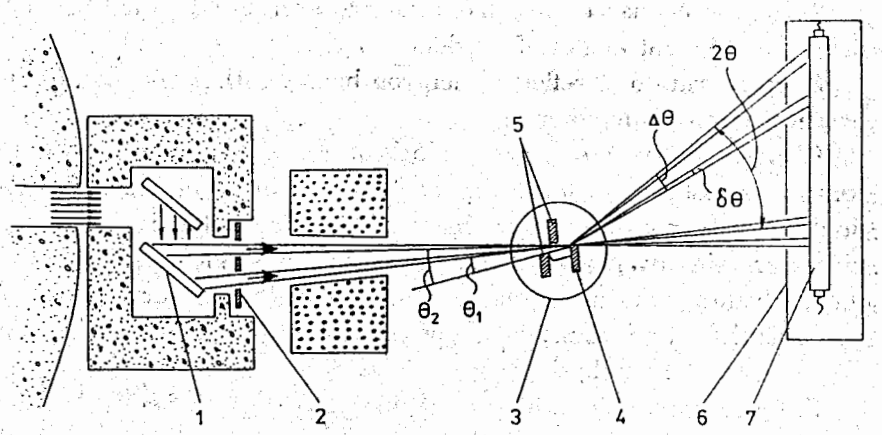

Fig. 6. The schematical top view of the proposed double-beam reflectometer for the steady state ET-RR-1 reactor. 1 — a block of double-crystal monochromators of changeable wavelength, 2 — a two-slit removeable Cd-diaphragm providing simultaneous irradiation of a sample by two beams at the grazing angles θ_1 and θ_2, 3 — a rotating table for sample mounting, 4 — a sample holder with a Cd diaphragm, 5 — a moveable table, 6 — a one-dimensional position-sensitive detector.

At a distance equal to the monochromator-sample distance, a detector (7) is placed on a moving table (6) providing displacement of the detector across a beam on an order of $200mm$.

The one-dimensional position-sensitive gas-filled (He^3, 6 atm) detector (7) with a resistive wire has a resolution of the order of $1.5mm$ and allows simultaneous registration of all reflected beams with about 80% efficiency. (It should be noted that use of a single detector requires the creation of a system to synchronize the scanning with the sample, following the $\theta - 2\theta$ law. With a position-sensitive detector, a synchronized scanning system is not necessary. This makes the construction of a reflectometer much simpler and excludes the possibility of systematic errors arising when the surface of a real sample being investigated is bent, since a change in the angle of a sample destroys the $\theta - 2\theta$ law).

Along the monochromator-sample and sample-detector parts, vacuum tubes are installed to avoid the loss of neutrons due to scattering on air.

A system of collimators along the monochromator-sample-detector part is to suppress the background of fast neutrons and gamma-rays.

The system for sample adjustment includes a laser whose beam goes into a vacuum system through a glass window and then, co-axial to the neutron beam, is directed to a sample with the help of a thin mirror.

Electronic systems for control and monitoring of the movable elements of the instrument, and for the collection of data from the detector, are in the CAMAC standard compatible with a PC/AT386 type personal computer. Archiving and processing of experimental data are performed with a PC/AT486 computer.

For the primary processing of experimental data and their graphical representation, as well as for the determination of the physical characteristics of investigated surfaces from the experimentally measured reflectivity, a complex of computer programs is being developed.

Table 1 summarizes the parameters of the proposed reflectometer. Table 2 is a list of the main components needed for construction of the reflectometer.

TABLE 1

Reflectometer Details

Scattering geometry	Horizontal
Number of incident beams	
min	1
max	3
Fixed angle between incident beams	$\Delta\theta = (2 \div 4) \times 10^{-3} rad$
Grazing angles $\theta_1, \theta_2, \theta_3$	$\theta_1 = (-10^{-2}\ rad \div 10^{-2}\ rad)$ $\theta_2 = \theta_1 + \Delta\theta$ $\theta_3 = \theta_2 + \Delta\theta$
Horizontal angular resolution $\delta\theta_1/\theta_1 \simeq \delta\theta_2/\theta_2 \simeq \delta\theta_3/\theta_3$	
max	2%
min	8%
	$\Delta\theta/\delta\theta \simeq 10$
Vertical angular divergency	$1.5 \times 10^{-2}\ rad$
Type of monochromator	double crystal
Monochromatic λ wavelength range	changeable: $(2-4)\AA, \delta\lambda/\lambda \simeq 2\%$
Position-sensitive detector	
resolution	1.5 mm
length	100 mm
efficiency	80%
height	50 mm
Neutron flux on sample from 2 MW reactor $(1/sec/mm^2/mrad^2)$	$\simeq 10^2$
Typical measuring time	5 hr
Electronic standard	CAMAC+Personal computer

TABLE 2

Main Construction Components

I.	Mechanical part
1.	Design work
2.	Development of shop drawings
3.	Purchase of materials
4.	Manufacture of mechanical parts
II.	Physical instruments and equipment
	Including: monochromators, position-sensitive detector (and electronic equipment for it), beam-monitor, step-drivers, laser.
III.	Electronic and automatic equipment
1.	Design of a measuring-and-storage module and an electronic system for automatic control.
2.	Development of non-standard electronic blocks.
3.	Purchase of standard electronic equipment, including : CAMAC blocks, and computational equipment.
4.	Development of computer programs for control of the main elements of the reflectometer, accumulation, storage and processing of experimental data.
IV.	Assembly and adjustment works
1.	On-site assembly.
2.	Adjustment.
3.	Test measurements and determination of the actual physical parameters of the instrument.

4 Conclusions

The performed considerations have shown that a very effective instrument can be built for use with the ET-RR-1 reactor to:

1) investigate the engineering aspects of the quality of surfaces, in particular, to test mirrors for neutron guides;

2) investigate the physical properties of the surfaces of bulks and films; and

3) train scientists and the reactor maintenance staff.

Actually, such an instrument as proposed could be operated with any steady state reactor. As soon as the characteristics of a steady state reactor are known in detail, the parameters of this reflectometer can be refined.

This work was performed with the support of the Russian Fundamental Research Fund (grant N93-02-2517).

Acknowledgements

The authors wish to express their appreciation to Dr. A.M.Balagurov for his useful comments. Thanks also to T.F.Drosdova for translating the text and to A.Schaeffer for editing it and to V.S.Rumyantseva for LaTex adoption.

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Received by Publishing Department
on December 1, 1993.

Аксенов В.Л. и др. Е3-93-430

Многопучковый нейтронный рефлектометр
для постоянного реактора

Предложена конструкция нейтронного рефлектометра для нейтронного контроля поверхности стекла, изучения тонких пленок и поверхностей на реакторе ET-RR-1 Управления по атомной энергии Египта.

Работа выполнена в Лаборатории нейтронной физики им. И.М.Франка ОИЯИ.

Сообщение Объединенного института ядерных исследований. Дубна, 1993

Aksenov V.L. et al. E3-93-430

Multi-Beam Neutron Reflectometer for a Steady State Reactor

A desing has been proposed for a neutron reflectometer to test glass surface by neutron controls and to study surfaces and thin films at the ET-RR-1 steady state reactor of the Atomic Energy Authority of Egypt.

The investigation has been performed at the Frank Laboratory of Neutron Physics, JINR.

Communication of the Joint Institute for Nuclear Research. Dubna, 1993