PREFACE

We would like to offer the readers the scientific activity report of the Frank Laboratory of Neutron Physics for 2011. The first part of the report presents a brief review of the experimental and theoretical results achieved in the main scientific directions – condensed matter physics, neutron nuclear physics, applied research and development and creation of elements of neutron spectrometers for condensed matter investigations. The second part includes the reports on the modernization of the IBR-2 pulsed reactor and the development of the IREN neutron source. A list of publications for 2011, the information regarding the seminars and conferences organized in FLNP and a statistical view on the FLNP personnel structure are presented as well. The experimental reports that cover the main scientific directions in greater detail complete the report.

In 2011 the main achievements of the Laboratory were:

- power start-up of the IBR-2 reactor after modernization;
- modernization of some spectrometers at the IBR-2 reactor and continuation of the development of new facilities for neutron studies in condensed matter physics;
- receiving of more than 150 applications for beam time at the IBR-2 spectrometers during the first call for proposals announced after the IBR-2 modernization;
- development of scientific experiments at local facilities and in collaboration with other scientific centers in Russia and abroad.

FLNP has cooperation agreements in the field of neutron investigations with more than 200 scientific institutes and universities from 43 countries from all over the world. A significant contribution to this cooperation is made by the JINR Member States.

The FLNP staff consists of more than 400 employees. The scientific staff includes 70 Ph.D. and 20 D.Sci. researchers and more than 50 researchers and specialists from the JINR Member States (besides the Russian Federation) with two thirds of them under 35 years of age.

The organization of annual conferences and schools covering all research fields of the FLNP activities helps to recruit young specialists — one of the top priority tasks of the FLNP Directorate.

All these facts confirm that the Laboratory continues to develop successfully and dynamically, carrying out investigations in the interests of the JINR Member States.

A.V. Belushkin

Director

1. SCIENTIFIC RESEARCH

NEUTRON NUCLEAR PHYSICS

In 2011, at the Frank Laboratory of Neutron Physics the applied research activities using the neutron spectroscopy techniques were actively carried out at the pulsed resonance neutron source IREN. A number of instrument development and analytical investigations were performed during the power start-up of the modernized IBR-2 reactor. Also, the instrument development activities on the preparation of experiments at the IREN facility were continued. The greater part of the fundamental investigations in the field of neutron nuclear physics was carried out on the neutron beams of nuclear research centers in Russia, Germany, Republic of Korea, China and France. The studies were conducted in the following traditional directions: investigations of time and space parity violation processes in neutron-nuclear interactions; studies of the fission process; experimental and theoretical investigations of electromagnetic properties of the neutron and of its beta-decay; gamma-spectroscopy of neutron-nuclear interactions, atomic nuclear structure, obtaining of new data for reactor applications and for nuclear astrophysics; experiments with ultracold neutrons; applied research.

3. Experimental and instrument development activities

3.1. Modernization of the detector system “Romashka-1”

In the framework of cooperation between the Institute for Nuclear Research and Nuclear Energy (INRNE) of the Bulgarian Academy of Sciences (BAS) and JINR in the field of investigation of neutron-nuclear interactions a low-background detector system “Romashka-1” for detecting gamma rays has been delivered from INRNE to FLNP JINR. A general view of the 12-section scintillation system is presented in **Fig. 15**.

The system is intended to determine the concentration of radioactive elements in the environment and to study the radiative neutron capture in the experiments conducted on the IREN facility in FLNP JINR by measuring gamma-ray multiplicity in the decay of radioactive nuclei. The gamma-ray spectrometry system consists of two sets of 6 NaI(Tl) monocrystals of trapezoidal cross section in the shape of a daisy that are placed in metal cylinders-containers about 30 cm in diameter (**Fig. 16**). A photoelectronic multiplier PEM-110 is optically connected to the butt end of each crystal.

The main characteristics of the gamma-spectrometry system “Romashka-1” are given in **Table 1**.

Table 1. The main characteristics of the gamma-spectrometry scintillation system “Romashka-1”.

Parameter	Value
Scintillation material	NaI(Tl)
Total volume of scintillators	~ 16.6 l
Length of individual scintillator elements	~ 200 mm
Detection efficiency 1.46 MeV □-rays of ^{40}K (10 Bq)	~ 97%
Minimum detected □-activity	~ 0.5 Bq
Energy resolution for detection of ^{60}Co□-rays of one section	~ 10 %
Energy resolution for detection of ^{60}Co□-rays of 12 sections	~ 15 %

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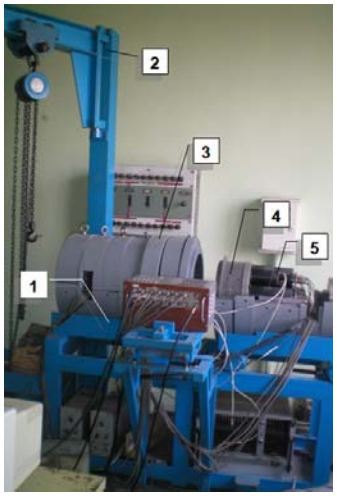

Fig. 15. 12-section gamma-spectrometry scintillation system "Romashka-1": **1** – iron support-holder, **2** – hoisting mechanism, **3** – segmental lead jacket, **4** – assembly of 6 NaI(Tl) scintillation crystals, **5** – photoelectronic multiplier.

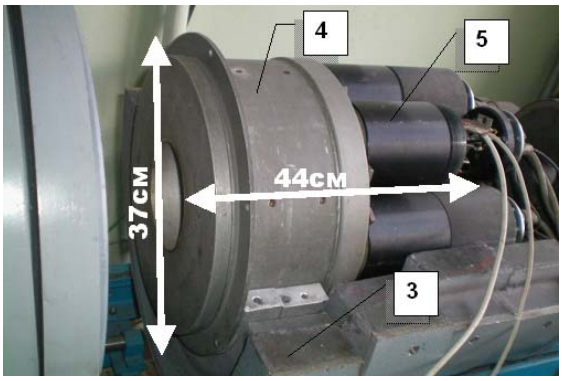

Fig. 16. An external view of one scintillation assembly: **3** – segmental lead jacket, **4** – six NaI(Tl) in the housing; **5** – electronic signal conversion-amplification modules PEM-110.

The modernization of the gamma-ray scintillation spectrometer includes the following activities:

– manufacturing of the 16-module photomultiplier high-voltage supply system by the firm "High-voltage systems" (Dubna) of the type shown in **Fig.17**;

- manufacturing of ADC of the board for 16 channels for simultaneous acquisition, digitization, accumulation and preliminary sorting of signals from all 12 sections of "Romashka" by the firm AFI Electronics-JINR (**Fig. 18**);

Fig.17. 6-channel high-voltage power supply source.

Fig. 18. ADC ADCM16-LTC 16-channel system.

- manufacturing of high-voltage and signal cables;

– development of software for processing experimental data.

The instrument will be located on beam 4 of IREN at a distance of about 31 m from a neutron-producing target.

3.2. Investigation of the fission process at low excitation energies

The fission fragment mass and kinetic energy characteristics play a key role in the description of the fission process. In the modern interpretation of the fission process the major role belongs to the shell effects, due to which the physicists have gained a better insight into the asymmetric mass fragmentation in the nuclear fission at low excitation energies. Owing to the possibilities of modern computers the calculation methods for the dynamic characteristics of nuclear fission (such as the nuclear surface potential energy dynamics at the nuclear surface fluctuations) are developing rapidly. As a result, higher requirements are imposed on the accuracy of experimental data, which characterize the fission process more comprehensively: mass and energy distributions of fission fragments, prompt neutron multiplicity and their dependence on the total kinetic energy and mass of fission fragments. In this connection, in FLNP the experimental methods for measuring the mentioned characteristics of nuclei in the resonance and mega-electron-volt energy range of neutrons inducing nuclear fission of transuranium elements are being developed. In the FLNP Nuclear Physics Department the techniques and equipment have been developed for carrying out nuclear fission investigations at the resonance neutron source IREN and the fast neutron source EG-5.

For the first time the technique of direct analysis of current pulses of a twin Frisch-gridded ionization chamber has been developed. The advantages of the method of direct processing of current pulses in comparison with the traditional conversion of current signals to step-function pulses by means of charge-sensitive converters have been demonstrated. The application of digital processing techniques has made it possible to get the best fission fragment mass and kinetic energy resolution in the investigations of spontaneous fission of Cf-252. A specialized software package has been developed for digital processing of signals for prompt fission neutron and fission fragment mass spectroscopy in low-energy fission experiments. The prompt fission neutron time-of-flight spectrum unfolding method that can be applied to single fission events and allows the determination of prompt neutron-induced fission energy has been developed.

A twin ionization chamber that allows along with the fission fragment mass spectroscopy the determination of fission axis orientation in space, has been constructed. The first results have been obtained that demonstrate reasonable angular ($\cos(\Delta x)$, $\cos(\Delta y)$, $\cos(\Delta z)$ ~0.05) and kinetic energy resolution for fission fragments (**Fig. 19**).

The signal formation in the Frisch-gridded ionization chamber has been studied using the finite difference method. The obtained solution of the Laplace equation has made it possible to find the relation between the geometric parameters of the chamber and the shape of pulses induced on the chamber electrodes in the process of the drift of ionization electrons. As a result, the formulae of dependence between the induced signals on the anode of the chamber and the direction of motion of a charged particle in the sensitive volume of the chamber have been derived. This has allowed us to resolve the ambiguities of interpretation of the grid inefficiency concept. The equipment and the software for digital signal processing that includes eight PC-built-in channels of fast analog-to-digital converters (8 bits, 250 MHz) have been developed, constructed and tested in the test data acquisition experiments with the twin ionization chamber (at EG-5) and the fast

neutron scintillation spectrometer (with a neutron source). The developed equipment of the time-of-flight analyzer together with the fast current amplifiers has been used in the experiments on the measurement of the IBR-2 thermal neutron beam intensity. The doubtless advantage of the current converter over the charge one has been demonstrated.

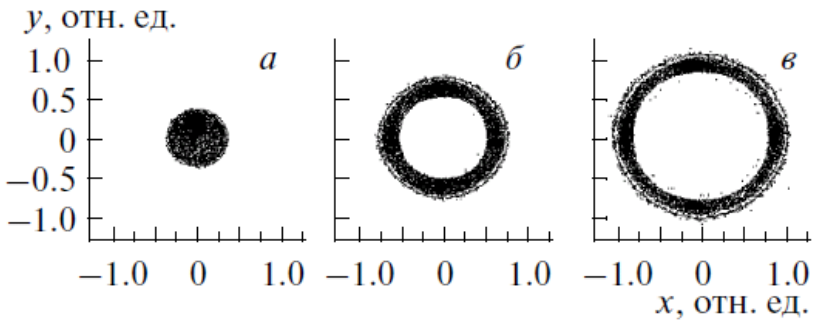

Fig. 19. Demonstration of fission fragment angular resolution using a position-sensitive twin Frisch-gridded ionization chamber.

3.3. Measurement of fission fragments using Medipix2 and Timepix detectors

In the framework of the collaboration between JINR and Czech Technical University in Prague a number of experiments have been conducted to study the properties of silicon pixel detectors of the Medipix family and the possibilities of their application for measuring heavy charged particles, specifically for searching and studying rare nuclear fission modes. The measurements of fission fragments and alpha-particles in the spontaneous fission of ^{252}Cf have been performed using several detectors Timepix and Medipix-2 included in the coincidence circuit, and in particular, using a start module specially developed for these measurements. A typical scheme of measurements is given in **Fig. 20**.

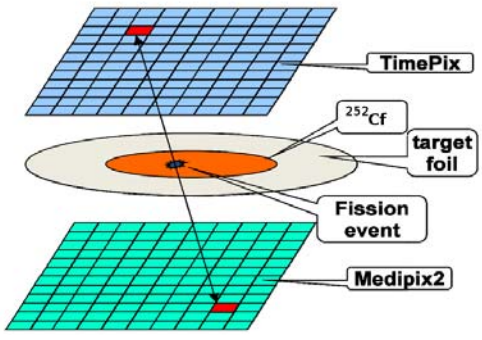

Fig. 20. Scheme of the measurement with Medipix2 and Timepix detectors.

The spontaneous fission source of ^{252}Cf was placed between two pixel detectors (in other measurements it was surrounded by four detectors) which allowed us to detect the arrival time and energy of fission fragments and alpha particles, as well as with a high accuracy the coordinates of the

particle's entry point in the detector. The possibility of measurement of fission fragments in coincidence with each other and with alpha particles has been demonstrated, which makes it possible to register ternary fission events with the help of pixel detectors. It has been shown that the Timepix detector exhibits the best characteristics for such investigations allowing one to work in two modes – measurement of the arrival time of a signal in each pixel and measurement of the energy transferred by a particle in the given pixel.

3.4. Activities on the preparation of a precision experiment for n,e-scattering length extraction

Fig. 21. An overall view of 8-channel time encoder.

The experimental installation AURA for measuring the angular anisotropy of slow neutrons scattered by noble gases in order to determine the (n,e)-scattering length has been assembled on beam №2 of the IREN facility. The development of the software for controlling the turn-table with neutron detectors has been completed, and the installation has been tested in the operating mode without a neutron beam. A new 8-channel fast time encoder (**Fig. 21**) has been developed and tested using the loading of noise pulses from the detectors (at low thresholds).

For a precise measurement of the angular anisotropy of neutrons scattered by inert gases in the energy region from a few MeV up to 1 eV it is necessary to know all corrections with an accuracy of no worse than 10^{-4}. Only in this case one can obtain the n,e-scattering length b_{ne} with the appropriate accuracy of 2-3 %. The corrections for efficiency variation of detectors registering slow neutrons scattered forward or backward taking into account the thermal motion of argon atoms have been calculated by the Monte Carlo method in the real geometry (**Fig. 22**).

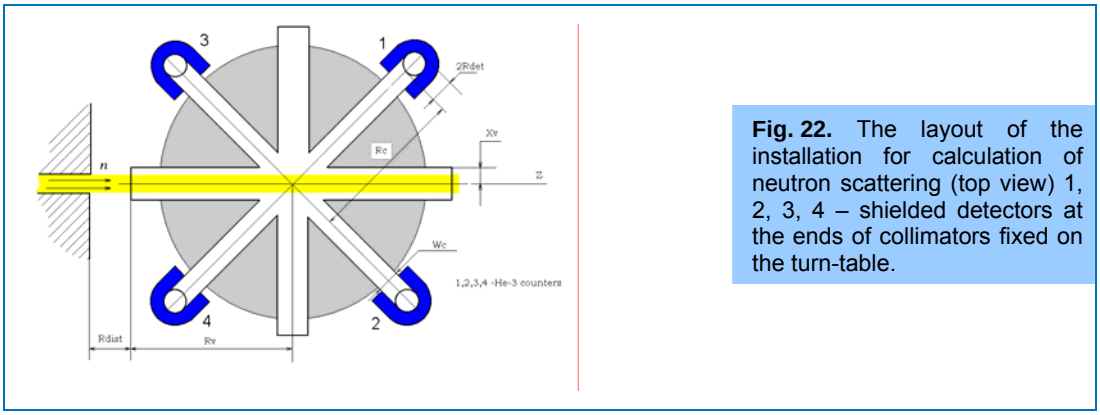

Fig. 22. The layout of the installation for calculation of neutron scattering (top view) 1, 2, 3, 4 – shielded detectors at the ends of collimators fixed on the turn-table.

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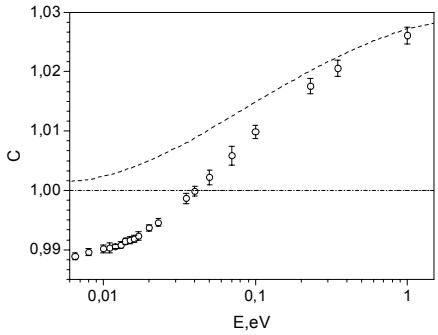
The ratio of efficiencies of counters detecting neutrons scattered forward and backward depends on the initial neutron energy and real angular distribution complicated by the thermal motion of gas atoms. This ratio should be accurately calculated for correction of the experimental results in each point of the energy region under study. For this purpose the Monte Carlo calculations have been performed for argon at a pressure of 10 atm and at initial neutron energies from 0.001 eV. (**Fig. 23**).

Fig. 23. The corrections $C(E)=\varepsilon(135^0)/\varepsilon(45^0)$ obtained from the Monte Carlo calculations for intensities of neutrons scattered forward and backward by argon. The dashed curve is a raw count of *C(E)* without taking into account the thermal motion of gas atoms.



The cumbersome calculations in the real geometry taking into account the thermal motion of gas atoms are necessary to reduce the uncertainty of the correction coefficient *C*(*E*) to the desired value. Only accurate account of this correction will make it possible to obtain the *R*(*E*) ratio in the neutron energy region of more than 0.1 eV.

3.5. Investigations of (n,p), (n,α) reactions

In accordance with the Protocol on Cooperation between JINR and Peking *University* the experimental and theoretical investigations of the reactions (neutron, charged particle) induced by fast neutrons have been carried out. The experiments are conducted at the Van de Graaf accelerators EG-5 in FLNP JINR (Dubna, Russia) and EG-4.5 of the *Institute of Heavy Ion* Physics of Peking *University* (Beijing, China) in collaboration with the University of Lodz (Poland), the National University of Mongolia (Ulaanbaatar, Mongolia) and the Oak Ridge National Laboratory (USA). Data on the neutron reactions with the emission of charged particles induced by fast neutrons are of much interest for studying the mechanisms of nuclear reactions, atomic nuclear structure and for determining alpha-particle optical potential parameters. The latter is of much importance for calculations of various scenarios in astrophysics. In addition, these data are essential for choosing construction materials and for performing calculations in the development of new facilities for nuclear power engineering.

The data treatment for the measurements of the ^{149}Sm(n,α)^{146}Nd reaction conducted in the neutron energy range between 4.5 and 6.5 MeV has been completed. The data on the cross sections and angular distributions for this neutron energy range have been obtained for the first time; a comparison with the available estimates and theoretical models has been performed. They are of great importance because of significant discrepancies between the estimates given by different nuclear data libraries, which are based on theoretical predictions, and since only very scarce data are available for cross sections in the (n,α) channel, in particular, in the given mass range (**Fig. 24**).

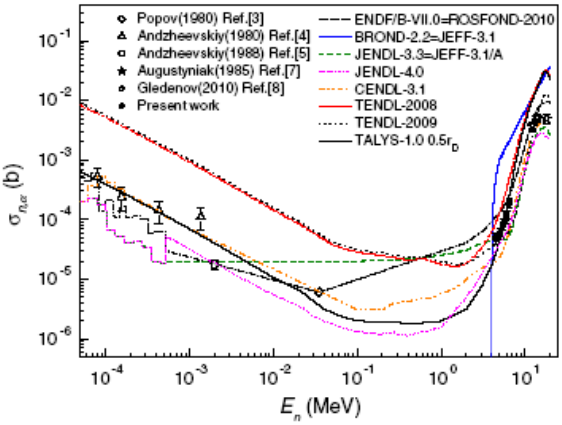


Fig. 24. Cross section of $^{149}Sm(n,\alpha)^{146}Nd$ in comparison with the estimates given by different nuclear data libraries and the calculations using the TALYS-1.0 software package.

The treatment and analysis of data from the measurements of the $^{35}Cl(n,\alpha)^{32}P$ and $^{40}Ca(n,\alpha)^{37}Ar$ reactions at a neutron energy E_n ~ 4.0 – 6.5 MeV have started. The measurements of the $^{nat}Mg(n,\alpha)$ reaction at E_n ~ 4.0-6.5 MeV have been performed. The samples of $^{54,57}Fe$ and ^{63}Cu have been prepared and in November, 2011, the measurements started on a neutron beam of EG-4.5.

3.6. Investigations of nuclear structure

By now the application of an original technique for determination of level density and radiative strength functions from the intensities of two-quanta cascades proceeding between neutron resonance and a group of its low-lying levels has made it possible to develop a radically new technique for determination of parameters of the phase transition of any nucleus from the superfluid to the normal state. The main result is that the breaking of the first four nucleon Cooper pairs occurs successively and the thresholds of the next pair breaking differ by the value comparable with the doubled nucleon pairing energy and decreasing with an increase in the nuclear excitation energy. Hence it follows that in the neutron resonance energy region their structure can differ to such a degree that this distinction can be revealed experimentally. For preliminary assessment of this possibility a technique for determination of distribution parameters of neutron and radiative resonance widths has been developed. It is based on the results of the analysis of the nuclear level density below the neutron binding energy obtained in Dubna and includes as a special case the Porter-Thomas distribution widely used for this purpose. The reanalysis of the distributions of the reduced neutron and total radiative widths of neutron resonances within the framework of the modified model of distributions of these quantities has been completed. The maximum approximation accuracy has been achieved on the assumption that the respective experimental data are the superposition of up to 4 distributions of amplitudes with different dispersions and mean values. This result corresponds to the obtained earlier conclusions on the dynamics of nuclear transition between Fermi- and Bose-states.

3.7. Investigations of the interaction of a relativistic deuteron beam with a massive multiplying target of natural uranium

The JINR research project «Energy and Transmutation of Radioactive Wastes» («E&T RAW») is aimed at exploring possibilities of a new electro-nuclear scheme based on the use of deep subcritical multiplying systems of natural (depleted) uranium or thorium for energy production and utilization of spent nuclear fuel. The period of execution of the project first stage is 2010-2012. It is

carried out within the framework of a broad international collaboration on the basis of the JINR facilities.

In 2011, using a new subcritical assembly "QUINTA" consisting of 500 kg of natural metal uranium, the distributions of neutron fluxes, fission rates and ^{239}Pu recovery as well as the time dependence of the delayed neutron yield after irradiation of the assembly with a pulsed deutron beam of the JINR nuclotron were measured in the energy range of E_d = 2 – 6 GeV. The tendency of the average energy of neutrons $<E_n>$ inducing fission of ^{238}U inside the assembly to grow with an increase in the energy of incident deutrons (discovered in the experiments in 2010) has been confirmed. The value of $<En>$ ran as high as ~ 30 MeV at E_d = 6 GeV. It has been found (with an error of 10-15 %) that the integral number of fissions in the assembly grows, at least linearly with increasing E_d, whereas the relative total yield of delayed neutrons generated in the fission of the assembly nuclei increased approximately six times as the value of E_d grew from 2 to 6 GeV. In December, 2011, the measurements on the nuclotron continued in a wider deuteron energy range and using a more advanced technique (**Fig. 25**).

The long-term research objective is to obtain reliable data on the nuclear-physical characteristics of the QUINTA assembly as a central zone of a large (22 tons) uranium target BURAN (**Fig. 26**) available in JINR, and the complex development of the measurement techniques with this target.

Fig. 25. QUINTA assembly at the radiation position. March, 2011.

Fig. 26. Large uranium target BURAN.

In addition, the obtaining of the fullest possible set of experimental data on the QUINTA assembly is of independent fundamental importance for modification of the models of interaction of relativistic particles with extended multiplying media and for verification of the corresponding codes.

3.8. Experiment to study the quasi-elastic scattering of ultracold neutrons upon reflection from the surface of hydrogen-free Fomblin oil

An experiment has been carried out to measure the probability of quasi-elastic UCN scattering ("weak heating" of UCN) following their reflection from the surface of hydrogen-free Fomblin oil. The experiment has been performed in ILL (PF2 instrument) with the LGS spectrometer (developed in FLNP). Its external view is presented in **Fig. 27**.

Fig. 27. An external view of the LGS spectrometer.

The temperature dependence of the probability of UCN "weak heating" (**Fig. 28**) and spectra of heated neutrons have been obtained. The comparison of the obtained temperature dependence with the results of the work [A.P. Serebrov et al., Physics Letters A 309 (2003) 218–224] taking into account the difference in the performance of the experiments, allows us to draw a conclusion that the spectrum of scattered neutrons remains the same at all temperatures and only the total probability of weak heating undergoes a change.

Nowadays there are two hypotheses explaining this phenomenon – UCN scattering by capillary waves and UCN scattering by nanodrops formed near the liquid surface. The comparison of the Fomblin-heated neutron spectra with the spectra obtained with nanodispersed diamond samples and the comparison of the obtained temperature dependence with the dependence calculated in the work [S.K. Lamoreaux, R. Golub, Phys. Rev. C 66, 044309 (2002)] point to the fact that the second hypothesis is more credible. To reach an unambiguous conclusion, additional calculations of the temperature dependence of UCN scattering by capillary waves should be performed.

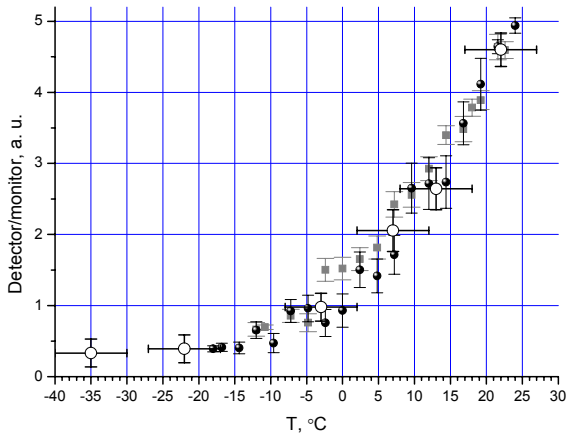


Fig. 28. The dependence of the ratio of heated neutrons counted for a cycle to the monitor count on the temperature of Fomblin oil (arbitrary units). Black circles – measurements with decreasing temperature. Grey squares – measurements with increasing temperature. White circles – data from [A.P. Serebrov et al., Physics Letters A 309 (2003) 218–224] reduced to our measurements at a temperature of 22 °C.

3.9. Status of a new experiment to test the equivalence principle for neutrons

A new UCN spectrometer EPIGRAV specially designed for the experiment to verify the equivalence principle for neutrons was constructed and tested in 2010 (**Fig. 29**).

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Fig. 29. The EPIGRAV spectrometer on the UCN source at ILL (Grenoble, France). December, 2011.

The operation of the instrument is based on the combined use of Fabry-Perot neutron interferometers and neutron flux modulator-chopper. The peculiarity of the instrument is the possibility of using the original time-of-flight technique based on the measurement of the detector count rate oscillation phase. The detection of UCN is performed by a scintillation detector synchronized with a modulator. A high degree of beam monochromatization makes it possible to work with the times of flight, which many times exceed the modulation period, thus ensuring a unique energy resolution of the instrument.

The analysis of the results of its first tests, however, pointed to the insufficient stability of its basic module: chopper-modulator. What is more, it was realized that under some conditions a failure in the operation of the modulator can lead to a serious breakdown. Therefore in 2011 a new chopper-modulator (**Fig. 30**) was designed and constructed, which demanded an essential change in the configuration of the spectrometer. Simultaneously, a mechanism for moving an interferential filter was improved and a new gas UCN detector with a thin converter layer of ^{10}B was manufactured. In cooperation with the group of the Institute of Nuclear Physics of the Johannes Gutenberg University of Mainz (Germany) new Fabry–Pérot interferometers (FPI) intended for use in the new spectrometer have been calculated, constructed and tested.

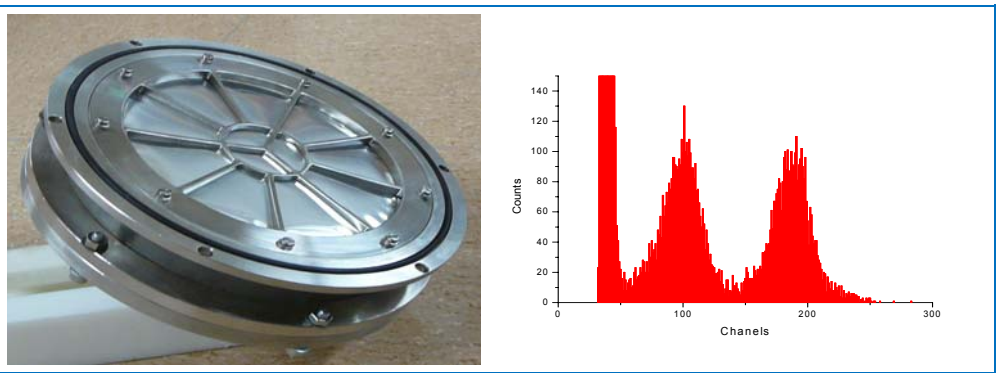

Fig. 30. A new detector with ^{10}B converter and its amplitude spectrum.

Comprehensive tests of the device have been conducted on the UCN beam of the Institute Laue Langevin (Grenoble, France). The new version of the chopper proved to be very good. During the tests it has worked without failures for about five hundred hours. The stability of frequency modulation was of the order of 2×10^{-4}. The spectrometer has rather high spectrometric qualities. The time resolution $\Delta t/t$ measured in the experiment was less than 2 % (**Fig. 31**). During the trials the spectrometer has been tested in all modes necessary for conducting a new gravitational experiment to verify the equivalence principle for neutrons. A number of test experiments to search for optimum conditions for the measurements have been carried out.

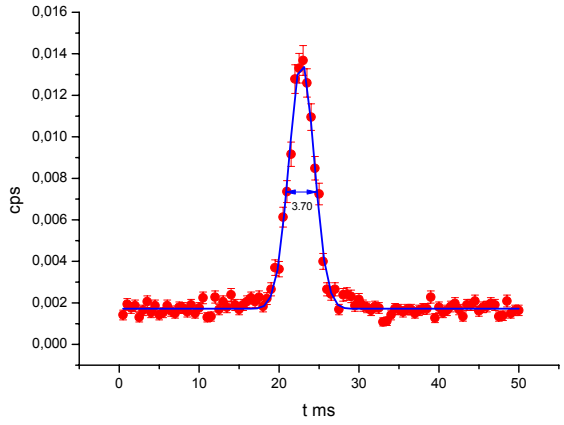

Fig. 31. A time-of-flight spectrum of ultracold neutrons monochromated by passing through a Fabry–Pérot interferometer.

3.10. Investigations of space parity violation effects

The measurements of the P-odd asymmetry in the radiative cross-section of natural lead have been performed at the PF1B cold polarized neutron beam facility in ILL (Grenoble, France). The experiment was conducted to obtain additional information to explain the anomalously high value of the neutron spin rotation in the measurements of transmission of transversely polarized neutrons through a sample.

The cold-neutron flux (average wavelength – 4.7 Å) was ~ 10^{10} 1/s. The neutron polarization was no worse than P_n = 92 %. A lead target (purity – 99.95 %) was positioned on the longitudinally polarized neutron beam between γ-quantum detectors. NaI (Tl) crystals 200 mm in diameter and 100 mm thick served as detectors. For absorption of scattered neutrons the target was placed in a box of lithium rubber (^{6}LiF) ~ 1.9 mm thick, open from the side of beam entrance to the target. In the "zero" experiment the measurements were carried out with the box without a sample. The neutron polarization was changed by an adiabatic flipper. To decrease the effect of the reactor power fluctuations, an integral method was used at neutron polarization switching frequencies (8.3 Hz) higher than the frequencies of the main spectrum of the neutron noise of the reactor. The measurements with the lead sample were carried out for 10 days. The measurement result corrected for the neutron polarization is $\alpha_\gamma^{\exp} = (3.3 \pm 2.9)\times10^{-7}$. The measurement result of the “zero” test on the beam without a lead target normalized to the usual components of the basic experiment and corrected for the neutron polarization is $\alpha_{0-test} = (1.0 \pm 2.0)\times10^{-7}$. The measurement time – 6.5 days. Taking the “zero-test” into account the asymmetry effect was found to be $a_\gamma(^{nat}Pb) = (2.3 \pm 3.5)\times10^{-7}$ or $\alpha_\gamma \le 8.1\times10^{-7}$ at the 90% *confidence level.* The achieved accuracy is still insufficient to perform the combined analysis of P-odd effects in lead.

3.11. Work in the framework of the project dedicated to the direct measurement of n-n scattering.

In cooperation with Gettysburg College (Gettysburg, Pennsylvania, USA) the results from the first experiments on the direct measurement of the n-n scattering cross section at the YAGUAR pulsed reactor (Snezhinsk, Russia) aimed at studying charge symmetry of nuclear forces have been prepared for publication. According to the preliminary data the background of unknown nature has been detected, which was assumed to be connected with the radiation-induced hydrogen desorption from the surface of the aluminum channel of the instrument under an extremely high dose of gamma radiation during the reactor pulse. The simulation of transport of photons and photon-generated electrons in the central metal tube of the n-n facility has demonstrated that the desorption with a coefficient $\eta(\gamma) = 0.01$ is induced by low-energy (15-200 eV) electrons. The paper titled "Direct measurement of nn-scattering: the gamma ray-outgassing complication" has been submitted for publication in "*Journal of Physics G: Nuclear and Particle Physics*".

3.12. Creation and development of UCN sources

In cooperation with the research team of the ultra-cold neutron source of the Los Alamos National Laboratory (Los Alamos, New Mexico, USA) the simulation and the measurement of the flux density of cold neutrons in the facility have been performed. This source utilizes fast neutrons generated by 800-MeV protons in a tungsten target of the accelerator. Using polyethylene, the source slows down neutrons to cold energies and in a special cryostat converts them to UCN in the open 1.5-liter volume of solid deuterium cooled by liquid helium. UCN are transported to the experimental hall through stainless steel pipes. The density of 30 UCN/cm^3 behind a biological shield has been achieved. This work has become a component part in the publication "Performance of the LANL spallation-neutron driven solid deuterium ultra-cold neutron source" submitted to "*Review of Scientific Instruments"*.

3.13. Search for new short-range spin-dependent interactions

Possible neutron experiments to search for new short-range spin-dependent forces have been considered. The spin-dependent nucleon-nucleon interaction between neutrons and nuclei may be responsible for various effects: phase shift of a neutron wave in neutron interferometers of different types, in particular of the Lloyd mirror configuration, neutron spin rotation in a pseudo-magnetic field, and transverse deflection of a polarized neutron beam by a layer of substance. The sensitivity assessment of these experiments has been made.

4. Theoretical investigations

4.1. Development of the IREN facility

The energy and angular distributions of neutrons produced under irradiation of various samples-radiators by electron beams of linear accelerators have been investigated. Neutron spectra have been obtained for radiators consisting of heavy elements (U, Ta, W, Pb) in relation to current, electron energy of the accelerator and parameters of the radiator, which is necessary for optimization of neutron sources that use modern electron linear accelerators. A consistent theoretical description of interaction of electrons and photons with nuclei makes it possible to describe successfully the production of neutrons without resorting to numerical simulations. The neutron distributions obtained thus far in the experimental measurements on various linear electron accelerators have been compared with the results of the calculations.

4.2. On the possibility to increase the IREN neutron flux

A neutron-generating target of IREN surrounded by a beryllium cylinder 11-41 cm in diameter is considered (**Fig. 32**). A considerable cross section of the (n, 2n) reaction for beryllium (of the order of 0.6 barn) at a neutron energy of 4-10 MeV may increase the IREN neutron flux if the real neutron spectrum from the source has a significant part of fast neutrons. The neutron yield and time distribution for a W-Be source have been estimated using the GEANT and FLUKA software packages (**Table. 2**).

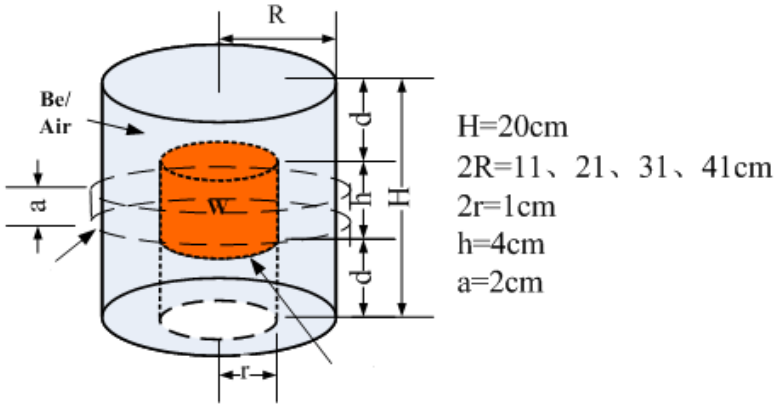

Fig. 32. The source design for the given calculations.

Table 2. Neutron yield per 1 electron (calculations using GEANT).

		Cylinder diameter			
		11 cm	**21 cm**	**31 cm**	**41 cm**
Electron energy of 30 MeV					
Yield in 4π	Air	0.00648	0.00642 0.0064*	0.00647 0.0063*	0.00640 0.0062*
	Be	0.00728	0.00760	0.00779	0.00767
	Be/Air ratio	1.12	1.18	1.20	1.20
Yield from cylinder lateral surface	Air	0.00575	0.00452	0.00362	0.00289
	Be	0.00662	0.00514	0.00343	0.00200
	Be/Air ratio	1.15	1.14	0.95	0.69
Electron energy of 50 MeV					
Yield in 4π	Air	0.0139	0.0139 0.0128*	0.0139	0.0139
	Be	0.0159	0.0166	0.0167	0.0167
	Be/Air ratio	1.14	1.19	1.20	1.20
Yield from cylinder lateral surface	Air	0.0123	0.00983	0.00776	0.00631
	Be	0.0145	0.0112	0.00738	0.00440
	Be/Air ratio	1.18	1.14	0.95	0.70

** - values obtained using FLUKA*

The effect of increasing number of neutrons after their passage through a beryllium layer of thickness L can be written as:

$$N = N_0 + N_0 \int_0^L [\exp(-n\sigma_t x) n\sigma_t dx] \frac{\sigma_{n,2n}}{\sigma_t} \quad \Rightarrow \quad N_0(1+\mu),$$

$$\mu = \frac{\sigma_{n,2n}}{\sigma_t}[1-\exp(-n\sigma_t L)]$$

In the case that $L > 10$ cm $\mu \approx \frac{\sigma_{n,2n}}{\sigma_t}$ and for neutron energies higher than 4 MeV $\mu \approx 1/2$.

The calculations presented in **Fig. 33**, have demonstrated the groundlessness of the idea about a possible time delay of neutrons due to their passage through beryllium (and, as a result, about possible neutron pulse widening). The estimated pulse widening was no more than 200 ns.

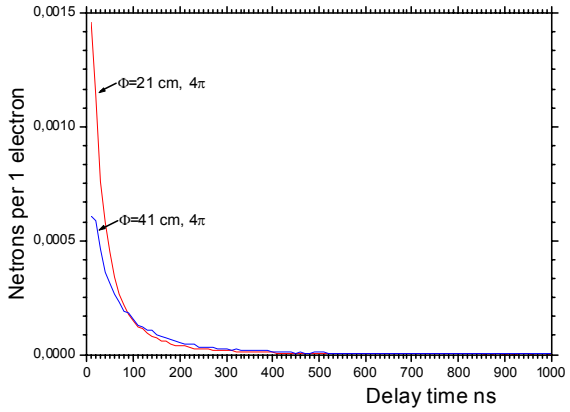

Fig. 33. Neutron time delay distribution when moving through beryllium (beryllium diameters – 21 and 41 cm).

4.3. Investigations of neutron-optical phenomena

The feasibility of experimental investigation of magnetic structures placed in an oscillating field is under discussion. It has been shown that the probabilities of reflection and transmission with and without spin flip have a resonance structure. The application of this phenomenon makes it possible to measure the magnetic field penetration profile, induction and magnetic susceptibility in alternating fields with a higher precision. It has been demonstrated that the refraction of neutrons in a magnetic prism under the action of an alternating field results in 8-fold splitting of an initially unpolarized beam. The intensity and polarization of each component depend on the magnitude and frequency of the alternating field. The possibility of measurement of magnetic permeability of films of angstrom-order thickness in alternating fields inside wave neutron resonators has been studied.

An experiment has been proposed on the amplification of an electromagnetic field under multiple total internal reflection from active media.

The Einstein-Podolsky-Rosen (EPR) paradox has been considered. The unavoidable redefinition of values of physical quantities has been shown to resolve the paradox. It has been demonstrated that according to the EPR logic the entangled states do not exist, and the measurement of violations of Bell's inequalities using downconversion photons does not mean a rejection of quantum mechanical locality. The consequences of the absence of the entangled states are under discussion.

1. SCIENTIFIC RESEARCH

5. Applied research

5.1. Nuclear-physical analytical techniques using charged particle beams

At the charged particle beams of the EG-5 accelerator (FLNP) the systematic studies of depth profiles of the elements in the near-surface layers of construction materials with the depth resolution of about 10 nm have been performed. In cooperation with IEE SAS (Bratislava, Slovakia) the content of light elements (including hydrogen) in layered structures produced on the silicon surface by means of PECVD (*plasma-enhanced chemical vapor deposition*) has been studied.

In cooperation with VSU (Voronezh) the studies of various layered structures for the technical use by means of the back-scattering of helium ions have continued.

In cooperation with MCSU (Lublin, Poland) the influence of the ion implantation on the optical properties of the natural oxide layer covering the silicon surface and A3B5-type compounds has been investigated.

The investigations of deuterium depth profiles have proved to be useful for studying the influence of the electronic screening effect on the $d(d,n)^3He$-reaction rate in the range of ultralow deuteron collision energies in ZrD_2 and TiD_2 targets. This work has been done by a large group of researchers from JINR, P.N.Lebedev Physical Institute of RAS (Moscow), National Research Tomsk Polytechnic University (Tomsk, Russia), Institute of Electrical Engineering of the Slovak Academy of Sciences (Bratislava, Slovak Republic), University of California (USA), University of Science and Technology (Krakow, Poland).

5.2. Development of analytical techniques using neutron spectrometry

At the IREN pulsed resonance neutron source the activities have been carried out on the development and application of the methods of elemental and isotope analysis using neutron spectrometry. The analysis of the boron content in ceramics of nanocomposite materials prepared in the Belorussian State University (Minsk) has been performed by measuring neutron transmission. In cooperation with the Sternberg Astronomical Institute MSU the objects of presumably extraterrestrial origin have been investigated by means of the resonance spectrometry method. In the samples from bottom sediments of a brook in an Altai mountain glacier a rather high iron content has been found. The work is in progress.

5.3. Analytical investigations at the IBR-2 reactor and IREN

Development of the NAA Sector experimental base.

IBR-2. In 2011, the rooms of the radioanalytical complex REGATA were repaired. The spectrometer equipment and software for processing gamma spectra were modernized, the software package was developed for automation of spectrum acquisition on the basis of Genie-2000 (Canberra) and for calculation of element concentrations determined by the neutron activation method. In the process of preparation of the NAA and Applied Research Sector for accreditation according to the ISO-17025 standard the universal database control system for NAA has been created in FLNP JINR.

IREN. In cooperation with «Development and Application Base in Physics (DAB-Physics)», Sofia, Bulgaria, the manufacturing of a pneumatic transport system for NAA studies at the IREN facility has been completed.

Method development. In October, 2011, during the first reactor cycle after the completion of modernization of IBR-2 the measurement of thermal, resonance and fast neutron flux densities was carried out in the irradiation channels of the REGATA pneumatic transport facility.

1. SCIENTIFIC RESEARCH

Biomonitoring. In 2011, in the framework of the international program "Heavy metal atmospheric deposition in Europe – estimations based on moss analysis" the studies on multielement atmospheric deposition in Croatia (Spiric et al., 2011) and in Serbia (Krmar et al., 2011) reflecting the contribution of the NAA Sector to the European Atlas were published. In cooperation with the Belgian Nuclear Research Centre SCK CEN a technique for the determination of depleted uranium in moss-biomonitors using k_0-method in NAA has been developed (P. Vermaercke et al., 2011). In October-December, 2011, NAA was performed for moss samples from three regions of the Russian Federation (Leningrad region, area near the city of Tikhvin; Kostroma region; Western Siberia, Iksinsky swamp), Belarus (Minsk and Gomel regions) and the ferrochrome production area in Norway.

Ecosystem condition assessment. A report for the Black Sea Economical Council and a publication in the international journal have been prepared on the results of the international project between Black See countries (BSEC-PDF) "Revitalization of urban ecosystems with the help of higher plants" with the participation of Russia (JINR), Bulgaria, Greece, Serbia, Romania and Turkey (2008-2010).

In December, 2011 within the framework of the project «The environmental assessment of the Nile delta area using nuclear-physical analytical methods» the neutron activation analysis of soils and bottom sediments from the territory under study was performed on the IBR-2 reactor in cooperation with the Egyptian specialists. Data treatment is in progress.

Radioecology. The program for investigation of distributions of ^{137}Cs and ^{210}Pb isotopes in mosses-biomonitors collected on the territory of Belarus and Slovakia 23 years after the Chernobyl accident has been completed. Gamma-spectrometry of the moss samples has been performed in the low-background laboratories at the Comenius University in Bratislava, Slovakia, and at the Nuclear Energy Corporation of South Africa (NECSA). In cooperation with the Slovakian and Norwegian scientists the data on the season variations of ^{137}Cs and ^{40}K isotopes in the ground air of Bratislava have been analyzed, which in the authors' opinion is the result of soil resuspension.

Geology. In cooperation with the Bucharest University, Romania, and the Geological Institute of RAS the multi-element analysis of bottom sediments and rocks of two semiclosed ecosystems of the glacial lake Balea (Fagaras mountains) and the crater lake St. Ana (Harghita mountains) has been carried out at the reactor of MEPHI in Moscow. The obtained results are of great interest for practical geology. The analysis of geological samples (ore) from one of the most promising non-ferrous metal deposits in Mongolia has been made using the neutron activation technique. In cooperation with the Vietnamese specialists and employees of FLNR JINR the behavior of rare-earth elements in the plant-soil system of Northern Vietnam has been studied. In December, 2011 the analysis of soils and medicinal clays from Romania was carried out on the IBR-2 reactor.

Analysis of materials of extraterrestrial origin. In December, 2011 on the REGATA facility the test neutron activation analysis was conducted for the sample of presumably extraterrestrial origin, which had been collected from a melting high-mountain glacier in Altai. The analysis of the same sample was performed on the IREN facility. The continuation of this work is scheduled for 2012.

Human and animal health, herbs. The studies on biomonitoring of atmospheric deposition with the use of land plants and their relation with the epidemiological data carried out in the NAA and applied research sector have been presented at the ***All-Russian scientific*** and ***practical conference*** «Monitoring of the health state, quality and lifestyle patterns of the population in Russia. The effects of behavioral risk factors on the population health» (June 7-8, 2011, Moscow).

Unique data on the element composition of some herbs from Mongolia and India applied in Asian alternative medicine have been obtained for the first time.

Biotechnologies. In 2011, in collaboration with the E.Andronikashvili Institute of Physics, I.Javakhishvili Tbilisi State University and I.Chavchavadze State ***University*** (***Tbilisi***, ***Georgia***) the studies on the development of methods for synthesis of silver and gold nanoparticles by certain

kinds of Actinomycetes including *Arthrobacter* as well as by blue-green algae *Spirulina platensis* continued. The effect of time and dose dependences on the formation of nanoparticles was investigated. A complex of spectroscopic and analytical methods – ultra-violet spectroscopy (UV-vis), X-ray diffraction (XRD), transmission electron microscopy (TEM), scanning electron microscopy (SEM), as well as neutron activation analysis (NAA) and atomic absorption spectroscopy (AAC) techniques was used for testing the experimental samples. Within the framework of joint studies with the South African Nuclear Energy Corporation (NECSA) NAA of a biomass with gold nanoparticles has been performed on the SAFARI-1 reactor (Pretoria, South Africa).

Materials science. In cooperation with the Scientific and Practical Materials Research Center of the National Academy of Sciences of Belarus the studies of the role of trace impurities in the technology of crystallization of cubic boron nitride continued. Starting from 2011 within the framework of the joint grant (JINR-Romania, 2011) the Romanian specialists in x-ray diffraction and scanning electron microscopy under the direction of Prof. A.Ene (University of Galati, Romania) take part in these studies.

2. NEUTRON SOURCES

THE IBR-2 PULSED REACTOR

In 2011, the activities on the IBR-2 research reactor were carried out in accordance with the tasks of the theme “Development of the IBR-2M reactor with a complex of cryogenic neutron moderators” with the maintenance of the regular operation of all reactor systems. Upon the completion of modernization of the IBR-2 reactor in 2010, during 2011 the physical startup was conducted in steady-state and pulsed modes followed by a successful power startup with the achievement of the designed power of 2 MW. In November-December two cycles of test physical experiments were performed on the extracted neutron beams at the reactor power of 2 MW in order to obtain more accurate and specific user characteristics of the modernized reactor. Also, a set of documents necessary for obtaining the Rostechnadzor license for the regular operation of the reactor has been prepared.

The program of the physical startup of the modernized IBR-2 reactor included the experiments aimed at determining the actual values of the critical parameters of the reactor core, the efficiency of the reactor control units, safety systems, the duration of power pulses and their amplitude fluctuation, as well as a number of other physical characteristics of the reactor necessary for confirmation of its design capabilities and determination of its safe operation margins. The program of the physical startup included the reactor operation in three modes:

- mode of achieving critical mass;
- steady-state power mode in the range of 1-5000 W;
- pulsed mode with a pulse repetition rate of 5 Hz at a mean power of up to 100 kW.

Upon completion of the work under the program of the physical start-up the State Acceptance Commission confirmed the readiness of the reactor for the power start-up. The aim of the power start-up was to verify and specify the design margins and conditions of safe operation of individual units and the reactor as a whole at a power of up to 2 MW.

Within the framework of the program of the power start-up the following measurements were to be carried out:

- determination of absolute power level;
- measurement of thermal characteristics of the reactor;
- measurement of power pulse fluctuations and MR-3 vibrations;
- measurement of power and Na-flow reactivity effects;
- measurement of reactivity balance;
- investigation of characteristics of the safety control system (SCS);
- measurement of isothermal reactivity coefficient;
- measurement of power pulse shape;
- radiation monitoring in the technological and beam outlet areas

In 2011, the work to develop and construct a complex of cryogenic moderators was carried out in two main directions:

1. More than 30 cooling cycles with and without loading mesitylene beads to the simulation chamber were carried out on the test stand installed on channel 3 in the IBR-2 experimental hall. Using the results of these experiments:

a) the technology of full loading of mesitylene beads to the simulation chamber for 3-5 hours has been developed;

b) hydrodynamical and thermophysical properties of the pneumatic conveying system have been studied, which is necessary for designing real units of pneumatic systems for transporting beads to the moderators;

c) the bead movement monitoring system on the basis of differential pressure diaphragm sensors has been developed;

d) the technical documentation necessary for installation of the cryogenic moderator on its regular place has been worked out.

2. Starting-up and adjustment works were continued on the refrigerator facility KGU-700/15.

IREN FACILITY

During the first half of 2011 the experiments on the element and isotope analysis of space dust samples were carried out and the electronics for multichannel detectors of gamma-quanta for nuclear data experiments were developed.

Starting from July, 2011, the activities to put a new klystron Toshiba E3730A into operation have been in progress.

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4. PRIZES AND AWARDS

MISCELLANEOUS


National Aeronautics and Space Administration
Presents the
Group Achievement Award
to
The Lunar Reconnaissance Orbiter (LRO) Team
For exceptional accomplishments in developing and operating the LRO spacecraft and instruments.
V.N. Shvetsov
Signed and Sealed at Washington, DC
this eleventh day of May
Two Thousand Ten
Administrator, NASA

Shvetsov V.N.

The Group Achievement Award of the National Aeronautics and Space Administration of USA for exceptional accomplishments in developing and operating the Lunar Reconnaissance Orbiter spacecraft and instruments

Tropin T.V.

Investigation and Description of Cluster Growth in Polar Solutions of Fullerene 60

Winner of the 2011 Competition for grants of the RF President for State support of young Russian scientists in the section “Physics and Astronomy”

Project “Cryogenic moderator-cold neutron source for investigation of nanostructures”

Winner in the «All-Russian Contest to support high-technology innovation youth projects» (organized by the National Association of Innovations and Development of Information Technologies with the support of the Russian Academy of Sciences)

Project “Creation of a cold bead neutron moderator on the IBR-2 fast pulsed reactor for investigation of nanomaterials and condensed matter”

Winner in the All-Russian Contest of innovation youth projects «Zvorykinsky award» in the nomination «Nuclear technologies»

The following FLNP employees were awarded with:

- badge “Academician I.V. Kurchatov”, class 4 – FLNP Chief Engineer **A. V. Vinogradov**
- badge of honor “For Service to Dubna” – FLNP Directorate Adviser **V. D. Ananiev**
- title “Honorary JINR Staff Member – FLNP leading researcher **Yu. N. Pepelyshev**

At every session of the Programme Advisory Committees for Condensed Matter Physics and Nuclear Physics a competition is organized for the best poster presented in the poster session for young scientists.

4. PRIZES AND AWARDS

T. Murugova's presentation at the 110th Session of the JINR Scientific Council, September 15-16, 2011

The posters "Structure peculiarities of α-crystalline studied by small-angle neutron and X-ray scattering" presented by T. Murugova and "Microbial synthesis of silver nanoparticles *Streptomyces glaucus* and *Spirulina platensis*" presented by I. Zinicovscaia were selected as the best posters by the 34th session of the PAC for Condensed Matter Physics and for Nuclear Physics, respectively, and recommended for oral presentation at the JINR Scientific Council.

JINR AND FLNP FELLOWSHIPS

Since 2010, the Association of Young Scientists and Specialists of JINR has been annually organizing a competition for scholarships in four categories. In 2011 the scholarships were awarded to:

1. grant for young PhD researchers
   - S.E. Kichanov
   - N.O. Ryabova
2. grant for young researchers
   - I.A. Bobrikov
   - A.V. Rogachev
   - Yu.S. Pankratova
   - D.V. Kustov
3. grant for young specialists
   - A.V. Kutergin
   - M.V. Bulavin
   - K.V. Udovichenko
4. grant for young workers
   - D.V. Kokunov
   - M.A. Kulikov

Since 2002, in FLNP a scholarship named after Academician of the USSR Academy of Sciences and first Director of the Laboratory of Neutron Physics **I.M. Frank** has been established in order to stimulate scientific and methodical research of young scientists.

In 2011 I.M. Frank scholarships were awarded to:

- In Neutron Nuclear Physics
  I.I. Zinikovskaia
- In Condensed Matter Physics
  A.V. Rogachev
- In Methodical Investigations
  L.A. Taibov

Since 2006, two scholarships have been founded to immortalize the memory of outstanding scientist, Corresponding Member of the USSR Academy of Sciences **F.L. Shapiro**.

4. PRIZES AND AWARDS

One of them is awarded annually to two young FLNP employees in the following research directions: UCN physics; polarized neutrons; neutron spectroscopy.

In 2011 F.L. Shapiro scholarships were awarded to:

- In «Polarized Neutrons» V.I. Petrenko
- In «Neutron Spectroscopy» Yu.V. Alekseenok

The other is awarded each semester to one student and one PhD student of the University Centre carrying out research studies in FLNP.

The following FLNP employees received this scholarship in 2011:

- Students Samoilenko S.A.
  Tomchuk A.V.
- PhD student Verkhogliadov A.Ye.

JINR PRIZES*

JINR Prizes are awarded annually for the best scientific, technical, methodical and applied research studies. In 2011, the following studies performed by the FLNP specialists or in collaboration with the employees of other Laboratories were awarded with the prizes of various degrees:

*the authors from FLNP are marked in bold

Experimental physics research:

First Prize

«Observation of the new type of ternary decay of heavy nuclei»

Authors: D.V. Kamanin, Yu.V. Pyatkov, A.A. Alexandrov, I.A. Alexandrova, V.E. Zhuchko, N.A. Kondratjev, **Yu.N. Kopatch**, E.A. Kuznetsova, W. Trzaska, W. von Oertzen

Scientific and technical applied research:

Second prize

«Proposal and investigation of new materials - low-temperature fluoropolymers for the ultracold neutrons storage chambers with very low neutron losses used in the new precision neutron lifetime measurement»

Author: **Yu.N. Pokotilovski**

Encouraging prizes

«Handbook of Neutron Optics"»

Authors: Masahiko Utsuro, **V.K. Ignatovich**

«The spin-state transitions and magnetic ordering in complex cobalt oxides»

Authors: **D.P. Kozlenko, N.O. Golosova, B.N. Savenko, S.E. Kichanov, E.V. Lukin, V.Yu. Kazimirov,** Z. Jirak

4. PRIZES AND AWARDS

FLNP PRIZES

Nuclear Physics:

First Prize

«Measurement of $^{149}Sm(n,a)^{146}Nd$ reaction cross section in the neutron energy range of 4 – 7 MeV»

Authors: Yu.M. Gledenov, G. Chjan, G. Huuhenhuu, M. Sedysheva, P. Shalansky, P. Keller, Yu.N. Voronov

Second Prize

«Dynamic effects in the interaction of neutron waves with normal and doubly refracting samples moving with acceleration»

Authors: A.I. Frank, D.V. Kustov, G.V. Kulin

Third Prize

«A proposed experiment on ball lightning model»

Authors: V.K. Ignatovich, F.V. Ignatovich

Applied and methodical research:

First Prize

«Differential time-of-flight spectrometer of very slow neutrons»

Authors: Yu.N. Pokotilovskyi, M.I. Novopoltsev, P. Geltenbort, T.A. Brenner

Second Prize

«Investigation of structural aspects in the formation of oxide nanoparticles in silicate glasses doped with cerium and titanium oxides»

Authors: S.A. Samoilenko, S.Ye. Kichanov, A.V. Belushkin, D.P. Kozlenko, V.M. Garamus, Ye.A. Trusova, G.P. Shevchenko, V.S. Gurin, L.A. Bulavin, S.K. Rakhmanov, B.N. Savenko

Encouraging prize

Author: Sh. Zeynalov.

Condensed matter physics:

First Prize

«Theoretical and experimental investigations of propagation of polarized neutrons in a layered structure placed in crossed static and oscillating magnetic fields»

Authors: V.K. Ignatovich, S.V. Kozhevnikov, Yu.V. Nikitenko, F. Radu

Second Prizes

«Effects of structural disorder in fine-grained HTSC $YBa_2Cu_3O_y$»

Authors: A.M. Balagurov, L.G. Mamsurova, I.A. Bobrikov, To Than Loan, V.Yu. Pomyakushin, K.S. Pigalskyi, N.G. Trusevich, A.A. Vishnev.

«Effects of formation of clusters and charge transfer complexes in C60/NMP solutions»

Authors: T.V. Tropin, E.A. Kyzyma, T.A. Kyrey, M.V. Avdeev, V.L. Aksenov

4. PRIZES AND AWARDS

Third Prize

«Antipolar phase in multiferroic BiFeO3 at high pressures»

Authors: D.P. Kozlenko, A.A. Belik, A.V. Belushkin, Ye.V. Lukin, G. Marshall, B.N. Savenko, E. Takayama-Muromachi.

Encouraging prize

«Investigation of deterministic fractals: extracting additional information from small-angle scattering data»

Authors: A.Yu. Chernyi, E.M. Anitas, V.A. Osipov, M. Balasoiu, A.I. Kuklin

AYSS PRIZES

Annually the Association of Young Scientists and Specialists (AYSS) organizes a competition for the best scientific, methodical and scientific and applied research studies performed by young scientists and specialists of the Joint Institute for Nuclear Research. In 2011, our employee **S.Ye. Kichanov** won the Second Prize in the nomination “scientific and technical applied research”.

2011

Annual Report

6. ORGANIZATION

STRUCTURE OF LABORATORY AND SCIENTIFIC DEPARTMENTS

Directorate:	
Director	*A.V. Belushkin*
Deputy Director	*V.N. Shvetsov*
Deputy Director	*Deleg Sangaa*
Deputy Director	*S.V. Kozenkov*
Chief engineer:	*A.V. Vinogradov*
Scientific Secretary	*O.A. Culicov*
Laboratory Scientific Leader	*V.L. Aksenov*
Advisor to Directorate	*V.D. Ananiev*

Reactor and Technical Departments	Head
IBR-2 reactor	Chief engineer: *A.V. Dolgikh*
Mechanical maintenance division	*A.A. Belyakov*
Electrical engineering department	*V.A. Trepalin*
Design bureau	*A.A. Kustov*
Experimental workshops	*A.N. Kuznetsov*

Scientific Departments and Sectors	Head
Department of neutron investigation of condensed matter	*D.P. Kozlenko*
Nuclear physics department	*V.N. Shvetsov*
Department of IBR-2 spectrometers complex	*S.A. Kulikov*

Administrative Services
Secretariat
Finances
Personnel

Scientific Secretary Group
Secretariat
Translation
Graphics

6. ORGANIZATION

DEPARTMENT OF NEUTRON INVESTIGATION OF CONDENSED MATTER

Sub-Division	Title	Head
Sector 1: Neutron Diffraction. Head: *A.M. Balagurov*		
Group No.1	HRFD	*A.M. Balagurov*
Group No.2	DN-2	*A.I. Beskrovnyi*
Group No.3	DN-12	*B.N. Savenko*
Group No.4	Geomaterials	*A.N. Nikitin*
Group No.5	SKAT /Epsilon	*Ch. Scheffzük*
Sector 2: Neutron Optics. Head: *M.V. Avdeev*		
Group No.1	Physics of Surfaces	*Yu.V. Nikitenko*
Group No.2	Physics of Nanostructures	*M.V. Avdeev*
Small angle scattering group		*A.I. Kuklin*
Inelastic scattering group		*I. Natkaniec*

NUCLEAR PHYSICS DEPARTMENT

Sub-Division	Title	Head
Sector 1. Correlation γ-spectroscopy and development of experimental installations. Head: *N.A. Gundorin*		
Sector 2. Investigation of neutron properties. Head: *Ye.V. Lychagin*		
Sector 3. Neutron activation analysis. Head: *M.V. Frontasyeva*		
Group No.1	Analytical	*M.V. Frontasyeva*
Group No.2	Experimental	*S.S. Pavlov*
IREN facility		*V.G. Pytaev*

DEPARTMENT OF IBR-2 SPECTROMETERS COMPLEX

Sub-Division	Title	Head
Group No.1	Detectors	*A.V. Churakov*
Group No.2	Electronics	*A.A. Bogdzel*
Group No.3	Information technologies	*A.S. Kirilov*
Group No.4	Sample environment and choppers	*A.P. Sirotin*
Group No.5	Cryogenic investigations	*A.N. Chernikov*
Group No.6	Cold moderators	*S.A. Kulikov*

6. ORGANIZATION

PERSONNEL

Distribution of the Personnel per Department as of 31.12.2011

Theme	Departments	People
-1104-	Nuclear Physics Department	79
-1069-	Department of neutron investigation of condensed matter	85
-1075-	Department of IBR-2 spectrometers complex	46
-1105-	IBR-2 reactor	46
	Mechanical and Technical Department Electric and Technical Department Central Experimental Workshops Nuclear Safety Group Design Bureau	45 28 37 7 6
	FLNP infrastructure: Directorate Services and Management Department Scientific Secretary Group Supplies Group	 9 24 3 4
Total		**419**

Personnel from the JINR Member States (besides the RF) as of 31.12.2011

Country	People	of which young specialists (<35 years)
Azerbaijan	8	8
Belarus	1	1
Bulgaria	5	2
Georgia	2	
Germany	2	
Moldova	2	2
Kazakhstan	3	3
Mongolia	9	6
Poland	5	
Romania	6	1
Ukraine	10	10
Serbia	1	1
Vietnam	2	2
TOTAL	**56**	**36**

6. ORGANIZATION

Our PhD students

In 2011 18 PhD students conducted experimental research at the FLNP facilities.

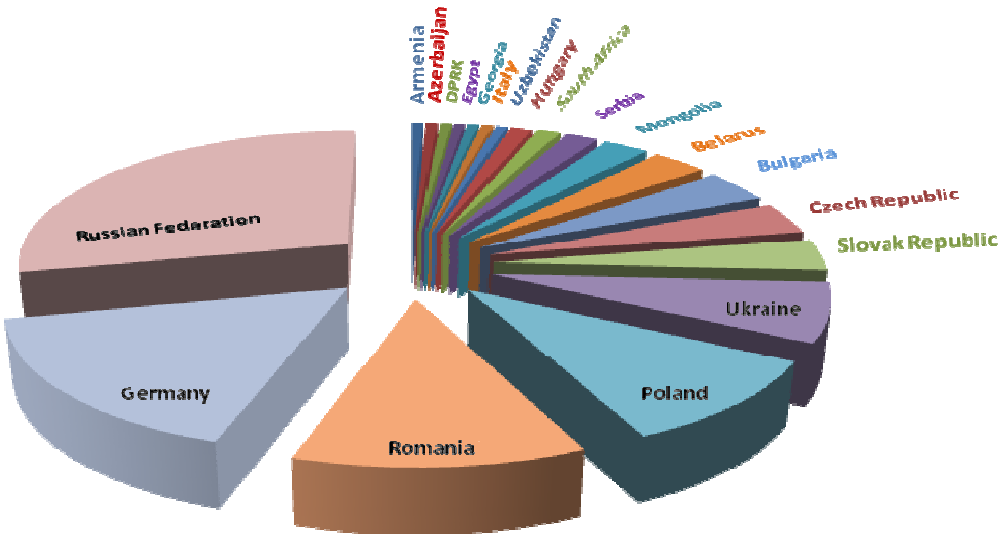
Name	Country	PhD student of
Djabarov S.G.	Azerbaijan	Institute of physics of the Azerbaijan Academy of Sciences
Milkov V.M.	Bulgaria	University of Sofia
Anghel L.	Moldavia	Institute of chemistry of the Moldavian Academy of Sciences
Zinikovscaia I.	Moldavia	University of the Moldavian Academy of Sciences
Neamsuren B.	Mongolia	National University of Mongolia
Erhan R.V.	Romania	University of Bucharest
Gruzinov A.Yu.	Russia	JINR University centre
Verhogleadov A.E.	Russia	JINR University centre
Bulavin V.V.	Russia	Tula National University
Mukhin K.A.	Russia	JINR University centre
Goriunov S.V.	Russia	JINR University centre
Goriainova Z.I.	Russia	Institute of ecology and evolution of the Russian Academy of Sciences
Nagornyi A.V.	Ukraine	National University of Kyiv
Solovev D.V.	Ukraine	National University of Kyiv
To Than Loan	Vietnam	Tula National University
Fan Thi Ngok Loan	Vietnam	Tula National University
Chan Tuan An	Vietnam	Tula National University
Dang Ngok Toan	Vietnam	Tula National University

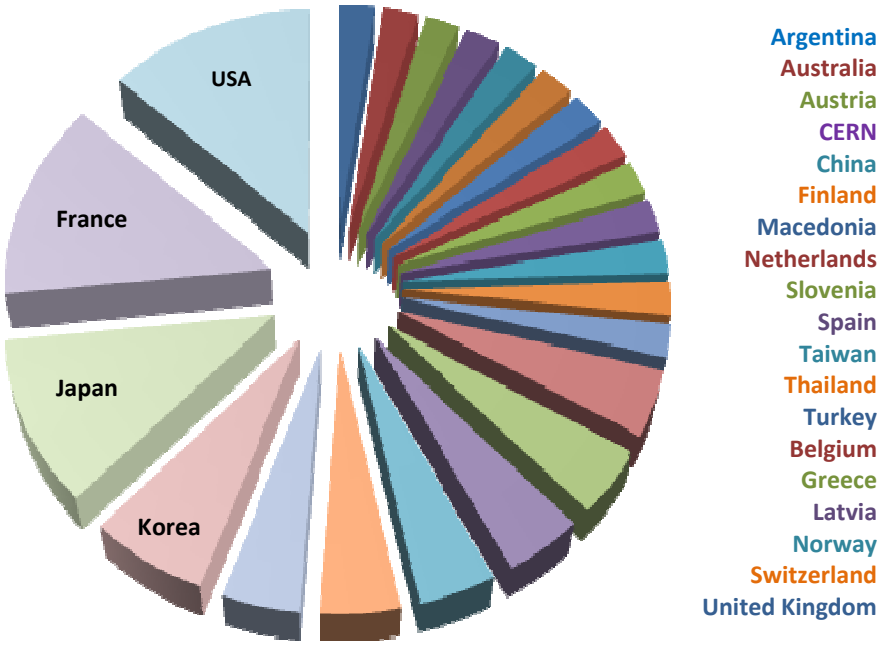
In 2011, 5 BSc and 5 MSc theses were defended using the experimental material obtained in FLNP. Two of our employees were conferred a Doctor of Science degree.

7. INTERNATIONAL COOPERATION AND USER INTERACTION

INTERNATIONAL COOPERATION

In 2011 the Frank Laboratory of Neutron Physics collaborated with 158 institutions from 20 JINR Member States or Associated Members of JINR and 45 institutions from 23 Non-Member States of JINR. The distribution of the institutions by country is presented below.





7. INTERNATIONAL COOPERATION AND USER INTERACTION

List of Visitors from the JINR Member States or Associated Members of JINR in 2011

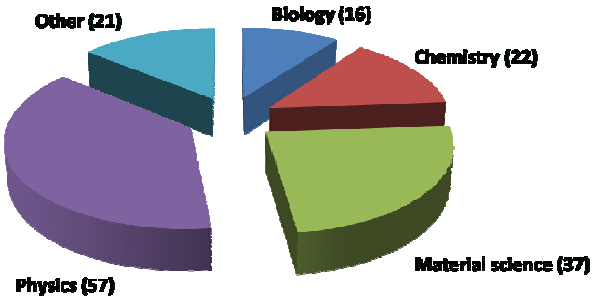
Country	Nr of visitors
Azerbaijan	1
Belarus	3
Bulgaria	2
Czech Rep.	8
Egypt	1
Germany	4
Hungary	1
Mongolia	3
Poland	9
Romania	14
Serbia	1
Slovak Republic	1
Ukraine	3

List of Visitors from Non-Member States of JINR in 2011

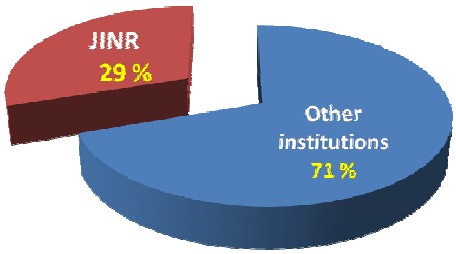
Country	Nr of visitors
France	1
Japan	2
Republic of Korea	2
Taiwan	4
United Kingdom	1

USER INTERACTION

The first call for proposals for experiments at the modernized IBR-2 reactor was open from November 15 to January 31. A total of 153 proposals from 17 countries were submitted.



Proposal distribution by science



Proposal distribution by applicant's affiliation

8. FLNP AND MASS-MEDIA

The most significant FLNP events in 2011 – the completion of the IBR-2 physical start-up and the power start-up of the reactor – were covered by:

- leading **Russian News agencies**

РИАНОВОСТИ www.ria.ru

- http://center.ria.ru/science/20110705/82438871.html
- http://ria.ru/science/20110705/397695590.html

NEWSru.com

- http://www.newsru.com/russia/05jul2011/reactor.html

- **television channels** with national (like Russia 1) or regional (like TV Podmoskovie and Dubna TV) audience

Reportage: Bridling a nuclear dragon 13.03.2011

05.07.2011

- and in **electronic publications**:
  - http://www.vokrugsveta.ru/news/12439
  - http://www.i-russia.ru/all/articles/6516/
  - http://www.nanonewsnet.ru/news/2011/v-dubne-zapushchen-unikalnyi-issledovatelskii-reaktor
  - http://vz.ru/news/2011/7/5/504927.html

Several news reports about the II International Scientific School for Young Scientists and Students "Instruments and Methods of Experimental Nuclear Physics. Electronics and Automatics of Experimental Facilities" (November 07-09, Dubna) were published:

- http://rusnanonet.ru/news/66954/
- http://www.nanonewsnet.ru/news/2011/eksperiment-bez-sovremennykh-priborov-ne-postavit
- http://www.mosreg.ru/news/65522.html
- http://dubna-oez.ru/news/177.htm
- http://dubna.ru/34/8791.html

8. FLNP AND MASS-MEDIA

Representatives of various mass-media were interested in the opinion of our specialists on the problem of nuclear safety in the world (TV Dubna, NTV, TV Podmoskovie) and about the development of the Skolkovo project (REN TV).
The international scientific community could get news on the FLNP events from the world wide distributed journals like:

Neutron News

All-Russian Neutron School for Young Scientists and Students on Modern Neutron Diffraction Studies
Volume 22, Issue 2, pages 3-11
http://www.tandfonline.com/doi/full/10.1080/10448632.2011.568865

User Meeting of Small-angle Neutron Scatterers at JINR, Dubna
Volume 22, Issue 4, pages 4-9
http://www.tandfonline.com/doi/full/10.1080/10448632.2011.617263

Pelletized Mesitylene-based Cold-neutron Moderator
Volume 22, Issue 2, pages 26-32
http://www.tandfonline.com/doi/full/10.1080/10448632.2011.570584

Nuclear Physics News

19th International Seminar on Interaction of Neutrons with Nuclei
Volume 21, No 3, pages 37-38
http://www.nupecc.org/npn/npn213.pdf

NMI 3 Newsletter

FLNP and JINR re-open the IBR-2 reactor
Issue 1, July 2011, pages 34-35
http://nmi3.eu/news-and-media/newsletter-.html

IBR-2 user programme resumes
Issue 2, November 2011, pages 34-35
http://nmi3.eu/news-and-media/newsletter-.html

The bilingual bulletin *News of the Joint Institute for Nuclear Research* and the weekly newspaper *"DUBNA: Science, Cooperation, Progress"* published by JINR also regularly include reports on the FLNP events and achievements.

9. ANNIVERSARIES

YU.M. OSTANEVICH

Yu.M. Ostanevich would celebrate his 75th birthday in 2011. His colleagues remember him as a remarkable person "with a unique ability of being himself and expressing his individuality in any situation, whether ordinary or unusual" – they wrote in the book of memoirs "Yu.M. Ostanevich: Scientist. Teacher. Friend." published in 2002. An example of lifetime recognition and high appreciation of his work was the speech of the Corresponding Member of the USSR Academy of Sciences F.L. Shapiro, the first Deputy Director of the Laboratory of Neutron Physics and one of its founders made at the Doctor Thesis defense of Yu.M. Ostanevich:

"I would like to tell a few words about Yu.M. Ostanevich as a physicist of our Laboratory. He is one of our greatest physicists. His distinctive feature is that it is difficult to find a research field where he would not be an expert, no matter whether it is in the application of computers or in cryogenic equipment. He is not afraid of excursuses into the theory plunging himself into the theory of relativity, nuclear physics or condensed matter theory. He devises challenges for himself and then successfully solves them. The fact that he defends his thesis after 12 years of work tells us that he is deeply preoccupied with various ideas and problems. His dissertation is a very interesting study on the liquid-gas critical state and he is an excellent specialist in neutron techniques. I believe that Yu.M. Ostanevich is not just a Doctor of Science according to his qualification, but an outstanding Doctor of Science."

The results of the voting supported this high appraisal. He was awarded with the degree of Doctor of Science omitting the PhD degree.

Key events in his research activity:

1953-1959	student of the Physics Department of the Moscow State University
1957-1959	engineer of the Institute of Nuclear Physics of MSU
1959-1960	senior laboratory assistant in the Laboratory of Neutron Physics
1960-1966	junior researcher
1966-1970	researcher
1970-1992	head of the Department of Neutron Measurements (later the Department for Neutron Investigations of Condensed Matter)
1971	Doctor of Science in Physics and Mathematics
1990	Professor of the Moscow Engineering Physics Institute (MEPhI)
2000	Laureate of the RF State Prize for the development and realization of new methods in time-of-flight neutron diffraction studies at pulsed and steady-state nuclear reactors (posthumously) (together with V.L. Aksenov, A.M. Balagurov, V.P. Glazkov, V.A. Kudryashov, V.V. Nietz, V.A. Somenkov, V.A. Trunov**)**

Inventions:

1970	Method for determination of the ratio of low currents. Authors: V.N. Zamrii, V.I. Lazin, Yu.M. Ostanevich
1977	Wire detector of slow neutrons. Authors: B.N. Ananiev, Y.M. Ostanevich, Ye.Ya. Pikelner
1980	Method of studying holograms without a reference beam. Authors: V.K. Ignatovich, Yu.M. Ostanevich, M.I. Podgoretskii

9. ANNIVERSARIES

YE.P. SHABALIN

To the 75th birthday jubilee of, a leading scientist of the Frank Laboratory of Neutron Physics.
Fragment from the article by A.V. Strelkov.

In the middle of 1959 a young promising specialist Zhenya Shabalin came to the Laboratory of Neutron Physics (LNP). After graduation from school with a gold medal for academic excellence, he was uncertain about his future profession. He was fascinated with the arts and even thought of becoming a film director. But on further consideration he entered the Moscow Engineering Physics Institute (MEPhI). After graduation he came to Dubna. At that time in LNP the construction of the world's first pulsed reactor IBR-2 was nearing completion and a young specialist Zhenya Shabalin took an active part in the start-up of the reactor. The start-up group was relatively small and Zhenya had the chance to contact closely with legendary by that time physicists D.I. Blokhintsev, I.M. Frank, F.L. Shapiro, Yu.Ya. Stavissky. This helped him to quickly grasp the principle of operation and design of the reactor, so that Zhenya, despite his young age, became a leading physicist and an authority for the reactor staff as soon as the IBR started its operation. An outstanding physicist and Deputy Director of LNP F.L. Shapiro knew for sure who he should ask to evaluate the possibility in principle of creating a new IBR that would be 1000 times more powerful than the old one and invited Shabalin to do it. Zhenya and his colleagues performed some calculations and showed that Shapiro's idea could be realized. Later on, the possibility of practical realization of construction of a more powerful reactor (IBR-2) was supported by D.I.Blokhintsev's enthusiasm. During the construction of IBR-2 Ye.P. Shabalin became close with D.I. Blokhintsev discussing with him not only some technical problems connected with the creation of a new IBR, but also some questions of science, history and arts. By then having become a mature engineer-physicist, he did not give up his youth's passion for the arts. He tried to make movies and cartoons at the film studio "Dubfilm", took part in the stage direction process and successfully played roles in the amateur satirical performances.

Meanwhile the old IBR was shut down for reconstruction into IBR-30 and Ye.P. Shabalin took an active participation in the work. In 1971 Ye.P. Shabalin was among the group of authors awarded with the USSR State Prize for the creation of a pulsed reactor with an electron accelerator. Ye.P. Shabalin played a special role at the final stage of the construction of IBR-2. The initial rated power of IBR-2 was 4 MW, however because of overcautiousness for the State Acceptance Committee the reactor was decided to be commissioned at a power of only 1 MW, which in the opinion of the project leaders would significantly enhance the safety of the reactor operation. But then, at the crucial moment a chief physicist of the project Ye.P. Shabalin appeared uninvited at the Committee meeting and proved and convinced its members that IBR-2 could operate at a power of 2 MW. Later on he received a severe reprimand from the security authorities who had overlooked the appearance of Ye.P. Shabalin at the meeting. The IBR-2 had operated for 20 years without any serious troubles and was successfully modernized. During this time Ye.P. Shabalin wrote a monograph on pulsed reactors, discovered a stochastic instability of the pulsed reactor behavior, suggested a number of devices, which improve the efficiency of the operation of a reactor as a neutron source. One of these devices is the so-called "cold" moderator, which makes it possible to increase many times the portion of slow neutrons that are the most useful in the majority of the experiments carried out at the reactor. At present, the installation of the cold moderator suggested by Ye.P. Shabalin is in progress.

Advancing in years Ye.P. Shabalin is not fond of some youth hobbies any more: football, photography, boat trips, but he has not lost his interest in literature yet. He wrote and published two adventure novels and a book of poems. And at present Ye.P. Shabalin is the chairman of the JINR Museum Council.

10. EXPERIMENTAL REPORTS*

DEPARTMENT OF NEUTRON INVESTIGATION OF CONDENSED MATTER

SAXS STUDIES OF ULTRASONICATED DISPERSIONS OF BIOMINERAL FERRIHYDRITE NANOPARTICLES USING THE ATSAS SOFTWARE PACKAGE ANALYSIS

L. Anghel, M. Balasoiu, A.V. Rogachev, T.S. Kurkin, A.I. Kuklin, L.A. Ishchenko, S.V. Stolyar, R.S. Iskhakov, Yu.L. Raikher, G.M. Arzumanian

CATION DISTRIBUTION IN Zn-SUBSTITUTED Ni-Ga-Fe SPINEL

S.S. Ata-Allah, A.M. Balagurov, A. Hashhash, I.A. Bobrikov, V.G. Simkin, Sh. Hamdy

SANS STUDY OF BIOCOMPATIBLE MAGNETIC FLUIDS STABILIZED WITH POLY(ETHYLENE GLYCOL)

M.V. Avdeev, V.I. Petrenko, A.V. Nagornyi, V.M. Garamus, A.V. Feoktystov, V. Závišová, M. Koneracká, G. Lancz, N. Tomašovičová, M. Timko, P. Kopčanský

MAGNETIC FIELD AND PARTICLE CONCENTRATION COMPETITIVE EFFECTS ON FERROFLUID BASED SILICONE ELASTOMER MICROSTRUCTURE

M. Balasoiu, I. Bica, V.T. Lebedev, A.I. Kuklin, Yu.L. Raikher

NEUTRON DIFFRACTION STUDY OF $LiFePO_4$ CATHODE MATERIAL DOPED WITH VANADIUM OXIDE

I.A. Bobrikov, V.G. Simkin, Chih-Hao Lee, Chi-Wei Hu, Tsan-Yao Chen, Sangaa Deleg, A.M. Balagurov

NEUTRON DIFFRACTION STUDY OF ATOMIC AND MAGNETIC STRUCTURES OF $La_{1-x}Sr_xFe_{2/3}Mo_{1/3}O_3$

I.A. Bobrikov, V.G. Simkin, S.Ya. Istomin, V.V. Vishnyakova, M.V. Lobanov, E.V. Antipov, A.M. Balagurov

THE STRUCTURAL STUDIES OF ANTIFERROELECTRIC-FERROELECTRIC PHASE TRANSITION IN SODIUM NIOBATE

S.G. Jabarov[a], D.P. Kozlenko, S.E. Kichanov, A.I. Mamedov, B.N. Savenko, R.Z. Mextieva, C. Lathe

THE STRUCTURAL STUDIES OF $Y_3Al_5O_{12}:Ce^{3+}/Lu_2O_3$ PHOSPHORS SYNTHESIZED BY COLLOID-CHEMICAL METHOD

S.E. Kichanov, E.V.Frolova, G.P.Shevchenko, D.P.Kozlenko, A.V.Belushkin, E.V.Lukin, G.E.Malashkevich, S.K.Rakhmanov, V.P.Glazkov and B.N.Savenko

EXPERIMENTAL DETERMINATION OF NEUTRON CHANNELING LENGH IN PLANAR WAVEGUIDE

S.V. Kozhevnikov, F. Ott, A. Rühm, and J. Major

RIETVELD TEXTURE ANALYSIS OF SKAT DIFFRACTOMETER DATA: RECENT ADVANCES AND PERSPECTIVES

S. Matthies, R.N. Vasin

DEVELOPMENT OF THE SESANS SPECTROMETER ELEMENTS BASED ON ROTATING MAGNETIC FIELDS

A. Rubtsov, A. Ioffe, V. Bodnarchuk, S. Manoshin

NEUTRON AND SYNCHROTRON X-RAY DIFFRACTION STUDY OF MODEL ORAL STRATUM CORNEUM LIPID MIXTURES

N.Yu. Ryabova, A.Yu. Gruzinov, A.V. Zabelin, S.G. Sheverev, A. Buchsteiner, T. Haußß

ISOTHERMAL COMPRESSIBILITY AND THICKNESS OF LIPID BILAYER SIMULTANEOUSLY MEASURED UNDER HYDROSTATIC PRESSURE

D.V. Soloviov, O.I. Ivankov, Yu.E. Gorshkova, T.B. Petuhova, Yu.S. Kovalev, A.P. Sirotin, V.I. Gordeli, A.I.Kuklin

STRUCTURE OF CLUSTERS IN AQUEOUS DISPERSIONS OF NANODIAMONDS BY SMALL-ANGLE NEUTRON SCATTERING: EXPONENTIAL/POWER-LAW APPROACH

O.V. Tomchuk, L.A. Bulavin, V.M. Garamus, V.L. Aksenov, M.V. Avdeev

***All experimental reports are published as submitted by the authors.**

10. EXPERIMENTAL REPORTS*

STUDY OF C_{60}/NMP/TOLUENE AND C_{60}/NMP/WATER SOLUTIONS BY UV-VIS SPECTROSCOPY AND SMALL-ANGLE NEUTRON SCATTERING
T.V. Tropin, T.O. Kyrey, O.A. Kyzyma, A.V. Feoktistov, M.V. Avdeev, L.A. Bulavin, L. Rosta, V.L. Aksenov

UPGRADE OF THE NEUTRON OPTICAL SYSTEM FOR THE DIFFRACTOMETERS Epsilon-MDS AND SKAT AT THE REACTOR IBR-2
K. Walther, Ch. Scheffzuek, F. Shilling, A. Frischbutter, A.P. Bulkin, V.A. Kufryashov, V.V. Shuravlev, A.V. Belushkin

NUCLEAR PHYSICS DEPARTMENT

NEUTRON ACTIVATION ANALYSIS OF SEDIMENTS AND ROCKS FROM TWO LAKES OF ROMANIA
O.G. Duliu, S.M. Lyapunov, M.V. Frontasyeva

APPLICATION OF SOME MICROORGANISMS FOR SYNTHESIS OF GOLD AND SILVER NANOPARTICLES
M.V. Frontasyeva, S.S. Pavlov, I.I. Zinicovscaia, E.I. Kirkesali, T. Kalabegishvili, I. Murusidze, A. Faanhof

***All experimental reports are published as submitted by the authors.**

SAXS STUDIES OF ULTRASONICATED DISPERSIONS OF BIOMINERAL FERRIHYDRITE NANOPARTICLES USING THE ATSAS SOFTWARE PACKAGE ANALYSIS

L. Anghel[a,b], M. Balasoiu[a,c], A.V. Rogachev[a], T.S. Kurkin[d], A.I. Kuklin[a], L.A. Ishchenko[e,f], S.V. Stolyar[e,f], R.S. Iskhakov[e,f] , Yu.L. Raikher[g], G.M. Arzumanian[a]

[a]Joint Institute of Nuclear Research, Dubna, 141980, Russia
[b]Institute of Chemistry of ASM, Chisinau, Republic of Moldova
[c]Horia Hulubei National Institute of Physics and Nuclear Engineering, Bucharest, Romania
[d]Institute of Synthetic Polymer Materials RAS, 117393, Moscow, Russia
[e]Siberian Federal University, 660041, Krasnoyarsk, Russia
[f]Kirensky Institute of Physics, Siberian Branch of RAS, 660036, Krasnoyarsk, Russia
[g]Institute of Continuous Media Mechanics, Ural Branch of RAS, 614013, Perm, Russia

New methods are developing to control disparity, chemical composition, the size, and the shape to get the best particles which can be well applied in different fields of science. A promising new dimension in this field is the use of microorganisms for the production of inorganic nanoscale particles. A growing need to understand the basics of this technique to facilitate application of the new methodology to laboratory and industrial needs is present.

This paper presents SAXS studies of ultrasonicated dispersions of biomineral ferrihydrite nanoparticles produced by bacteria *Klebsiella oxytoca* using the ATSAS software package [1] analysis.

The SAXS intensity is experimentally determined as a function of the scattering vector q whose modulus is given by $q = 4\pi \sin\theta / \lambda$, 2θ is the scattering angle and λ the wavelength of the incident X-ray beam. For an ensemble of structurally homogenous, randomly oriented particles, the intensity of scattered radiation can be expressed as:

$$I(q) = N(\Delta\bar{\rho}V)^2 P(q) S(q) \quad (1)$$

In Eq. (1), N is the number of particles per unit volume; $\Delta\bar{\rho}$ is the contrast, which reflects the difference between the electronic density of the scattering components; and V is the volume of each particles; $P(q)$, the form factor, encodes the ensemble average structure of the particles in reciprocal space; and $S(q)$, the structure factor, encodes correlation distances between particles in the reciprocal space.

SAXS measurements on ferrihydrite nanoparticles were performed at the Brucker Nanostar SAXS spectrometer in function at the Institute of Synthetic Polymer Materials RAS, Moscow. The experimental setup covered the q range 0.007 – 0.23 Å^{-1}. The experimental scattering curves presented in Fig. 1, were analyzed using the software ATSAS 2.4, which is suitable for small angle scattering data analysis. In order to eliminate concentration or aggregation influence on the modeled experimental data, a linear extrapolation to zero concentration was done for the scattering data obtained on the samples with different concentrations of ferrihydrite particles. In this case, the structure factor $S(q)$ becomes equal to 1 over the whole q range, and Eq. (1) reduces to:

$$I(q) = N(\Delta\bar{\rho}V)^2 P(q) \quad (2)$$

Radius of gyration, R_G, which represents the distance of the scattering object parts from its center of gravity, was calculated in a q range of 0.024–0.034 Å^{-1} using the Guinier approximation:

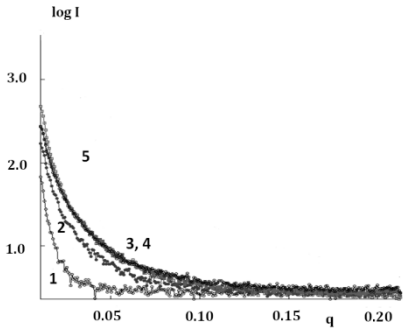


Fig.1. SAXS scattering curves from ferrihydrite samples at different concentration: buffer (1); ferrihydrite concentration 3.70×10^{-3}M (2); 5.54×10^{-3}M (3); 7.41×10^{-3}M (4); 1.48×10^{-2}M (5) plotted by PRIMUS; the logarithm of the scattering intensity $I(q)$ vs modulus of the scattering vector q is shown.

$$I(q) = I(0)\exp(-q^2R_G^2/3) \quad (3)$$

A rough value of 6.41±0.13 nm for the radius of gyration independently of the shape of the investigated particles was obtained. In order to calculate more precisely the values of R_G, the pair distribution function of biomineral nanoparticles was computed using the fitting procedures included in GNOM software from ATSAS package. Typically, the particle distance distribution function, $p(r)=\gamma(r)r^2$, where $\gamma(r)$ is the characteristic function of the particles, is calculated by an indirect Fourier transformation to avoid problems due to the discrete sampling of the $I(q)$ curve over a finite range. The indirect Fourier transform essentially constructs trial $p(r)$ functions that are Fourier transformed and evaluated in comparison with the experimental scattering. In the GNOM program, a regularizing multiplier is used to balance the smoothness of the trial $p(r)$ function with the goodness of the fit to the data. Thus, the radius of gyration is obtained from the $p(r)$ function using formula:

$$R_G^2 = \int_0^{D_{max}} r^2 p(r)dr \Big/ \int_0^{D_{max}} p(r)dr \quad (4)$$

In Eq. (4), D_{max} denotes the maximum distance inside the scattering particle. This method to determine R_G takes into accounts all of the collected experimental data, not only those limited to small q domains, as is used in the Guinier approximation. Calculated $p(r)$ distribution function in the q range 0.02 – 0.26 $Å^{-1}$ is presented in Fig. 2.

The elongated tail of the $p(r)$ function within the r - range of 120 – 200 Å indicates the presence of macromolecules in the scattering solutions. This fact, that biogenic nanoparticles removed from bacterium *Klebsiella oxytoca* are still wrapped in an organic sheath, was previously shown by HRTEM analysis. We remark also that the value of radius of gyration of 6.73±0.16 nm calculated from pair-distribution function is close to those obtained earlier from the Guinier approximation.

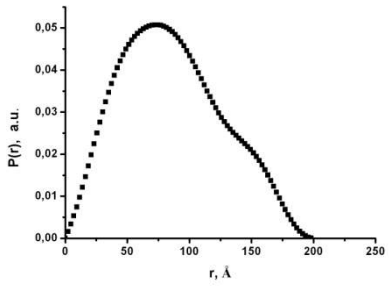


Fig.2. Function $p(r)$ calculated from the scattering curve obtained from extrapolation to zero concentration

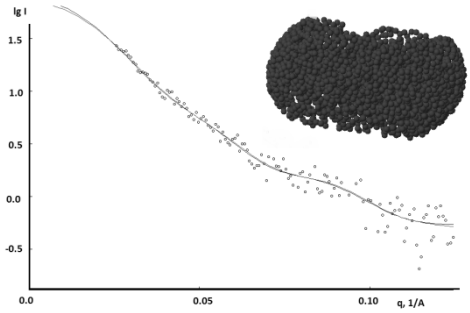


Fig.3. *ab initio* reconstruction of water dispersed ferrihydrite nanoparticles.

The overall shape of the particles was further computed by the program GASBOR. This software performs an *ab initio* reconstruction of molecular structure by a chain-like ensemble of *dummy residues*. Fig. 3 displays the resulting plot for the scattering curve obtained from a sample of biogenic ferrihydrite nanoparticles. The identified elongated 3D object resembles quite closely the rod-like model reported earlie. As any other method that generates 3D structures from the 1D scattering data, the resulting pattern is not unique and might be optimized with supplementary experimental data.

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CATION DISTRIBUTION IN Zn-SUBSTITUTED Ni-Ga-Fe SPINEL

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Spinels with $A^{2+}B^{3+}{}_2O_4$ formula are ternary oxides that have numerous and important technological applications, especially as magnetic, super-hard and high-temperature materials. From physical point of view, the cubic-to-tetragonal phase transformation and Jahn–Teller (JT) distortion in the tetragonal phase have attracted considerable attention during many years. For instance, the $NiFe_2O_4$ compound for the first time has been investigated by means of neutron diffraction about 60 years ago [1].

In the *normal* cubic spinel structure the oxygen ions form a cubic close packed lattice with the A and B cations occupying, respectively, tetrahedrally and octahedrally coordinated interstices. In the "*inverted*" spinel structure A- and B-cations substitute each other, which means that the real composition is $(A_{1-x}B_x)[A_xB_{2-x}]O_4$, where in parentheses and square brackets cations in A- and B-positions are shown, respectively. In general case, the inversion parameter x can be smaller than 1 (*partially inverted* spinel), but as it was shown in Ref. [2] for $NiFe_2O_4$ the degree of inversion is 1.00 and structural formula can be written as $Fe[NiFe]O_4$.

In its bulk form, $Fe[NiFe]O_4$ shows ferrimagnetic order below 850 K. Its magnetic structure consists of two antiferromagnetically coupled sublattices. A first sublattice is formed by ferromagnetically ordered Fe^{3+} ($3d^5$, magnetic moment M = 5 μ_B) ions occupying the tetragonal A sites, while the second sublattice contains ferromagnetically ordered Ni^{2+} ($3d^8$, M = 2 μ_B) and Fe^{3+} ($3d^5$, M = 5 μ_B) ions occupying the octahedral B sites. This type of ordering results in a saturation magnetization of 2 μ_B / f.u. [3].

In the Reactor and Neutron Physics Department (NRC, Cairo) the substituted spinel compounds are investigated by means of X-ray and Mössbauer techniques to shed more lights on crystallographic structure and the microscopic picture of the magnetic ordering in these diluted ferrimagnets. In particular, the Zn-substituted $Cu_{1-x}Zn_xFe_{2-y}Ga_yO_4$ compositions were investigated in details [4] and it was shown that at $x \geq 0.25$, tetragonal-to-cubic transformation occurs.

In continuation of these studies we analyze the $Ni_{0.7}Zn_{0.3}Fe_{2-y}Ga_yO_4$ compounds with y = 1.0 and 0.5. As it was determined from X-ray and magnetic measurements these compositions have cubic symmetry and the second one (y = 0.5) is ferrimagnetic. The diffraction patterns of samples S-1 (y = 1.0) and S-2 (y = 0.5) have been measured with High Resolution Fourier Diffractometer (HRFD) at the IBR-2 pulsed reactor at room, low (10 K), and high (473 K, S-2 only) temperatures. The example of raw data is shown in Fig. 1.

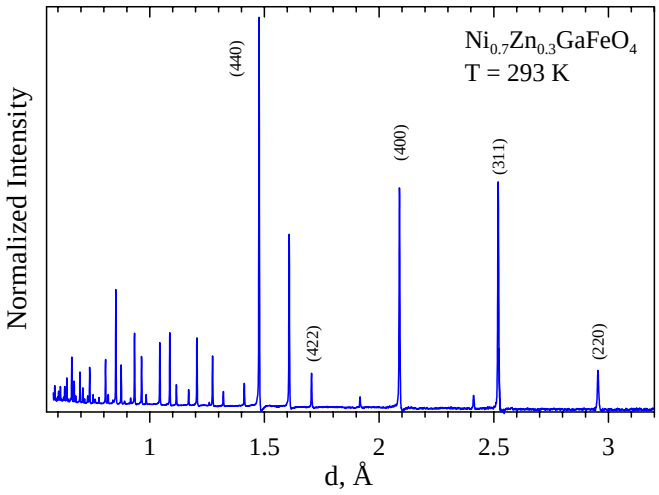

Fig 1. Diffraction pattern of the sample-1 measured with HRFD and normalized on effective neutron spectrum. Miller indices of several strong diffraction lines are indicated.

The Rietveld refinement using MRIA package was done in the frame of the Fd3m (№227) space group, the first setting (without center of symmetry). In this group the atomic positions for AB_2O_4 spinel with Z = 4 are: A in (8a), B in (16d), O in (32e) with x ≈ 3/8. The thermal factors were introduced in isotropic approximation. The next neutron scattering lengths were used in refinements: b_{Fe} = 0.954, b_{Zn} = 0.568, b_{Ga} = 0.729, b_{Ni} = 1.030, b_O = 0.581 in 10^{-12} cm units. Supposing that Fe is substituted for Zn in the A-site and all Ga atoms are in B-site it can be obtained that the coherent scattering lengths for A and B sites are: b_A = 0.838, b_B = 0.868 for S-1 and b_A = 0.838, b_B = 0.924 for S-2 again in 10^{-12} cm units.

Refinements (n_A, n_B, x_O, and B_O) of all measured patterns (an example is presented in Fig. 2) show that n_A is systematically smaller than 1 and, contrary, n_B is systematically higher than 2. The only hypothesis, which is compatible with these changes, is interchange of Fe in A-site and Ga in B-site. Numerical calculations show that in both samples around 50% of Ga atoms are shifted in the A-site. This result is important for analysis of conduction mechanism and magnetic structure of these compositions.

The magnetic contribution in the diffraction peaks is not strong but substantial. For instance, in Fig. 3 the temperature dependence of (331) line for which nuclear structure factor is close to zero is shown. The refinement of the magnetic structure is in progress now.

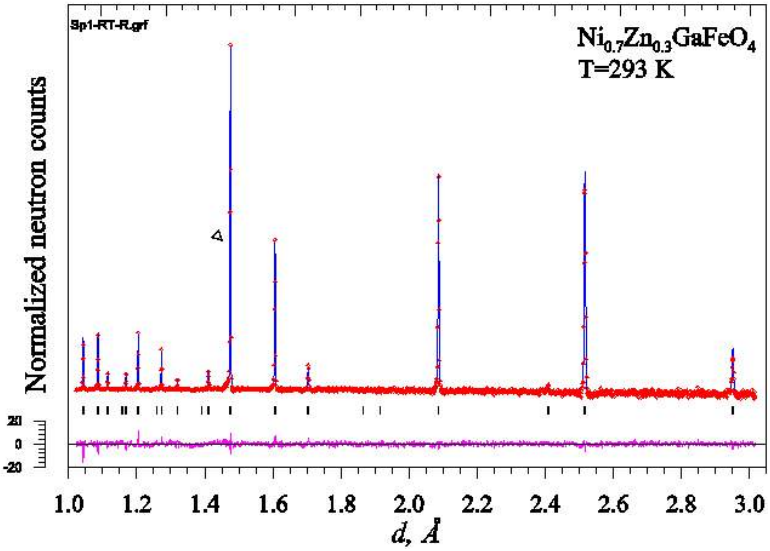

Fig. 2. Rietveld refinement of the S-1 diffraction pattern, measured at room temperature.

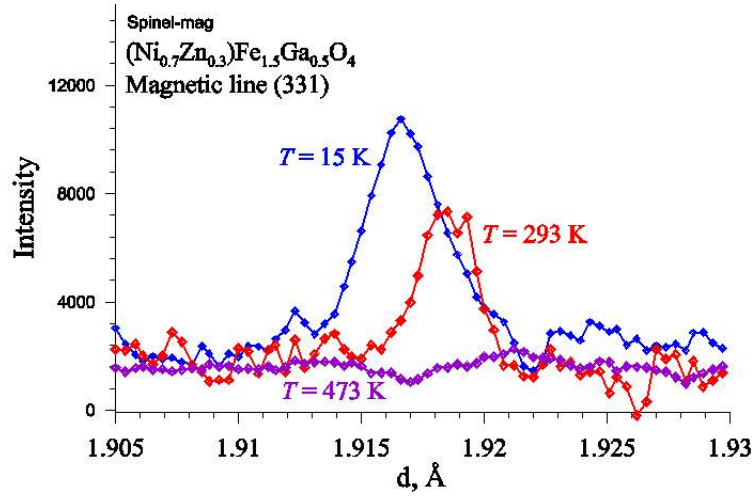

Fig. 3. Diffraction peak (331) of S-2 measured at low, room and high temperatures. Magnetic contribution is absent at 473 K.

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SANS STUDY OF BIOCOMPATIBLE MAGNETIC FLUIDS STABILIZED WITH POLY(ETHYLENE GLYCOL)

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Water-based magnetic fluids have found application in a variety of fields in biotechnology and medicine (e.g. cell separations, diagnostic magnetic resonance imaging, hyperthermia, magnetic drug targeting, etc.) [1]. The coating of the nanoparticles is one of the most important factors responsible for their compatibility in the organism. The surface of ultrafine magnetic particles can be covered with molecules with different end groups. For our purpose poly(ethylene glycol) (PEG) was chosen because it is non-immunogenic, non-toxic, non-antigenic, biocompatible and soluble in water and organic solvents [2]. Highly insoluble anticancer agents can be attached to PEG, so the solubility of the modified drug will exceed that of the original drug, increasing possibility of more effective drug delivery [3]. The aim of our work was to characterize the structure of new type of biocompatible magnetic fluid prepared by surface modification with oleate and PEG for the proposed use in medicine.

The co-precipitation method of ferric and ferrous salts in an alkali aqueous medium was used to prepare spherical magnetite particles. After washing the precipitate by magnetic decantation and heating up to 50°C, the surfactant sodium oleate ($C_{17}H_{33}COONa$) was added to the mixture to prevent agglomeration of the particles. Magnetite particles stabilized by oleate bilayer were dispersed in water. Agglomerates were removed by centrifugation (9000 RPM for 30 minutes). Finally, PEG was used to improve biocompatibility of the prepared magnetic fluid. PEG with a molecular weight 1 kDa, dissolved in water, was added to magnetic fluid at 50°C at a given weight ratio PEG/Fe_3O_4. The most important ratio was 0.25 (final sample MFPEG). A magnetic fluid – magnetite particles coated with sodium oleate and PEG – was formed in this way. The PEG adsorbtion on the magnetite surface was revealed by means of attenuated total reflectance - Fourier transform infrared (ATR-FTIR) spectroscopy, as well as by differential scanning calorimetry (DSC).

The samples were studied by small-angle neutron scattering (SANS) with the contrast variation at the SANS-1 instrument, HZG, Germany. The curves were obtained in a standard way in a q-range of 0.04-2 nm^{-1}. The influence of PEG was studied in view of structuralization of magnetic particles by SANS with the contrast variation based on hydrogen-deuterium substitution in the carrier. The new approach of the modified basic functions [4], recently applied for different classes of magnetic fluids [5-9], was used with the main accent to reveal the structural information about various aggregates in the system. The initial samples were dissolved with the ratio 1:3 by different mixtures of light/heavy water, thus varying the D_2O content over the interval of 0-70 % in the final fluid. The addition of PEG to an oleate-stabilized MF may cause considerable structural changes. Large (D > 100 nm) fractal-like aggregates of individual (non-aggregated) magnetic particles with the magnetite core size of 8 nm were observed at high PEG/magnetite ratio (ca. 2.5 by mass) as opposed to compact (D < 40 nm) aggregates present without added PEG [9]. At smaller PEG/magnetite ratio of 0.25 the initial aggregates changed less significantly (Fig.1), which is an indication of only partial substitution of sodium oleate with PEG on free magnetite surface. The MFPEG of the considered PEG/Fe_3O_4 ratio was interesting because of its application in the preparation of magnetic nanospheres, which carried the anticancer drug Taxol [10]. The changes in

the scattering curves (Fig.1) were analyzed in terms of the modified basic functions $\tilde{I}_c(q)$, $\tilde{I}_s(q)$, $\tilde{I}_{cs}(q)$ [4]. The model expression:

$$I(q) = \tilde{I}_s(q) + \Delta\tilde{\rho}\tilde{I}_{cs}(q) + (\Delta\tilde{\rho})^2 \tilde{I}_c(q) \quad (1)$$

was fitted simultaneously to all curves at different modified contrast defined as

$$\Delta\tilde{\rho} = \overline{\rho}_e - \rho_s \,, \quad (2)$$

where $\overline{\rho}_e = \langle\rho V^2\rangle/\langle V^2\rangle$ is the averaged scattering length density (SLD), ρ, over all particles (with volume V) in the system, and ρ_s is SLD of the solvent. First, $\overline{\rho}_e$ (also called the effective match point) was found from the minimum of the forward scattering intensity (obtained by the Indirect Fourier Transform (IFT)) as a function of the D_2O content in the carrier. We were mostly interested in $\tilde{I}_c(q)$ (shown in inset to Fig. 6), which is the averaged shape scattering function taking into account the type and size polydispersity. Two regions with the features of the Guinier law can be seen in $\tilde{I}_c(q)$ and are related to compact aggregates (small q-values) and micelles of free sodium oleate (large q-values). As followed from IFT, in the spherical approximation the mean size of the aggregates was 33.0 ± 0.5 nm, while the maximal size exceeded 40 nm. Since SLDs of PEG and sodium oleate are close to that of light water, at 0 % of D_2O the scattering came mainly from magnetite. From the comparison of this scattering with $\tilde{I}_c(q)$ by the IFT treatment the difference in the maximal sizes was detected. It corresponded to the thickness of the stabilizing shell, which was estimated to be 2.0 ± 0.1 nm. The analysis of the second specific q-region gave the size of about 4 nm and concentration of above 1 vol. % for micelles of sodium oleate.

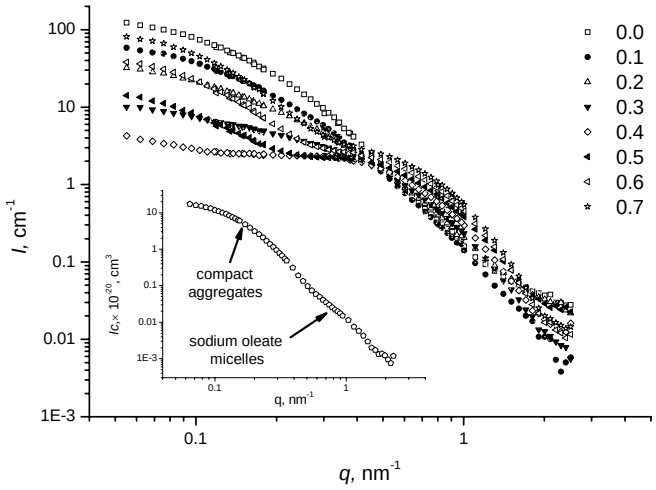

Fig 1. SANS Contrast variation of MFPEG. $I(q)$ at D_2O contents (volume fractions in solvent) from 0 to 0.7 (37°C). Inset shows the averaged shape scattering function with indicated scattering levels.

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MAGNETIC FIELD AND PARTICLE CONCENTRATION COMPETITIVE EFFECTS ON FERROFLUID BASED SILICONE ELASTOMER MICROSTRUCTURE

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The study of the properties of filled elastomers is a challenging and exciting topic for both fundamental science and industrial application. It is know that the addition of hard particulates to a soft elastomer matrix results in properties that do not follow a straightforward rule of mixtures. The progress of structure-properties relationships research for such systems evolves into several directions: filler type and structure, magneto(electro) - hydrodynamic reinforcement and interactions between fillers and elastomers. Development of novel technologies of magnetic nanomaterials shows the trends of the creation of composites with anisotropic molecular and magnetic structures such as elastomers filled with tiny ferroparticles. The combination of polymers with magnetic materials displays new and often enhanced properties. The magnetic particles couple the shape of the elastomer to the external magnetic field. Combination of the magnetic and the elastic properties leads to a number of striking phenomena that are exhibited in response to impressed magnetic field [1]. The synthesis and the study of structure and physical properties of these advanced materials combining the functional properties of elastic polymers and ferromagnetic substances should be considered as a perspective way to provide the understanding of construction principles of a wide class of materials for electronics, electrical engineering, medicine, aero- and cosmic industries. Also from the fundamental point of view it is needed a comprehensive analysis of the relationship between macromolecular and disperse phase structures and their ordering under the action of magnetic field and by the variation of magnetic component content. Neutron and X-ray scattering techniques are highly useful for determining the morphology of the formed filler structures [2-4].

The aim of present work is the small-angle neutron scattering examination of subtle structural features of polymeric matrix and ensemble of embedded ferroparticles as resulted from the conditions of preparation of ferroelastomers by the variation of concentration of ferroparticles and strength of the external applied magnetic field during the polymerization.

Ferroelastomers were prepared using the radical polymerization of dimetylsiloxane with addition of ferrofluid based on magnetite. In the samples the concentration of magnetic component (ferrofluid) was varied: 1.27; 3.9; 5.88 % mass. The magnetic field (B = 0; 280; 560; 1120 Gauss) was applied perpendicular to the plane of polymeric film (thickness of ~ 0.5 mm) (Table 1). It was prepared also the polymeric matrix without ferroparticles (reference sample).

The small-angle neutron scattering experiments (SANS) have been carried out at ambient temperature (20^oC) on the diffractometer "Membrane" (PNPI) in the range of momentum transfer $q = (4\pi/\lambda)\sin(\theta/2) = 0.03\text{-}0.8\ nm^{-1}$, where θ is scattering angle and $\lambda = 0.3$ nm is neutron wavelength ($\Delta\lambda/\lambda = 0.25$).

The scattering patterns for the elastomers containing 5.88; 3.9 and 1.27 % mass of magnetite are presented in Figure 1(a-c) where the data for the matrix are shown also. It is evident (Figure 1) that the scattering from original polymeric matrix is relatively strong at $q \leq 0.2\ nm^{-1}$. The addition of ferroparticles into matrix does not provide any substantial contribution to the total scattering intensity at low momenta even at high concentration of magnetite (5.88 % mass.). The scattering from ferroparticles dominates only at larger momenta, $q \geq 0.4\ nm^{-1}$ due to their small size as compared to inhomogeneities in polymer matrix. It should be noted that the

application of magnetic field (B = 1120 Gs, sample P_{15}) during the polymerization did not initiate any dramatic structural changes. It is observed a moderate increase in scattering: the ratio of intensities for samples P_{15} (strong field) and P_{12} (no field) does not exceed factor 2 in the interval of q = 0.4-0.8 nm^{-1}. This means that the polymer network prevents the aggregation of ferroparticles and provides their more homogeneous spatial distribution to be achieved for required material quality. Large-scale inhomogeneities in polymer matrix are visible despite of low scattering ability of polymer having the density of coherent length $K_S = 6.3 \cdot 10^8$ cm^{-2}. It is by 2 orders in magnitude lower than that for magnetite having $K_M = 6.971 \cdot 10^{10}$ cm^{-2}. Thus, the contrast factor for magnetite regarding to matrix is also high, $\Delta K_M = K_M - K_S = 6.91 \cdot 10^{10}$ $см^{-2}$, but it is compensated by small characteristic volume of a particle as compared to large inhomogeneities in matrix, as it is seen in Figure 1b and Figure 1c.

A detailed analysis of q-dependencies of scattering intensity for matrix enables us to built the two-level model structure and related scattering function

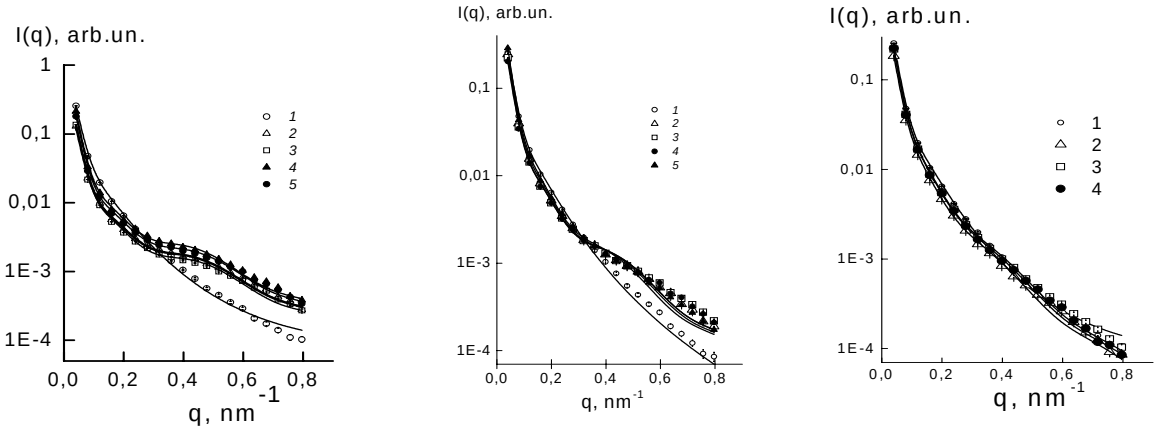
$$I(q) = I_{01} \exp[-(qR_g)^2 / 3] + I_{02}[1 + (qR_c)^2]^{-2} \qquad (1)$$

The first Guinier-term describes the large scale inhomogeneities (domains, radius gyration R_G), and the second Debye-term is related to the smaller globular objects (domains) with correlation radius R_C.

For ferroelastomers we have used the model (2) taking into account the scattering from large-scale polymer domains (gyration radius R_G) and ferroparticles (correlation radius r_C) separated in space at the distance L comparable to small polymer domain diameter ~ $2R_C$:

$$I(q) = I_{01} \exp[-(qR_g)^2 / 3] + I_{02}[1 + (qR_c)^2]^{-2}[1 + n \sin(qL)/(qL)] \qquad (2)$$

In expression (2) the parameters I_{o1}, I_{o2} represent the contributions of large polymer domains and ferroparticles in scattered intensity at q →0. The value of n is the average number of particles correlated with a given particle at the characteristic distance L. The function (2) describes satisfactory the behaviors of scattering intensities for ferroelastomers at all the concentrations and the magnitudes of induction B.



a) b) c)

Figure 1. Scattering intensities I(q) vs momentum transfer q for matrix P_1 (1) and ferroelastomers P_{12}-P_{15} (2-5) with high content of magnetite (a); ferroelastomers P_{21}-P_{25} (2-5) with moderate content of magnetite (b); ferroelastomers P_{32}, P_{33}, P_{35} (2-4) with low content of magnetite (c) (synthesis in magnetic field perpendicular to the plane of polymer film). Lines are the approximation functions (1) and (2) for matrix and ferroelastomers.

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NEUTRON DIFFRACTION STUDY OF $LiFePO_4$ CATHODE MATERIAL DOPED WITH VANADIUM OXIDE

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$LiFePO_4$ (LFP) is one of the most promising cathode materials for the next generation of lithium-ion battery because its appealing electrochemical features including excellent chemical/thermal stability, low material cost, non-toxicity, and a high theoretical capacity. However, the poor ionic as well as electrical conductivity and the incomplete utilization of capacity are fatal defects significantly hinders its applications. In studies performed by Liu *et al*. [1] it was found that the doping of a heteroatom (Ti, Zr, V, Nb, and W) could promotes the performance of $LiFePO_4$ at high current due to the enlarged lattice volume that provides more space for lithium-ion transfer. Meanwhile, Jint *et al*. [2] found that introduction of a small amount of vanadium into the carbon-coated LFP particles would significantly improves the rate capability and low-temperature performance due to the formation of conductive V_2O_3 nano-grains. Thus, to probe the crystal evolutions of materials upon varying charge and discharge conditions is of essential importance in terms of depicting the effects of VO on Li trajectories in LFP.

By applying high resolution neutron diffraction, we anticipate depicting the structure evolution of x·VO doped LFP samples as a function of temperature. As the first step, two samples of VO doped LFP with x = 0 and 0.0075 have been measured with HRFD instrument at the IBR-2 pulsed reactor at room and low temperature. The last measurement has been performed to elucidate the magnetic structure of the compound, which is known as antiferromagnetic below $T_N \approx 52$ K [3]. Neutron diffraction patterns have been measured by several detectors: high-resolution back-scattering (up to 3.7 Å), low-resolution (up to 4.5 Å), and PSD at 2θ = 15° (up to 16 Å). Example of the high-resolution patterns is shown in Fig. 1.

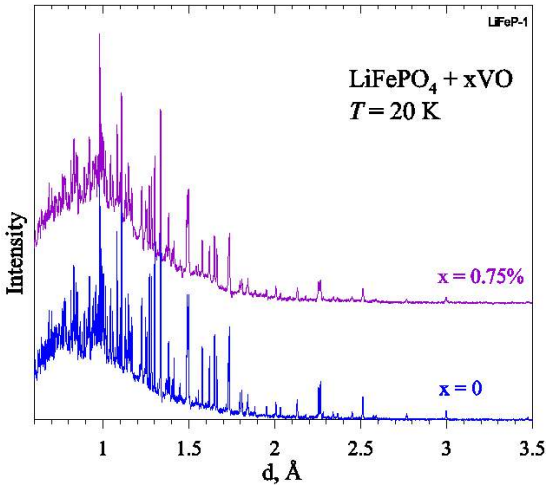

Fig 1. Diffraction pattern of the x = 0 and 0.75% samples measured with HRFD at low temperature.

The Rietveld refinement using MRIA package [4] was performed in *Pnma* (№62) space group with the model from Ref. [3]. In this group the atomic positions for perovskite structure with Z = 4 are: Li in (4a) (0,0,0), Fe, P, O1, and O2 in (4c) (x,1/4,z), O3 in (8d) (x,y,z). The thermal factors were introduced in isotropic approximation. The following values of neutron scattering lengths were used in refinements: b_{Li} = -0.222, b_{Fe} = 0.954, b_P = 0.513, b_O = 0.581 in 10^{-12} cm units. Examples of refinement are presented in Fig. 2.

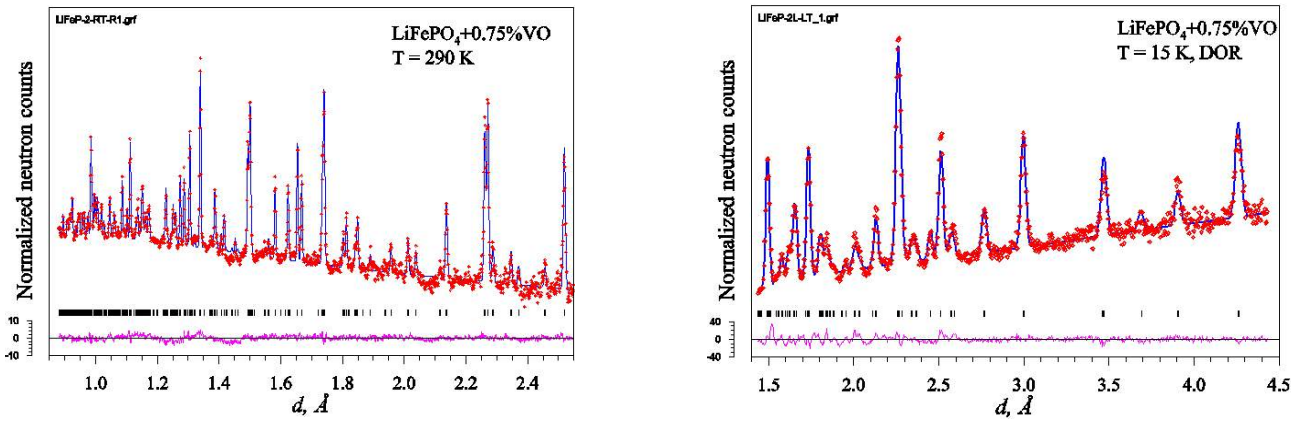

Fig 2. Rietveld refinement of the diffraction pattern (x = 0.75%), measured with high-resolution at room temperature (on the left) and low-resolution at low temperature (on the right). At this stage of refinement no magnetic phase has been introduced. There are some irregularities in the background, which reflect structural disorder at local level.

The refined atomic coordinates are in very good agreement with G. Rousse *et al.* [3] data. Moreover, the atomic coordinates are practically the same for both samples and both temperatures – the maximal changes in atomic bonds is around 0.03 Å. It means that there is no noticeable structural transformation during temperature lowering and owing to introducing of VO. The only marked difference between two samples is in level of microstrains in crystallites: they are at the conventional level for the x = 0 sample and are 1.5 higher for the x = 0.75% sample. For further analysis it would be useful to measure diffraction peaks width dependences for several VO contents.

No additional reflexions appear at temperature lowering, though intensities of several peaks increase markedly, namely, of (101), (210), and (301) peaks, which indicates that the magnetic structure corresponds to the propagation vector **k** = 0. The refinement in the frame of model from Ref. [3] shows good agreement between experimental and calculated magnetic intensities. Temperature scan has been performed and the Neel temperature has been obtained as $T_N \approx 50$ K from ratio (101) and (211) intensities (Fig. 3).

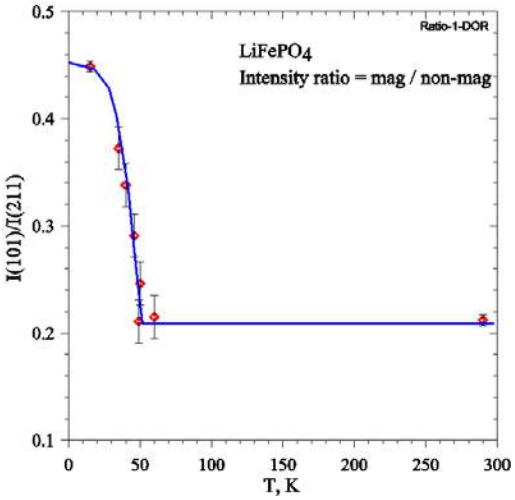

Fig. 3. Ratio of intensities of “magnetic” (101) and “non-magnetic” (211) peaks (x = 0 sample) as a function of temperature. The Neel temperature is close to 50 K.

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NEUTRON DIFFRACTION STUDY OF ATOMIC AND MAGNETIC STRUCTURES OF $La_{1-x}Sr_xFe_{2/3}Mo_{1/3}O_3$

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The $LaFe_{2/3}Mo_{1/3}O_3$ (LFM) perovskite like compound has been synthesized for the first time and characterized in 1996 [1]. X-ray data shows that it is crystallized in the *Pnma* space group ($GdFeO_3$ structural type) without any order in the B-sites. The compound is antiferromagnetic below T_N = 520 K, but exact type of magnetic structure was unknown so far. Its electrical conductivity is high enough (1.5 Ω*cm at 25°C) for considering LFMO as potential electrode material for SOFC.

Under La → Sr substitution the unit cell parameters are changed as typical for solid solutions. According to X-ray diffraction results the atomic structure of $La_{1-x}Sr_xFe_{2/3}Mo_{1/3}O_3$ is stable up to x = 0.67. No additional diffraction lines appear which can be attributed to Fe/Mo ordering. Structure refinement is hampered due to "pseudocubic" lattice symmetry: resolution of conventional X-ray laboratory equipment is not enough to see clearly orthorhombic splitting. The Neel temperature depends on Sr content and reaches ~700 K for x = 0.5.

To refine atomic and magnetic structures of Sr-substituted LFM neutron diffraction patterns for x = 0.3 (at 20°C и 350°C) and 0.5 (at 20°C and 450°C) samples have been measured at HRFD diffractometer at the IBR-2 reactor in Dubna. Several detectors were used for data acquisition: high-resolution back-scattering (up to 3.7 Å), low-resolution (up to 4.5 Å), and PSD at 2θ = 30° (up to 16 Å). Example of the high-resolution patterns is shown in Fig. 1.

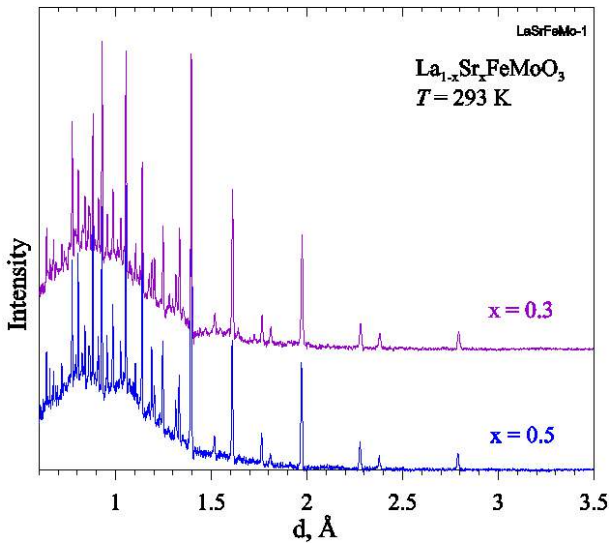

Fig 1. Diffraction pattern of the x = 0.3 and 0.5 samples measured with HRFD at room temperature.

At high temperature intensity of some diffraction lines strongly reduces, as it is seen in Fig. 2, which is connected with vanishing of AFM order. For both samples all peaks can be indexed with *Pnma* space group in the standard setting: $a \approx c \approx \sqrt{2}a_c$, $b \approx 2a_c$, where $a_c \approx 3.9$ Å is the parameter for the ideal cubic perovskite.

The Rietveld refinement using MRIA package [2] was performed in *Pnma* (№62) space group. In this group the atomic positions for perovskite structure with Z = 4 are: (La/Sr) (4c) (x,1/4,z), (Fe/Mo) (4b) (0,0,1/2), O1 (4c) (x,1/4,z), and O2 (8d) (x,y,z). The thermal factors were introduced in isotropic approximation. The following values of neutron scattering lengths were used in refinements: b_{La} = 0.824, b_{Sr} = 0.702, b_{Fe} = 0.954, b_{Mo} = 0.672, b_O = 0.581 in 10^{-12} cm units.

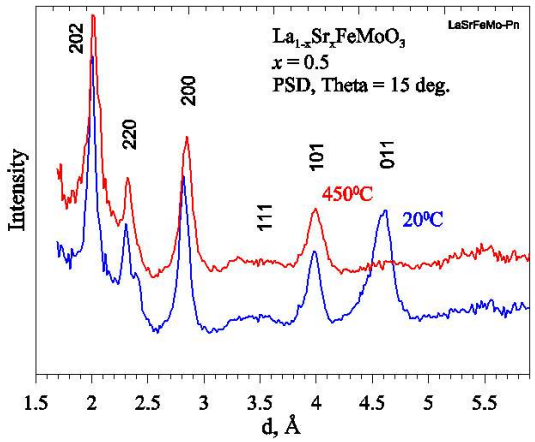

Fig 2. Diffraction pattern of the x = 0.5 sample measured with PSD at room and high temperatures. At T = 450°C the (011) peak at d ≈ 4.6 Å is practically absent.

Refinements of structural parameters of both samples (an example is presented in Fig. 3) show that LSFM has typical orthorhombically distorted perovskite structure. The special features are slightly modulated incoherent background, which can be connected with correlative disorder, and quite large width of diffraction peaks. Supposing that the second effect is connected with microstresses in crystalline grains they can be estimated as $\Delta a/a \approx 0.004$ at room and 0.008 at high temperatures. For conventional orthorhombic perovskites $\Delta a/a$ is of order 0.002, which means that for LSFM the microstresses are approximately 2 times larger and become twice more at high temperature.

The magnetic structure was solved as G-type, according to the classification of Ref. [3], with **k** = 0: magnetic moments of neighboring Fe atoms have opposite directions. For such model the first diffraction peaks with strong magnetic contribution are (011) (4.56 Å), [(112), (211), (031), (≈ 2.38 Å), [(013), (132), (231), (≈ 1.81 Å). Intensity of just these peaks strongly reduced at high temperature. Because of strong diffraction peaks overlapping the direction of magnetic moment can not be reliably determined. The refinement of the magnetic moment value will be performed later.

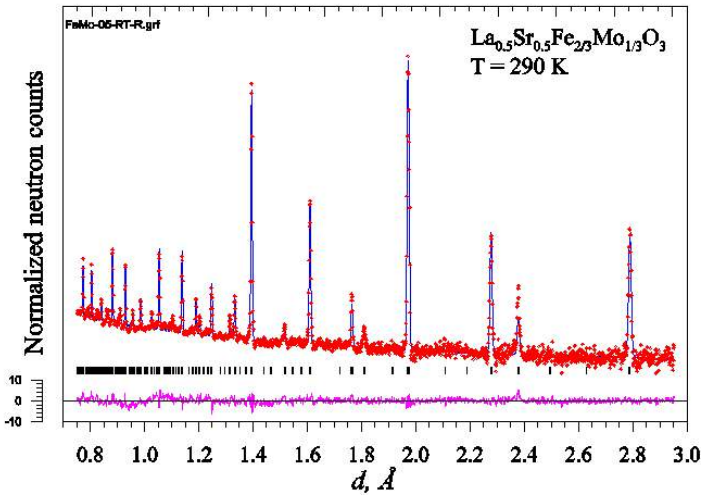

Fig. 3. Rietveld refinement of the diffraction pattern (x = 0.5), measured at room temperature. At this stage of refinement no magnetic phase has been introduced.

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THE STRUCTURAL STUDIES OF ANTIFERROELECTRIC-FERROELECTRIC PHASE TRANSITION IN SODIUM NIOBATE.

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The perovskite ferroelectric materials have been extensively studied during last years due to fundamental interest and technological applications [1]. In this class of materials, lead-free alkaline niobates: $KNbO_3$, $LiNbO_3$ and $NaNbO_3$ have high piezoresponse comparable to classical PZT ceramics. One of the members, $NaNbO_3$ is a well-documented antiferroelectric which finds applications in high density optical storage, enhancing non-linear optical properties, as hologram recording materials, etc [1, 2].

At ambient conditions the sodium niobate is antiferroelectric. At low temperature a ferroelectric phase with rhombohedral symmetry occurs below 190 K [3] in $NaNbO_3$. The phase transition from antiferroelectric to ferroelectric is associated with a small tilt of the NbO_6 octahedron [4]. The structure futures of antiferroelectic and ferroelectric phases and primary mechanisms of such phase transition remain unexplored. The knowledge of relationship between ferroelectric or antiferroelectric states and crystal structure features, which can be derived from high pressure investigations, is very essential for understanding the nature of physical phenomena observed in sodium niobate.

We have studied the structure changes in $NaNbO_3$ by means of energy-dispersive X-ray diffraction at high pressure up to 4.0 GPa. The X-ray diffraction experiments were carried out using the multianvil X-ray system MAX80 at F2.1 beamline of storage ring DORIS-III. The sample was placed in the cylindrical boron nitride container with an internal diameter of 1 mm. The upper half was filled with the sample, the lower half contained sodium chloride powder for pressure calibration. The cubic boron-epoxy chamber with sample container was compressed by six tungsten carbide anvils in a large hydraulic press. The temperature at the sample was produced by an internal graphite heater and controlled by thermocouples. Diffraction patterns were recorded in an energy dispersive mode using white synchrotron X-rays from the storage ring DORIS-III. The incident X-ray beam was collimated to 100 × 100 μm with a divergence smaller than 0.3 mrad. Spectra were recorded by a Ge solid-state detector with a resolution of 153 eV at 5.9 keV resulting in a resolution of diffraction patterns of $\Delta d/d \approx 1\%$. The Bragg angle 2θ was fixed at 9.093°, counting times for each diffraction pattern were about 4 min.

At pressure P=1.6 GPa the significant changes in pressure dependences of diffraction peaks position and its width were observed. Those changes are corresponding to the phase transition from antiferroelectric to ferroelectric phase in sodium niobate. The pressure dependence of the lattice parameters and unit cell volume of both phases are shown in Fig. 1.

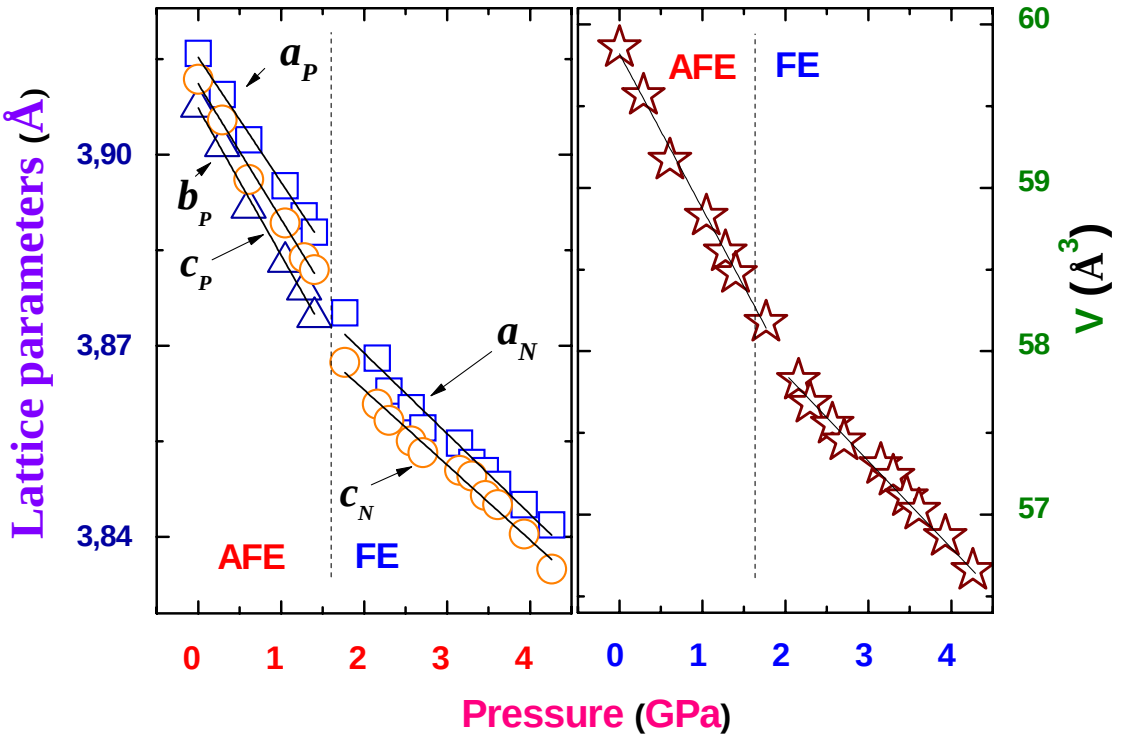

Figure 1. *The pressure dependencies of unit cell parameters and volume of the rhombohedral ferroelectric FE (a_N and c_N) and orthorhombic antiferroelectric AFE (a_P, b_P and c_P) phases of sodium niobate.*

The linear compressibility $k_i = -(1/a_{i0})(da_i/dP)_T$ ($a_i = a, b, c$) of unit cell parameters are k_a = 0.0050(6), k_b = 0.0071(7), k_c = 0.0054 (5) GPa^{-1} for the orthorhombic antiferroelectric phase and k_a = 0.0032(8), k_c = 0.0042(5) GPa^{-1} for the rhombohedral ferroelectric phase.

The pressure dependence of unit cell volume was approximated by the Birch-Murnaghan equation of state [6]. The calculated values are B_0 = 37(3) GPa and B' = 4(1) for the antiferroelectic phase and B_0 = 45(3) GPa, B' = 4(1) for the ferroelectric phase of sodium niobate.

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THE STRUCTURAL STUDIES OF $Y_3Al_5O_{12}:Ce^{3+}/Lu_2O_3$ PHOSPHORS SYNTHESIZED BY COLLOID-CHEMICAL METHOD.

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At the present time one of the topical problems in optics is a search for phosphors, which under excitation with light could efficiently radiate energy in a given spectrum range with minimal losses [1]. The promising materials are crystalline systems based on yttrium aluminum garnet $Y_3Al_5O_{12}$ (YAG). For example, neodymium-doped YAG:Nd^{3+} is a well-known laser material, but cerium-doped yttrium garnet $Y_3Al_5O_{12}:Ce^{3+}$ is used in several applications such as solid-state lighting, displays, scintillators [2]. The Ce^{3+} ion is responsible for nanosecond decay time and an intense yellow-green emission wavelength. So it are suitable yellow-emitting phosphors for the application to white light-emission diodes - LEDs [3].

The physical and optical properties of $Y_3Al_5O_{12}:Ce^{3+}$ depend on crystal structure features. YAG:Ce^{3+} has the cubic garnet crystal structure with a lattice parameter a=12.011(3) Å. In garnet structure, there are 24 dodecahedral sites, 16 octahedral sites, and 24 tetrahedral sites.

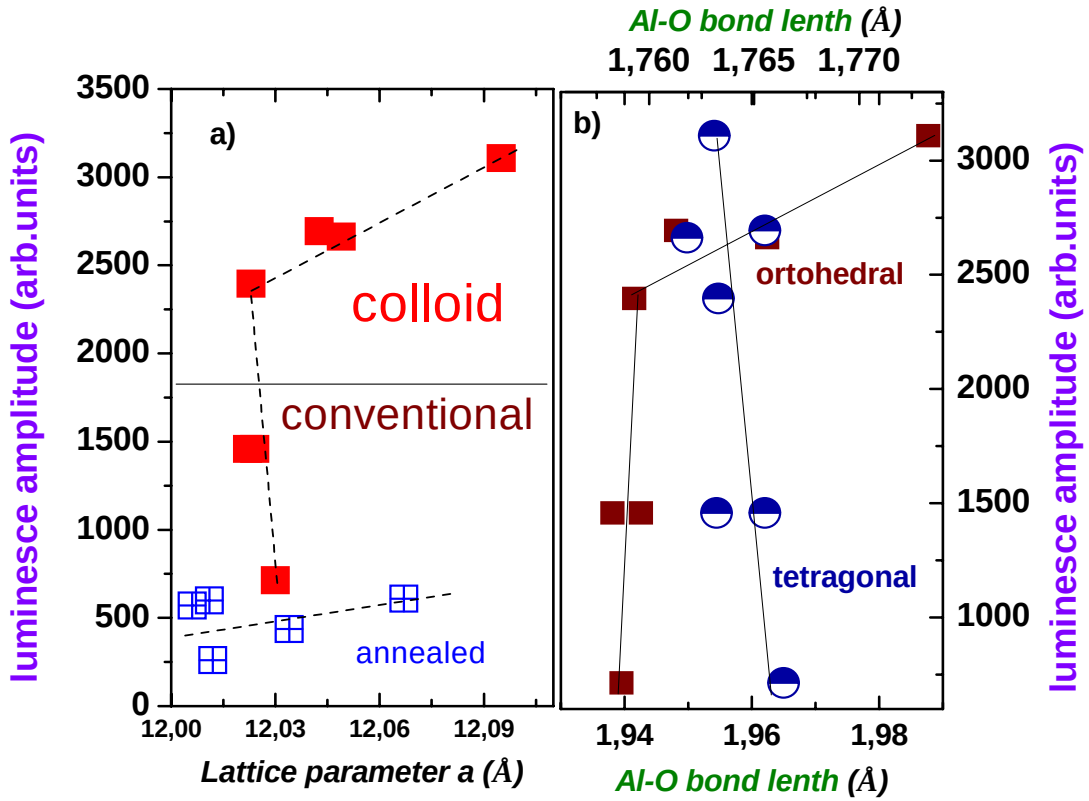

Figure 1. a) *The total luminescent intensity as function of lattice parameter of phosphors YAG:Ce^{3+}/Lu_2O_3, obtained colloid-chemical and conventional methods. The red symbols are corresponded by initial sample and blue ones with annealed samples.* ***b)*** *The total luminescent intensity as function of Al-O bonds in tetragonal and octahedral oxygen environment.*

The optical properties of such phosphors are strongly depends of crystal parameters as bond lengths and valence angles due metastable defect structure forming. The chemical-colloid methods of oxide system synthesis [4] to allow tuning of such defect structure and vacancies concentration and, as result, the optical properties of garnet-based phosphors.

Our optical measurements of $Y_3Al_5O_{12}:Ce^{3+}$ have been shown, that way of Ce^{3+} doping via additional insertion of Lu_2O_3 oxide, can be drastically improve light emission intensity of those phosphors. For structural aspects of this effect studies the neutron diffraction experiments have been prepared.

Neutron diffraction measurements at ambient conditions were performed with the DISK diffractometer at the IR-8 reactor, Moscow, Russia. The sample with a volume about 25 mm^3 was placed in vanadium container. The typical exposition time for each pressure point was to 20 h.

The lattice parameters and bond lengths have been calculated from obtained neutron data. It was found that yttrium aluminum garnets, obtained by colloid-chemical methods are characterized by greater lattice parameter (Fig. 1a) and longer length of Al-O bond in octahedral oxygen coordination (Fig. 1b). As result, the total luminescent intensity of those compounds is drastically higher than for compound obtained by conventional chemical method (Fig.1a). So difference in lattice parameters and bond lengths is result of forming stable defect substructure in oxygen lattice [4]. It was found such defect substructure formed in octahedral coordination environment of aluminum (Fig. 1b). The defect substructure in oxygen lattice have been broken by additional annealing of compounds, and as result the enhanced luminescence intensity was suppressed to intensity level observed for yttrium aluminum garnets, obtained by conventional methods (Fig. 1a).

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EXPERIMENTAL DETERMINATION OF NEUTRON CHANNELING LENGH IN PLANAR WAVEGUIDE

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Neutron planar waveguide transforms the conventional neutron beam into a very narrow (about 0.1 μm width) and slightly divergent (about 0.1°) microbeam. Such narrow beam can be used for investigation of nanostructures with submicron space resolution. In [1] non-polarized microbeam was demonstrated experimentally. In our experiment [2] magnetic waveguides were investigated and polarized neutron microbeam was demonstrated.

Neutron wave function is resonantly enhanced inside a guiding layer (channel) and neutron channeling phenomenon takes place. A decay length of a neutron wave in the channel along the interfaces is an important parameter of waveguides. The theory of neutron channeling in planar waveguides can be found in [3]. Experimentally neutron channeling phenomenon was observed in [4-7]. But the channeling length was not measured experimentally. We report experimental results on direct measurement of the neutron channeling length in planar waveguide.

The measurements were done at the polarized neutron reflectometer N-ReX$^+$ (reactor FRM II, Garching, Germany). The fixed neutron wavelength 0.426 nm (1 % FWHM) was used. The angular divergence of the primary beam was 0.006°. The sample Fe(20 nm)/Cu(140 nm)/Fe(50nm)//*glass* (substrate) was investigated [3]. The sample sizes were 30×30×5(substrate) mm^3. The polarized neutron beam (+) was used. The scheme of experiment is shown in Fig. 1. The incidence angle of the primary beam is α_i. The primary beam tunnels through the upper layer with the thickness a into the guiding layer with the thickness d. Then neutrons channel at the resonance condition in the guiding layer and the neutron microbeam leaves the edge under Fraunhofer diffraction conditions. We measure the intensity of the outgoing microbeam $I(x) = I(0) \cdot \exp(-x / x_e)$ as a function of the length x of the absorbing Gd_2O_3 powder on the sample surface. Here $I(0)$ is the measured intensity of the microbeam without the Gd_2O_3 powder (x = 0) and x_e is the channeling length to be defined.

In Fig. 2a the integrated (over the incidence angle interval at resonance n=0 and the diffraction angle interval $\Delta\alpha_f = \pm 0.1°$) intensity of the microbeam is shown. Fit by exponential function gives the channeling length $x_e = 3.2 \pm 0.5$ mm. In Fig. 2b this dependence is shown in natural logarithm scale. One can see that the experimental points are described by the straight line within statistical errors which gives the error of the defined channeling length. From the theory, the channeling length increases with an increasing of the thickness a of an upper layer and the thickness d of a guiding layer. The channeling length also depends on optical potentials of the layers of waveguides. The channeling length calculated from the theory for the incident angle of the resonance α_{i0}=0.37° is equal to x_e = 3.5 mm. Thus, the experimental value of the channeling length coincides with the calculated value within error bar.

The support of the management and staff at the FRM II, Garching, is gratefully acknowledged. This work was supported by a Focused Neutron Research Funding of the Max Planck Society, Munich. The authors are grateful to J. Franke for the technical help during the experiments.

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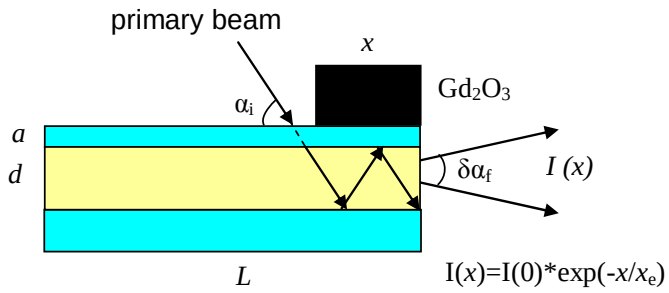

Fig. 1. Geometry of the experiment.

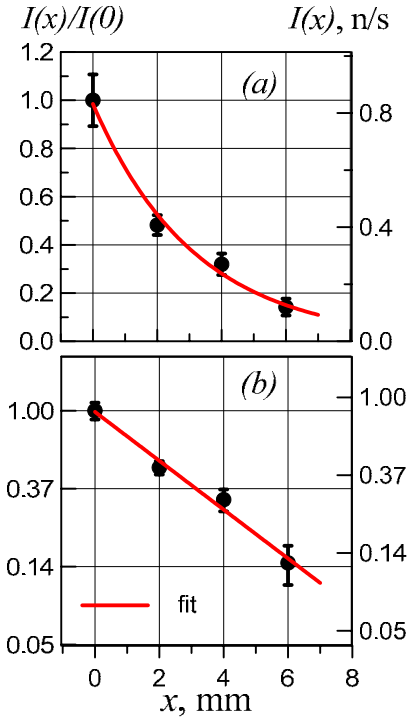

Fig. 2. Integrated microbeam intensity at the resonance angle α_{i0}=0.37° as a function of the Gd_2O_3 powder length.

RIETVELD TEXTURE ANALYSIS OF SKAT DIFFRACTOMETER DATA: RECENT ADVANCES AND PERSPECTIVES

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The texture specialized diffractometer SKAT [1] is located on the beamline 7a of the pulsed fast reactor IBR-2. The rather long (≈ 103.8 m) flight path of the thermal neutrons and the chosen scattering angle of $2\theta = 90°$ permit to investigate lattice spacing in the range of $d \approx 0.6…4.7$ Å, with a resolution of $\Delta d/d \approx 0.55\%$. That is sufficient to study the lattice preferred orientations (LPO) of most rocks and engineering materials. Commonly a finite (usually < 6 for each phase) number of complete experimental pole figures (PF) is used to determine the orientation distribution function (ODF) of the crystallites of the main phases in a sample. The construction of the experimental PFs with a 5ºx5º angular grid (19 detectors and 72 sample positions lead to 1368 spectra) requires the manual selection of intensive non-overlapped diffraction peaks from the TOF diffraction patterns.

This approach has some drawbacks. E.g., in the case of rocks containing several minerals with a low crystal symmetry (such as biotite, albite, orthoclase, etc.), the selection of non-overlapped peaks is often possible (if possible at all) only at high d ranges (e.g., $d > 3.5$ Å), where the counting rates in the SKAT spectra are quite poor. In this case only a small part of the available rich diffraction information in the measured spectra is used. The "Rietveld texture analysis" [2, 3] developed in the last decade permits to analyze many diffraction patterns (even highly peak overlapping regions) as a whole, and, beyond the ODF determination, also to receive some additional information about the sample: phase volume fractions, cell parameters, residual stresses, etc.). The freeware program system MAUD [2-4], utilizing the modified Rietveld method, also possesses a number of options for the determination of the ODFs for multiphase samples from TOF diffraction spectra. Therefore a possibility existed to apply MAUD to the typical set of SKAT spectra. In order to adapt the SKAT data for a MAUD analysis a user-friendly C++-based program SKAT2MAUD has been developed and thoroughly tested during the last year. In future the program may easily be adjusted for the processing of data from other IBR-2 diffractometers like FSD, EPSILON-MDS or new SKAT version with several detector rings. Using MAUD the analysis of conventional SKAT spectra from manifold samples of different type (powders, single-phase polycrystals with LPO, single-phase polycrystals with LPO and residual stresses, multi-phase polycrystals with LPO of each phase) has been performed and showed excellent results.

As an example below some results are presented concerning the texture analysis of biotite in the gneiss samples OKU818 (composed by 39.9 vol.% quartz, 37.4 vol.% andesine plagioclase and 22.6 vol.% biotite) from the Outokumpu scientific drill borehole. Samples of roughly cubic shape cut from the same block have been investigated on the SKAT and HIPPO (Los-Alamos) [5] diffractometers. The data have been processed by MAUD in case of HIPPO (the long been developed routine analysis procedures are described in [3, 5]) and in case of SKAT by two methods: using MAUD and the up to now applied conventional method (analyzing pole figures) described in [6]. In order to compare the derived ODFs with account for the real angular resolution of the diffractometers in the pole figure space the original HIPPO ODF has been smoothed by a Gaussian function in the orientation space with a FWHM = 20° (the HIPPO PF resolution depends on the scattering angles of the detector rings and rises from 10 up to 19° [5]). The original SKAT ODFs have been filtered (with preserving texture sharpness) by a Gaussian function with the FWHM = 7.5° (the mean angular size of the SKAT detectors in the pole figure space of about 3° is much lower than for HIPPO, but the 5ºx5º measuring grid of SKAT reduces its available resolution to effectively 7.5°). The results presented in Table 1 show that the Rietveld texture analysis applied to both diffractometers yields nearly the same results, while the conventional pole figure analysis of the SKAT data (using only some isolated peaks in the TOF spectra) clearly underestimates the

sharpness of the biotite texture. The difference of the f_{min} values (isotropic texture background "phon") for the final HIPPO and SKAT MAUD ODFs is most probably connected with the lower SKAT counting statistics and the corresponding difficulties to correctly estimate and to subtract the background from TOF spectra. Thus, even though the SKAT diffractometer is able to investigate larger samples (in order to achieve a representative grain statistics) and possesses a relatively high angular resolution (necessary to correctly detect sharp orientation distributions), in practice the better counting statistics of the HIPPO diffractometer at LANSCE is very valuable for most quantitative texture studies and allows to perform the measurements in a reasonable time (e.g., the OKU818 measurements have been performed in 8 hours on HIPPO vs. 36 hours on SKAT). This drawback should be overcome with the improved SKAT diifractometer that expected to have a several times higher flux at the sample due to the new neutron guide.

Table 1. Characterization of the biotite ODF in the OKU818 gneiss sample: minimum ODF value f_{min}, maximum ODF value f_{max}, texture index F2, part of non-randomly oriented crystallites SH>1 and the part of orientation space volume occupied by these crystallites CPGs>1.

Instrument (processing)	f_{min}	f_{max}	F_2	SH>1, %	CPGs>1, %
HIPPO (MAUD)	0.03	26.45	6.2	58.5	17
SKAT (MAUD)	0.08	26.88	6.1	53.8	17
SKAT (conventional PF)	0.01	21.05	3.5	44.4	24

Another advantage of the analysis of the SKAT data using MAUD opens additional possibilities for the optimization of future experiments. Due to the use of a great number of diffraction peaks (now including overlapped ones too) the formal number of considered experimental PFs is great. Consequently, only a limited coverage of the experimental PF will be sufficient for the ODF reconstruction preserving the same angular resolution. This infers that it is e.g. possible to perform measurements with less sample orientations than before, drastically reducing the total measurement time for a given sample (for up to 12 times!, cf. Fig.1) and/or simultaneously to increase the counting statistics for each sample position (e.g., measuring only 1/6 of the complete pole figure coverage, but measuring each position for 1.5 hours instead of 0.5 hours).

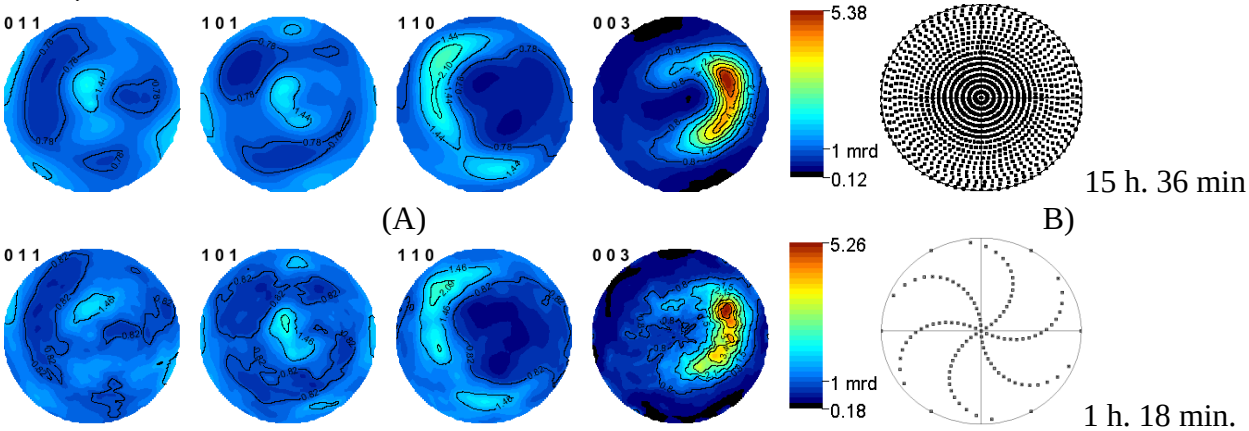

Fig.1. Pole figures of the quartz in the quartzite sample 26a. Equal area projections, linear scale contours. Conventional PF analysis of all data (texture index F2=2.35) (A), corresponding PF coverage and measurement time (B); analysis of 1/12 part of data with MAUD (texture index F2=2.24) (C), corresponding PF coverage and estimated measurement time (D).

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Development of the SESANS spectrometer elements based on rotating magnetic fields

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The new spin-echo method based on the use of rotating magnetic field was proposed recently [1]. After this there were carried out number of experiments in order to test the idea and estimate the perspectives. In the experiment described here we constructed the prototype of one spin echo arm which consist of two spin flippers with rotated magnetic field (RMF). Magnetic fields in spin-flippers are confined in flipper plane and are rotated in the plane normal the propagation direction, *yz*, of the neutron beam polarized along the vertical *z*-axis (see fig. 1,2). All three components of polarization vector passed through the set up were measured in dependent on the applied current frequency. Experiment was carried out on TREF spectrometer (JCNS) at FRM II reactor.

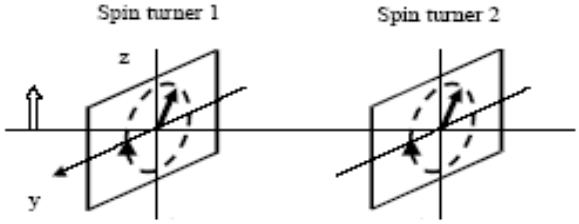

Fig.1 One arm of NSE set-up composed by two spin turners.

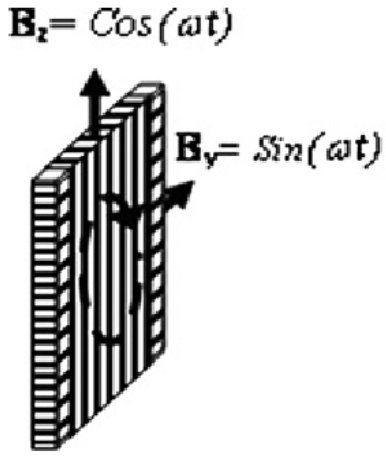

Fig. 2 The spin turners as rectangular electromagnetic coils with two windings perpendicular to each other. Each windings feed by the sinusoidal current with phase shifting of $\pi/2$ between them.

To analyse the behaviour of the neutron spin in such a flipper we calculate the magnetic fields distribution inside and outside of the flipper with use of the MagNet [2] software. Based on the calculated magnetic fields we compare the experimental data on the neutron spin passage through the flippers with simulation of this process in the VITESS [3] package. For this purpose it was updated the VITESS modules using magnetic field. In the new version the magnetic field can be loaded from the external source. This is a great advantage because in previous version magnetic fields configuration can be defined by limited number of parameters and functions. Using of the new option allows taking into account the influence of such factors as border effects, effects of non-

full field shielding and so on. At Fig.3 one can see the experimental dependence of Pz polarization component measured at the exit from the set-up shown at Fig.1 and it's VITESS simulations.

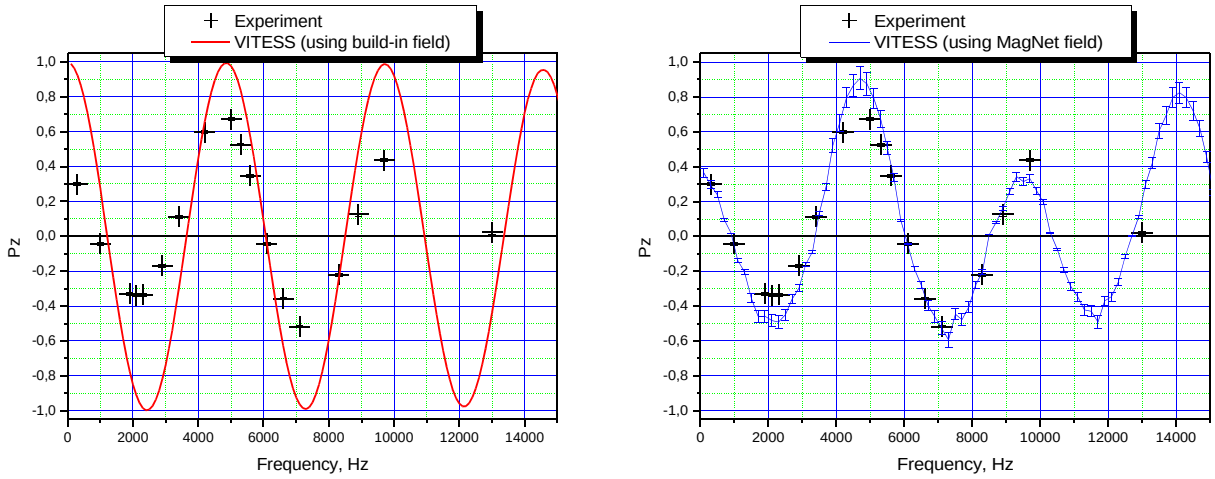

Fig.3 Frequency dependence of the *z*-component of the polarization in compare with VITESS simulation. Solid line on the left picture is simulation with magnetic fields generated in VITESS. Solid line on the right picture is simulation with magnetic fields generated by MagNet.

Thus the cooperative of two software packages gives new possibilities in description of experimental data with passage of neutron polarization through the time-dependent magnetic field configuration. It allows improving the construction of NSE elements and it efficiency.

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NEUTRON AND SYNCHROTRON X-RAY DIFFRACTION STUDY OF MODEL ORAL STRATUM CORNEUM LIPID MIXTURES

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The epithelium covering the oral cavity (oral stratum corneum, OSC) provides a protective barrier for underlying tissue, but it is not so distinct like the epidermal stratum corneum (ESC). The OSC lipid matrix consists mainly of the phospholipids (PL), cholesterol (Ch), fatty acids (FA), cholesterol sulphate (ChS), triglycerides and short-chain ceramides (CERs). Although the lipid composition of the OSC is well defined the physical studies of the nanostructure of rare OSC-lipid bilayers are lacking [1] and their role in formation of the barrier function is still unclear. Neutron and X-ray diffraction experiments have been performed to characterize the structure of model OSC lipid mixtures.

The model lipid mixtures CER6/Chol/FA/ChS/PL = 28/23/15/8/26 (w/w) with individual fatty acids (FA) have been studied. To prepare the samples the $SM_{bovine\ brain}$, DPPC, DPPE (PL=SM/DPPC/DPPE=1/2/1 m/m), CER6, Ch, ChS, palmitic acid (PA, C16:0), stearic acid (SA, C18:0), arachidic acid (AA, C20:0), behenic acid (BE, C22:0), and lignoceric acid (LA, C24:0) and their mixture FFA =SA/AA/BA/LA = 29/21/15/8/26 (w/w) have been used. Neutron diffraction experiment on the oriented samples has been carried out on the V1 membrane diffractometer, located at cold-neutron source of the BER-II research reactor (BENSC, HZB), with a neutron wavelength of 5.23 Å and a sample-to-detector distance of 102.52 cm. Small-angle-X-ray diffraction patterns from multilamellar vesicles (MLVs) have been collected on the small angle time resolved station DICSI at the K1.3a beamline of the Siberia-2 storage ring of the Synchrotron Radiation Source at the NRC "Kurchatov Institute".

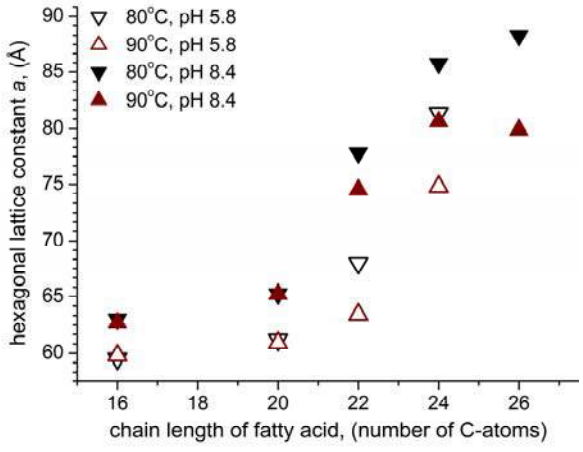

Fig. 1. The lattice parameter of the inverted hexagonal phase H_{II} as a function of hydrocarbon chain length of fatty acid for the membranes CER6/Chol/FA/ChS/PL at 80 and 90°C.

MLVs of OSC-model mixtures with PA, AA are characterized by presence of two lamellar phases with repeat distance of 46.4 and 42.2-43.8 Å in the temperature range of 20-37°C, at pH 5.8 and 8.4. At the same conditions the lipid mixtures with LA, BA and CA have a dominating phase with periodicity of 47-52.9 Å at pH 5.8 and 48.1-61.8 Å at pH 8.4. The inverted hexagonal phase H_{II} coexists with liquid crystalline lamellar phase at the temperature of 80-90°C. The parameter of the H_{II} phase increases with the hydrocarbon chain length of fatty acids and also increases with a change of pH from 5.8 to 8.4 for all lipid mixtures with individual fatty acids (Fig. 1). The lamellar phase of the membranes with long-chain fatty acids (AA, LA, BA and CA) is suppressed at pH 8.4.

The oriented membrane CER6/Chol/FFA/ChS/PL is characterized by coexistence of several structure phases. The main phase with repeat distance $d \sim 55$ Å at 98% relative humidity (phase "a" at Fig. 2) and the minor phase with $d \sim 58$ Å (phase "d") are likely phospholipids-enriched phases. The low swelling phase with repeat distance $d \sim 44$ Å at 58% relative humidity and $d \sim 46$ Å at 98% relative humidity (phase "b" at Fig. 2) has the structure similar to that of ESC-model membrane, first described in [2].

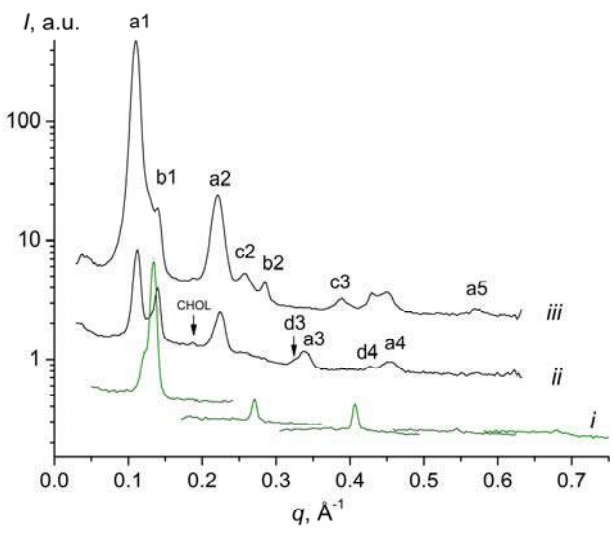

Fig. 2. Neutron diffraction patterns of oriented membrane CER6/Chol/FFA/ChS/PL = 28/23/15/8/26 (w/w) measured at 20°C, 58% RH, D_2O/H_2O = 8/92 (i), 37°C, 98% RH, D_2O/H_2O = 8/92 (ii) and 37°C, 98% RH, D_2O/H_2O = 80/20 (iii). Numbers indicate the diffraction orders for phases "a", "b", "c" and "d".

Earlier, electron microscopy revealed an inverse correlation between permeability and ceramide content for both skin and oral tissues [3]. One can suppose that there are some ceramides-rich domaines in the native oral lipid material, which responsible for control of permeability.

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ISOTHERMAL COMPRESSIBILITY AND THICKNESS OF LIPID BILAYER SIMULTANEOUSLY MEASURED UNDER HYDROSTATIC PRESSURE

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Introduction

The IBR-2M pulsed reactor (JINR, Dubna, Moscow region) was launched in operation in 2011. One of the instruments at the reactor is a YuMO small-angle neutron scattering spectrometer which is used for a wide range of scientific and technical applications.

The spectrometer is characterized by a relatively short data acquisition time which depends on the type of the studied material and varies from minutes to hours. In case of the results of the studies of lipid membranes under hydrostatic pressure which are described below the characteristic time was several minutes due to a high flux of the neutron beam [1]. In addition, the sample environment of the SANS instrument was also improved providing new opportunities for the studies in different fields of science. The neutron experiments were performed at YuMO spectrometer by a two-detector system mode with ring wire detectors [4,5]. The beam was collimated to a diameter of 14 mm on the sample. The data treatment was performed by SAS program [6] both with and without a smoothing mode [7]. In this report we describe a high pressure setup (4 kbar) recently installed at the YuMO [2]. We also present the new SANS D-P-V-T on lipid membranes data obtained simultaneously. It should be mentioned that the first test experiments with P-V-T measurements were done in [3].

Experiment

Synthetic 1,2-dipalmitoyl-sn-glycero-3-phosphocholine (DPPC) was purchased from Avanti (Birmingham, Al) and was used without further purification. Water (18 MV/cm) was obtained with Millipore (USA). Multilamellar vesicles (MLVs) were prepared in the following way: DPPC was mixed with water. Homogeneous dispersion was obtained by a temperature

cycling method around the main phase transition temperature with a temperature range ±20. The final concentration of the lipid/water was 10 mg/ml.

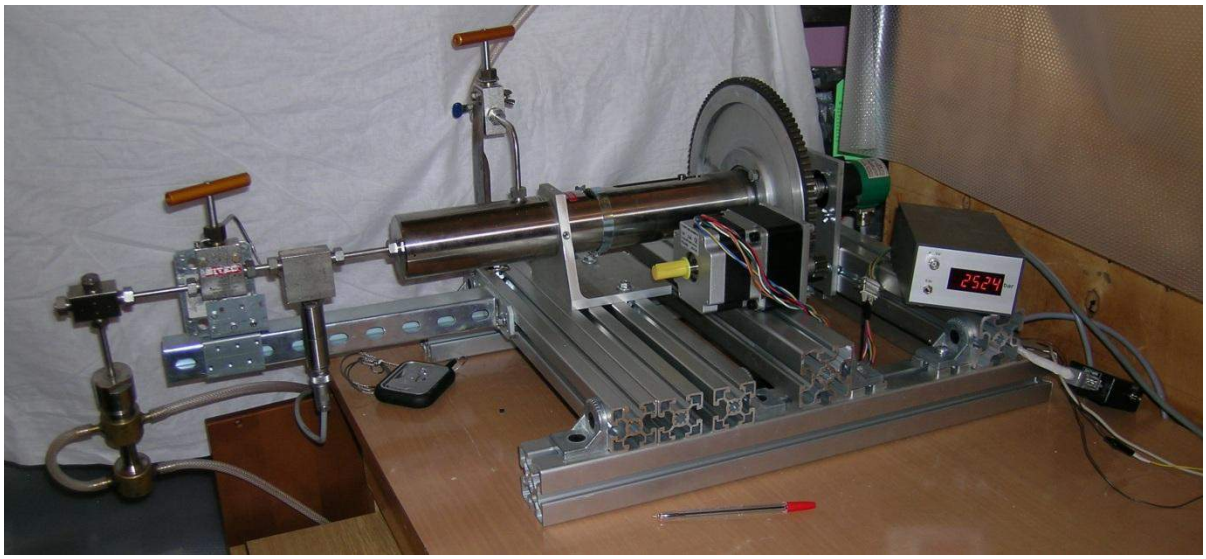

Fig. 1. Volumetric high-pressure setup of the YuMO spectrometer, adapted for SANS experiments.

The hydrostatic high-pressure setup was improved, namely, it was adapted to parallel volumetric measurements. For this purpose the rotation angle sensor was mounted at the hand pressure pump. It provides control of the change of the volume with the accuracy up to $\pm 2*10^{-6}$ ml. In addition, the pressure pump was equipped with a motor which gives a possibility to change the volume of the system at a rate about $2*10^{-3}$ ml/s. The electronic pressure sensor allows one to control pressure with the accuracy of about ± 0,1 bar. The temperature in the pressure cell is controlled by the LAUDA thermostat with an error ± 0,05°C.

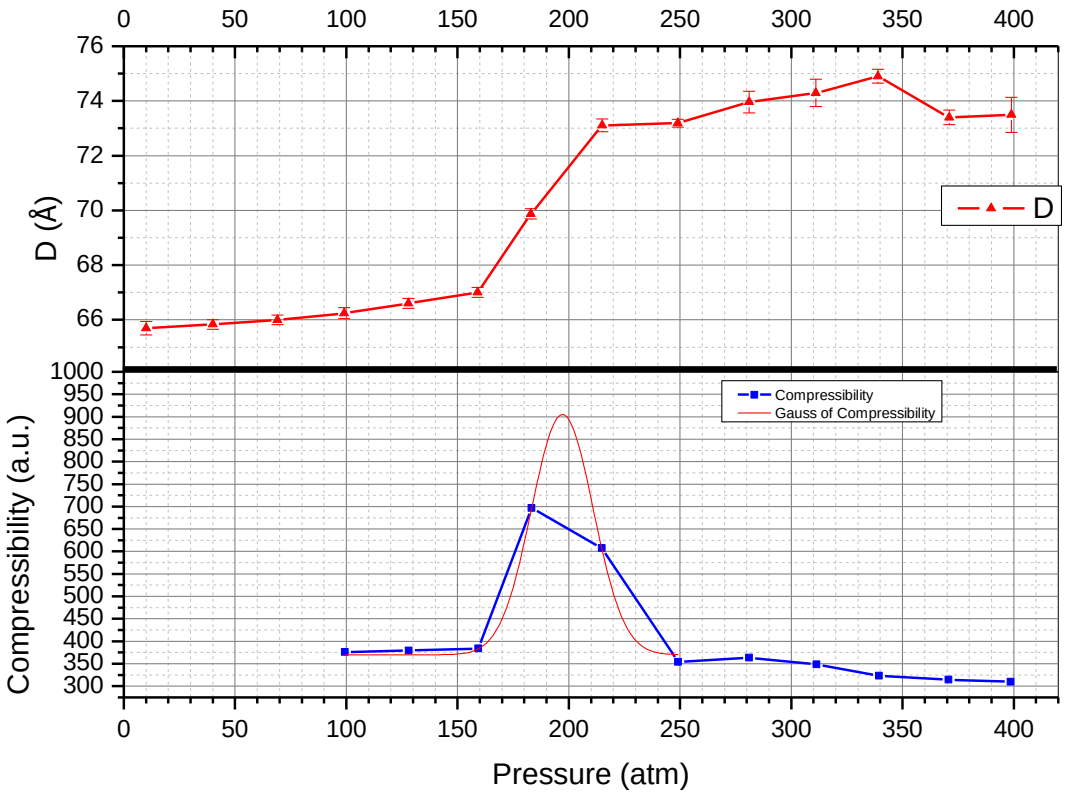

Fig.2. The DPPC main phase transition at 50°C and from 10 to 400 atm range. a) Bilayer repeat distance dependence D vs pressure and b) Membrane isothermal compressibility as the function of pressure.
The measurements were performed simultaneously.

Experimental data on bilayer repeat distance and membrane isothermal compressibility as the function of pressure are presented in Fig.2.

Simultaneous D-P-V-T measurements were done in the following pressure range: from 10 up to 400 atm with a step of 30 atm.

Acknowledgements

The work was supported by the work of Federal Target Program "Scientific and Pedagogical Staff of an Innovative Russia" (state contracts P-1160 and 02.740.11.0542). We are grateful for supporting Seventh Framework Programme [FR7/2007-2013] under grant agreement №211800.

We are grateful to Dr.A.Kh. Islamov for fruitful discussion.

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STRUCTURE OF CLUSTERS IN AQUEOUS DISPERSIONS OF NANODIAMONDS BY SMALL-ANGLE NEUTRON SCATTERING: EXPONENTIAL/POWER-LAW APPROACH

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Detonation nanodiamonds (DND) are formed by an explosion of unbalanced oxygen explosives in the absence of extra sources of carbon. DND particles are nanocrystallites of size 5–10 nm which, in the course of the explosion, produce almost inseparable aggregates through nondiamond components. Methods of "clarifying" dispersions which depend on grinding of DND in wet conditions have recently being developed [1]. As a result, stable colloid solutions based on various solvents are synthesized in which individual nanoparticles unite into clusters because of high free surface. In [2] a water–nanodiamond system was studied by the small-angle neutron scattering (SANS) method. The fractal structure of clusters was analyzed using an incomplete two-level model [3]. From a qualitative comparison of the scattering curves it was inferred that, when concentrated, the clusters partially penetrate each other. Similarly, qualitative comparison allowed for an analysis of changes in the scattering curves at different relations between the proton and deuterium components of the buffer (contrast variation), which, within uniform approximation, allowed for the estimation of the composition of the clusters.

The aim of the present work was to simultaneously study the fractal structure of nanodiamond clusters and the effect of cluster interaction, based on a complete two-level model [3]. We used SANS curves obtained at the SANS-1 small-angle diffractometer in HZG (Geesthacht, Germany). In the SANS curves for different solution concentrations (Fig.1a), we observe two power-law regions, which in the double logarithmic scale are linear, and the Guinier regime is observed in the small-q range. The scattering represents the two-level organization of particles in an aqueous suspension of DND: the cluster level and the level of nanodiamond particles. The power character of the curves in the first level (small q) suggests that the studied system exhibits fractal properties [4]. The curves were described within the framework of a universal exponential/power-law approach (Fig.1b) [3] (for notation see Ref.2).

From the derived value of the index $P = 2.4 < 3$, it follows that the organization of the nanodiamond particles in aggregates corresponds to mass fractals with dimension $D = P$, which is independent of DND concentration. The latter implies that the power decrease amplitude in the cluster level (B) depends only on the nanodiamond particles content of dispersion [4]. The D value points to the mechanism of the cluster–cluster aggregation limited by diffusion at the formation of the studied aggregates [5]. The index value in the other level $P_S = 4.2 > 4$ gives an indication of the so-called diffusion character of the surface of DNDs making up the aggregates [4]. The initial section of the spectrum gives the apparent radius of gyration R_g. At low concentrations (weak interaction between clusters), it corresponds to the cluster size. For uniform particles of spherical shape, the particle radius is related to R_g as $R=(5/3)^{1/2}R_g$, hence we arrive at the cluster size estimate at a level from 35 to 50 nm. The observed R_g of aggregates first increases with DND concentration (Fig.1b), which efficiently corresponds to a rather strong attraction component in the potential of cluster interaction. At the same time, from analysis of scattering into a zero angle G we draw a conclusion about the general repulsion of clusters, which coincides with qualitative analysis in [2]. The same is true, for example, for the interaction of free monocarbon acids in liquid colloid solutions of magnetic nanoparticles [6]. At concentrations over 5 wt % the radius of gyration begins to decrease and becomes smaller than the initial R_g at the lowest concentration. This indicates that the effective radius of interaction grows smaller than the cluster radius and agrees with the

conclusion about the partial overlapping of branched clusters [2]. The other quantities G_S, B_S increase linearly with concentration, accounting for the increase of DND particles in the solution.

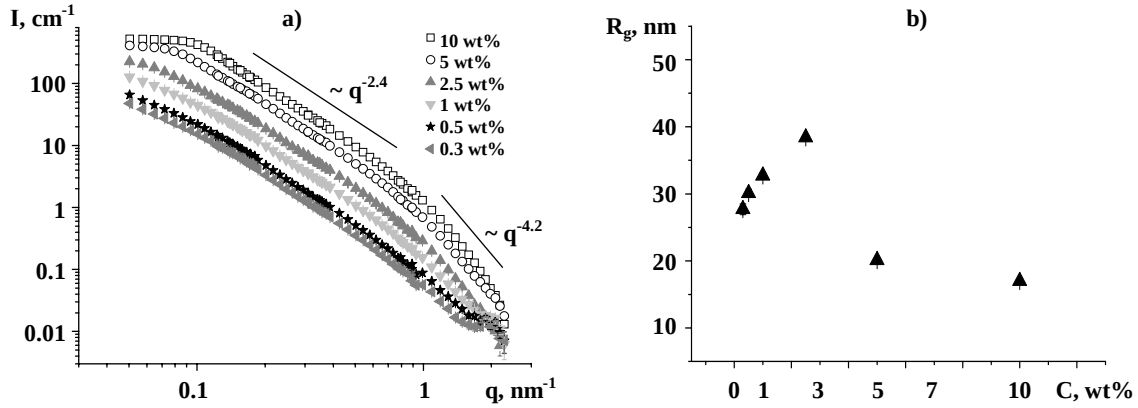

Fig.1. (a) SANS curves from DND water dispersions at different concentrations; (b) concentration dependences of the R_g-parameter.

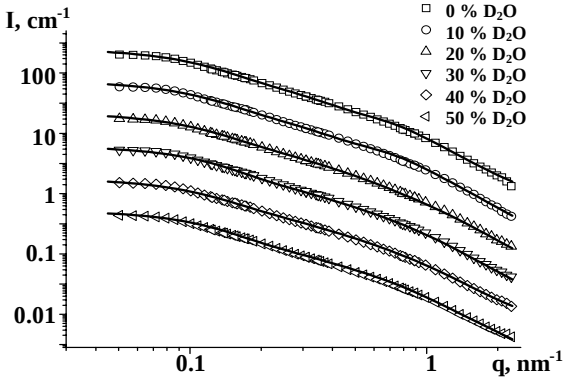

Fig.2. Contrast variation for a water–DND liquid system. For convenience of perception, the intensities are divided by: 10 for the system 10% D_2O; by 100 for the system 20% D_2O; by 1000 for the system 30% D_2O; by 10000 for the system 40% D_2O; by 100000 for the system 50% D_2O. Solid curves are approximations of exponential/power-law model.

The contrast variation procedure (Fig.3) allowed us to find the match point of the aggregates. The obtained value of the scattering length density is smaller than that of crystal diamond (ρ_{CD} = 11.8(3) × 10^{10} cm^{-2}), suggesting the presence of a non-diamond component in the DND particle composition. Since amorphous carbon and noncarbonic admixtures are almost entirely removed from the surface of nanodiamond particles during the preparation of dispersion [1], we may suggest that around the diamond's core there is a graphene shell, which is responsible for the diffusion character of the DND surface. The existence of such a shell follows from the spectroscopic data [2,7]. Also, the decrease of R_{gS} with contrast from 4.6(8) to 2.8(5) nm is consistent with this conclusion, because the shell is partially shaded from the scattering viewpoint as the share of heavy water in the buffer increases. Scattering decays monotonically with increasing deuterium component content of the buffer, P is invariant to contrast, which is evidence for the homogeneity of the aggregates under study.

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STUDY OF C_{60}/NMP/TOLUENE AND C_{60}/NMP/WATER SOLUTIONS BY UV-VIS SPECTROSCOPY AND SMALL-ANGLE NEUTRON SCATTERING

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Solutions of fullerene C_{60} in nitrogen-containing solvents and their mixtures with other solvents are characterized by the evolution of their UV-Vis, IR and Raman spectra in time. Two main processes, namely the formation of C_{60} clusters and change in the solute-solvent interaction, contribute to these phenomena [1,2]. These solutions exhibit sharp solvatochromism (change in the UV-Vis spectrum) under slight variations either the fullerene concentration or the solvent composition. Particularly, the addition of highly polar water with dielectric constant $\varepsilon \sim 80$ to the solutions of C_{60} in N-methyl-2-pyrrolidone (NMP) with $\varepsilon = 32$ is accompanied by a specific increase in the absorbance over the wavelength range of 450-550 nm [3-7]. It is supposed that this change in the UV-Vis spectrum is a result of the formation of charge transfer complexes between C_{60} and H_2O. Since NMP is miscible with both high-polarity (such as water) and low-polarity (such as toluene) solvents this later statement can be carefully checked. In the given work we studied the absorbance characteristics and structure of the solution of fullerene C_{60} in the mixtures NMP/toluene and NMP/water obtained by adding the third component to the initial C_{60}/NMP solution. For this purpose the combination of UV-Vis spectroscopy and small-angle neutron scattering (SANS) was used.

Fullerene C_{60} was dissolved in NMP with stirring during 15 minutes at room temperature. The initial mauve color of the solution after the preparation turned to brownish-yellow with time. Ternary solutions C_{60}/NMP/toluene and C_{60}/NMP/water were obtained by adding the third components to C_{60}/NMP with various proportions in different times after the preparation of the initial solution. Absorption spectra were obtained at the Hitachi U-2000 UV-Vis spectrophotometer in a wavelength range of 200-1000 nm. SANS experiments were carried out the 'Yellow Submarine' set-up of the Budapest Neutron Center.

UV-Vis spectra of C_{60}/NMP and C_{60}/NMP/toluene solutions are compared (Fig.1*a*) in the range of 300-600 nm, where the main specific changes take place. Particularly, they concern the characteristic peak at $\lambda \sim 330$ nm, for which the toluene addition to C_{60}/NMP system is accompanied by a small bathochromic effect (see inset to Fig.1*a*) with a maximal shift of about 4 nm towards higher wavelengths in the limiting case (toluene content of 98 vol. %).

The discussed spectrum region changes considerably when the toluene content exceeds 95 vol. %, so one can talk about a sharp solvatochromism implying somewhat a transfer from C_{60}/NMP to C_{60}/toluene absorption spectrum. However, even at the high content of toluene the color of C_{60}/NMP/toluene solution remains brownish yellow, which is a typical feature of the pure C_{60}/NMP solution. The observed effect is compared (Fig.1*b*) with the solvatochromism occurred at the addition of water to the C_{60}/NMP system, where the bathochromic shift of the peak at $\lambda \sim 330$ nm is about ten times larger than in C_{60}/NMP/toluene. Also, some additional absorption bands (plateau) in the range of 450-600 nm appear. All this points out a specific interaction of water with fullerene dissolved *first* in NMP. The observable changes in the character of the spectrum at the water addition take place at significantly lower water content (around 50 vol. %) [5-8] as compared to toluene. The same threshold of the water content was reported for the similar system C_{60}/pyridine/water [4].

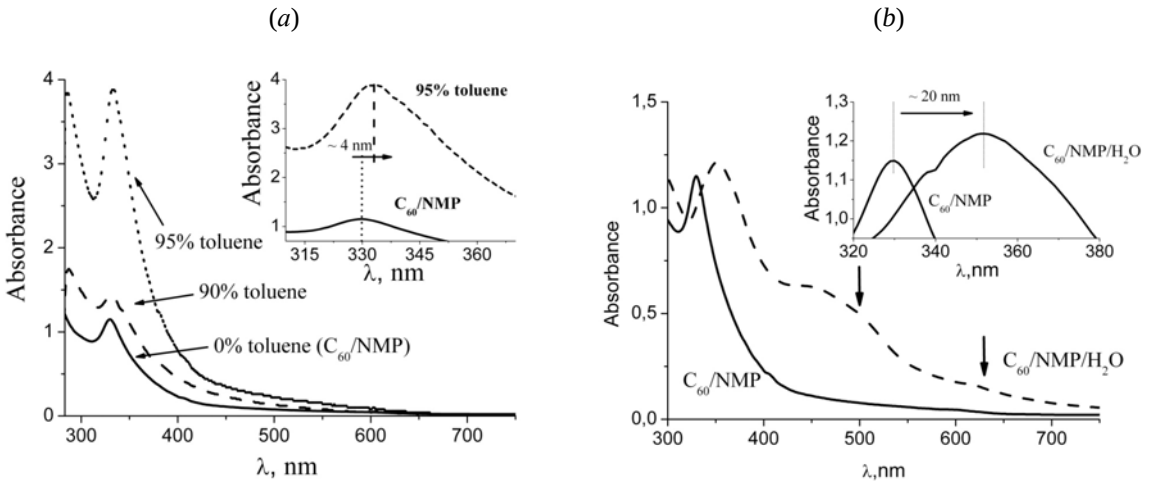

Fig.1. Solvatochromism under addition of toluene (*a*) and water (*b*) to C_{60}/NMP in various proportions. Final volume fractions of toluene in the mixtures are indicated in the graph. Water is added in 50 vol. %. Insets show bathochromic effects for characteristic peak at λ ~ 330 nm. Arrows denote newly appeared absorption bands in case of water addition.

The found difference in the effects of low-polar and polar additives can be related to the solvation processes. Toluene, as a low-polar component in C_{60}/NMP/toluene system, increases the so-called selective solvation, when C_{60} is preferably surrounded by NMP molecules. The corresponding solvation shell starts to be destroyed only at the toluene content close to pure C_{60}/toluene solution. Vice versa, at the water addition the interaction of C_{60}–NMP favours the formation of either the new complexes C_{60}–H_2O, which do not appear, if fullerene is added directly to water, or mixed solvation shells C_{60}–NMP/H_2O.

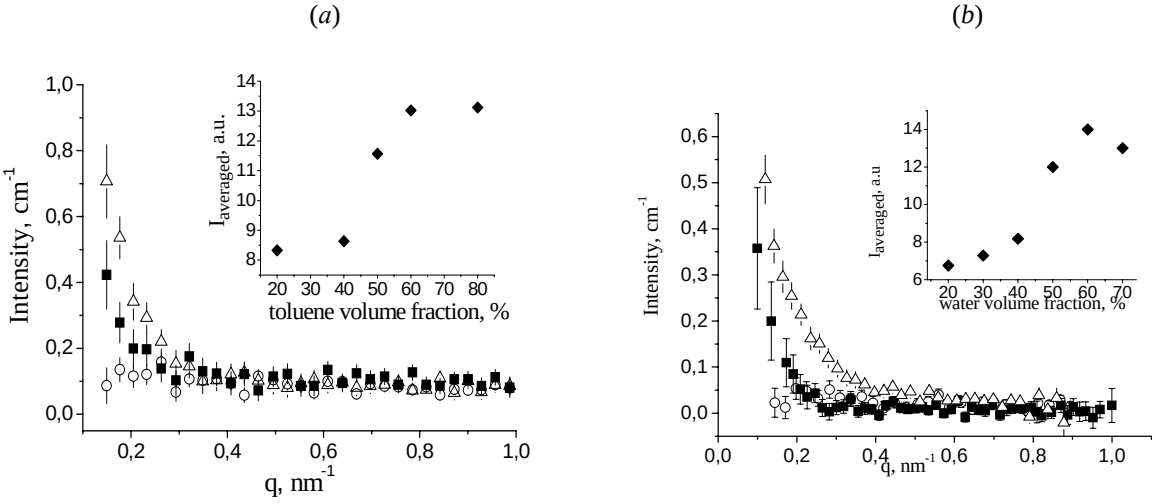

Fig.2. SANS data for C_{60}/NMP/toluene (a) and C_{60}/NMP/water (b) at various content of the third component in solutions. The curves are reffered to one fullerene concentration. Insets show dependence of the averaged intensity (q-range of 0.1-0.5 nm^{-1}) on the volume fraction of the third component.

At the same time, the small-angle neutron scattering (SANS) experiments (Fig.2) do not show any principal differences between toluene and water regarding the cluster reorganization in sufficiently old solutions. In both cases there is observed a sharp increase in the SANS intensity after the additive volume fraction becomes more than 40 % in the mixture. Such increase is explained by partial dissolution of the large (size above 100 nm) initial clusters whose preferable size lies after that in the region of nanolevel (10-100 nm). This means that the obsevred

solvatochromism in the studied solutions is not determined by the cluster reorganization, but is a consequence of the change in the donor-acceptor complexes of C_{60} with solvents.

The work was supported by the RF President grant MK-1055.2011.2.

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UPGRADE OF THE NEUTRON OPTICAL SYSTEM FOR THE DIFFRACTOMETERS EPSILON-MDS AND SKAT AT THE REACTOR IBR-2

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At beam line № 7 of the reactor IBR-2 were situated two diffractometers EPSILON-MDS and SKAT and the spectrometer for inelastic neutron scattering NERA. Both diffractometers EPSILON-MDS and SKAT have shared a common neutron guide with a section of 50 mm × 170 mm (w × h) and stood out due to a long flight path of more than 100 metres. The large cross section of the neutron beam was virtually split into two equal parts — the upper one provided the texture diffractometer SKAT with neutrons, the lower one served the strain diffractometer EPSILON-MDS.

It became evidently that this resolution had some drawbacks. Due to the close distance between this diffractometers and the weak shielding strong scatterer on the sample table of one diffractometer had generated a noticeable background for the other diffractometer.

During the shut down of the reactor the neutron-optical system of these diffractometers has been renewed: Instead of one straight guide two bent guides have been built. The set-up of the neutron guide system is finished. In the meantime the decision was made to renew the neutron guide for the spectrometer NERA, too. This should be done by the end of the year 2011 and then the diffractometers can be mounted at their new sides.

This is to introduce the new neutron optical system for the diffractometers EPSILON-MDS and SKAT. Between two pulses the reactor generates delayed neutrons from the fission during the power pulse and neutrons from the so-called satellites. All these neutrons do not obey any time correlations and can be seen as background. In order to reduce this background there is a background chopper installed at a small distance from the surface of the moderator, *i.e.* at a distance of 5.5 m.

The background chopper is a disk chopper rotating synchronously with the main reflector of the reactor, *i.e.* with a frequency of 5 Hz. Because there are three neutron guides the transparent part of the disk is calculated to be optimally with 60°. The diameter of the disk is 1205 mm. The cover sheets are made from a Ni-steel-alloy but the main part is a carcass made from an Al-alloy. The carcass is filled with bricks of sintered TiH_2 and a small amount of BC_4 (3 mass-%). The thickness of the disk is 100 mm.

Neither Ti nor H is a strong absorber but hydrogen has a rather high incoherent cross section. Therefore the attenuation of the beam is due "scattering out" the neutrons of their flight path. The background chopper maintains a wavelength band for the diffractometer EPSILON-MDS up to 14 Å, but due to frame overlap this band is limited to 7.2 Å.

Directly behind the background chopper starts the neutron guide splitter. The splitter has an overall length of 14 m and includes two channels of dimensions of 50 mm × 95 mm (w × h) and one channel with dimensions of 50 mm × 160 mm (w × h) cross section. Inside the splitter the neutron

guides are straight. In order to get a distance of minimum 1200 mm between the facilities at the end of the flight path the axis of the guide for NERA is inclined by -0.08° to the symmetry axis of the beam. The inclination of the guides for SKAT and EPSILON-MDS is +0.8° and 1.5°, respectively.

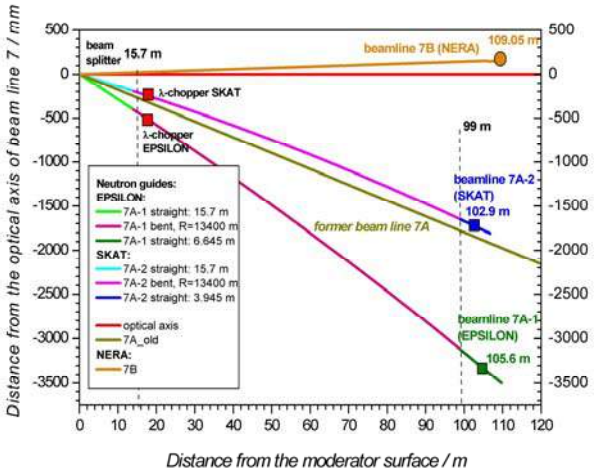

Fig. 1 (left): *Set-up of the neutron guide system at beamline 7 of the pulsed source IBR-2M.*

Fig. 2 (right): *Neutron guide system at beamline 7: 7A-1 (yellow) for the strain diffractometer Epsilon-MDS, 7A-2 (blue) for the texture diffractometer SKAT and 7A-2 (green) for the inelastic spectrometer NERA.*

For a flight path of 105 m the minimal velocity of neutrons is 525 m/s or the corresponding wavelength 7.54 Å for travelling from the moderator to the sample and detector within one frame between two pulses. These facts limit the measurement of peaks with lattice spacing up to 5.33 Å at angles of 2θ= 90°. For instance, many feldspar minerals have large unit cells and therefore they show diffraction peaks beyond this limit.

A frame overlapping can be avoided by suppressing every second power pulse. By this way, a doubling of the wavelength band can be achieved, but the intensity will be also reduced by 50 %. At the distance 22.5 m (EPSILON-MDS) and 25.6 m (SKAT) from the surface of moderator are installed additional choppers of drum-type. If needed, they are switched on and rotate with 150 rpm, *i.e.* the half of the frequency of the rotating main reflector.

After the splitter the neutron guides for both diffractometers are bent with a radius of 13 400 m and the bent part has a length of 85 m. This means, that the exit window of the neutron guide is shifted for 100 mm in respect with a straight guide. The last parts of the guides are straight again in order to get a more homogeneously neutron distribution at sample position.

Acknowledgements
The project has been supported by the German Federal Ministry of Education and Research (BMBF) under contract number 05K09CPA and the Frank Laboratory of Neutron Physics at JINR Dubna.

Neutron activation analysis of sediments and rocks from two lakes of Romania

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Introduction

Bâlea Lake (Făgăraş Mountains) and St. Ana Lake (Harghita Mountains) are two medium size lakes of different origin: Bâlea Lake is a typical high altitude proglacial lake while St. Ana Lal, located in the Ciomadu extinct volcano caldera, is the unique volcanic lake in Romania, Although both lakes have different location and origin, they are characterized by a total absence of any source of industrial pollution in their direct neighborhood. For this reason, and by taking into account the distance of about 115 km between them, these lakes could be chosen as good indicators for an extended screening of industrial pollution. On the other hand, Bâlea as well as St. Ana lakes, due to their picturesque landscape, are visited during summer time by a great number of tourists, so a certain degree of anthropogenic influence is expected to exist. Both lakes collect pluvial water from relative restricted areas (about 234 ha in the case of lake Bâlea and 147 ha for the lake St. Ana lake) so the mineralogical and elemental composition of their sediments will reflect the geochemical characteristics of surrounding geological formations. Accordingly, the sediments of Lake Bale are expected to reflect the mineralogical composition of the Suru Formation, mainly consisting of metamorphic rocks such as amphibolitic schists, quartzo-felspatic gneissic rocks and mylonites as well as limestones. On the contrary, the sediments of St. Ana lake which occupies the bottom of now extinct Ciomadu volcano, mainly consist of fragments of weathered andesite together with an considerable amount of phytodetritus, the last one originated from the coniferous and deciduous forests that cover the caldera walls.

Irrespective of their tourist interest, both lakes have received little attention regarding geochemistry, and particularly, the level of pollution with heavy metals, although their importance as reference systems for zero level industrial pollution cannot be ignored. For this reason, in summer 2008 and winter 2009 we performed two campaigns of a systematic investigation concerning the geochemistry of both sediments and neighbor geological formations by collecting six sediments cores as well as an appreciable number of samples of rocks to be analyzed by instrumental neutron activation analysis (INAA).

Experimental

Sediments collected from the Bâlea Lake consisted, at their upper part, mainly of blackish, seldom green-grayish silt, with fragments of vegetation and few aquatic invertebrates, while the deeper ones contained also sand and fine to coarse gravel. The granulometric analysis showed the predominance of silt up to 80%, with an almost normal distribution, while vegetal fragments, sand as well as gravel or lithic fragments accounted for the rest of 20%. On the contrary, St. Ana sediments were more homogenous, consisting of brownish silt, rich in fragments of vegetation and in some places, exhaling an odor of H_2S.

INAA was carried out at the research reactor of WWR IRT type, at the Moscow Engineering Physics Institute (Moscow). Well homogenized samples weighing 100–200 mg, together with the reference materials: IAEA-433, IAEA 140/TM, IAEA SL-1 and IAEA Soil-7, were irradiated in the vertical experimental channels with a thermal neutron flux density of 10^{12} n $cm^{-2}s^{-1}$ for 15–20 h. All gamma spectra were recorded by using a HPGe detector of ORTEC GEM 25185 type with an energy resolution of 1.85 keV for the 1332 keV of Co^{60} line.

Results and discussion

The results for the seven elements are reported in Table 1.

Investigating the distribution of above mentioned elements in Bâlea and St. Ana lakes, we have been interested to establish to which extent to these elements could be attributed an anthropogenic origin, and, at the same time, to evidence the relationship between sediments and the lithologiy of their, although reduced as surface, drainage basins.

Accordingly, in Table 1 we have reproduced the content of investigated heavy elements, to which, for comparison, we have added also the natural occurring, nonpolluting Sc. As reference systems, we have used the Upper Continental Core (UCC) and Pacific and Indian Oceans MORB as well as Romanian Regulations concerning the content of some heavy polluting elements in soils. The content of these elements in the sediments and rocks in different formations is given by means of box and whiskers diagrams in Fig. 1.

To identify and characterize possible sources of the determined elements Correlation Analysis (CA) and Principal Component Analysis (PCA) (Davies, 2002) were performed by means of StatSoft® Statistica 6.0.

The PCA plot of all seven elements (considering samples as variables and elemental contents as cases) is reproduced in Fig. 2.

Table 1. The average values, (Aver), and standard deviations, (St. Dev.) of investigated heavy elements in St Ana sediments (12 samples), St. Ana andesites (3 samples), Bâlea lake sediments (6 sampes), Bâlea lake rocks (8 samples). For comparison, the content of the same elements as defined by Romanian Regulations (Anonymous, 1997) together, with UCC (Taylor and McLennan, 1983), as well as Pacific and Indian MORB (ERDA, 2010) contents are reproduced too. Concentration of elements is given in mg/kg.

	Sample		Sc	Cr	Co	As	Se	Br	Sb
St. Ana Lake	Sediments	**StDev**	4.5	31	4.2	4.1	1.7	23.7	2.4
		Aver	0.9	3	0.4	1.0	0.4	7.4	0.9
	Andesite	**Aver**	4.5	29	3.6	2.4	1.2	28.3	1.3
		StDev	1.2	10	1.0	0.7	0.3	16.4	0.0
Bâlea Lake	Sediments	**Aver**	20.0	272	42.3	5.5	3.0	5.7	1.4
		StDev	0.8	39	5.3	1.8	0.5	1.5	0.5
	Paragneiss Paragneiss Paragneiss Paragneiss + chloritic schists with garnets Amphibolite +amphibolitic schist Paragneiss + amphibolitic schist Paragneiss + chloritic schists Paragneiss + chloritic schists	**Aver**	24.9	291	39.3	4.3	2.0	0.4	0.2
		StDev	16.0	274	24.5	-	0.8	0.2	0.2
Reference systems	RR normal		-	30	15	5	1	50	5
	RR alert		-	100	30	15	3	100	12.5
	UCC		11	35	10	1.5	0.05	nd	0.2
	MORB Pacific		31.8	714.3	nd	nd	nd	nd	nd
	MORB Indian		39.1	254.4	nd	nd	nd	nd	nd

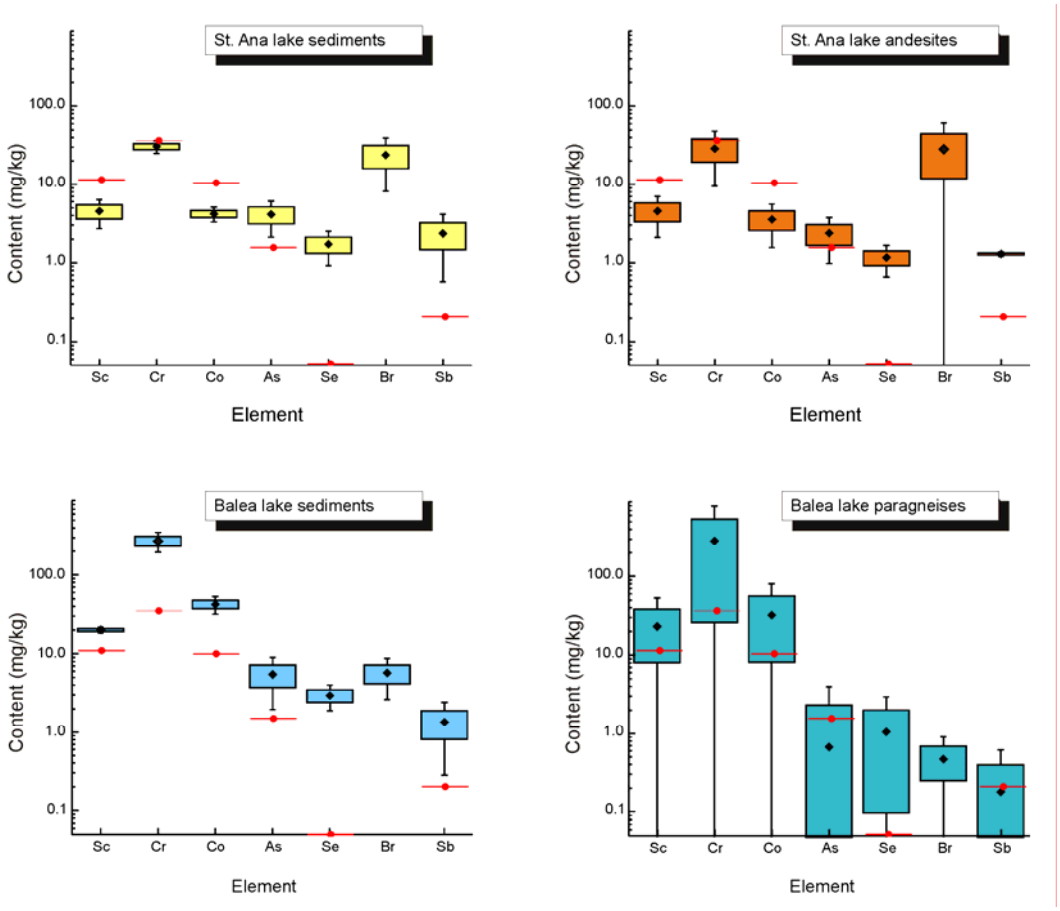

Fig. 1. Box and whiskers diagrams of determined elements in investigated systems

In addition, by means of PCA considering samples as cases, an attempt was made to understand the interrelationship between sediments and adjacent parent rocks (Fig. 3).

In this way, we can consider that observed clusters and sub-clusters, based only on the content of the considered seven elements, reflect with enough precision the differences between investigated samples regarding their natural provenience.

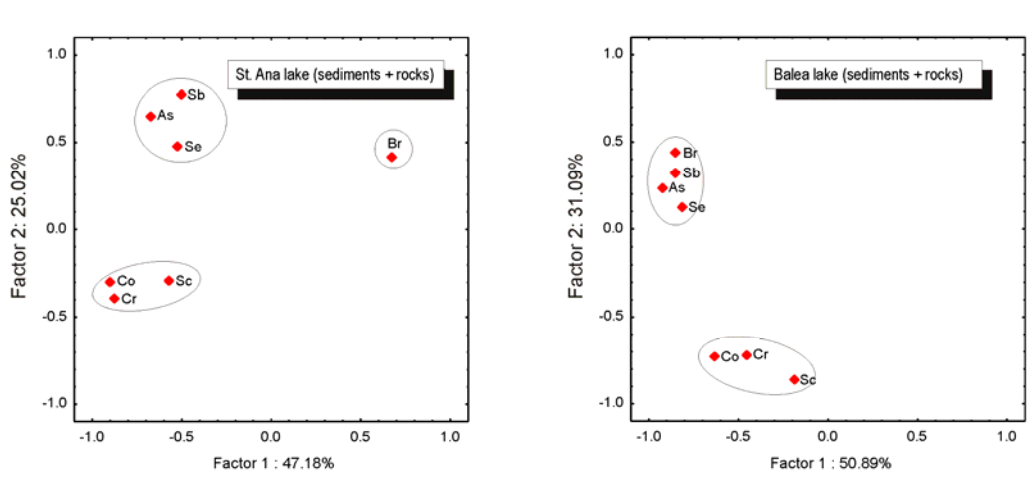

Fig. 2. A bivariate PCA plot illustrating the relative similarity of heavy elements clusters for both lakes (R mode analysis)

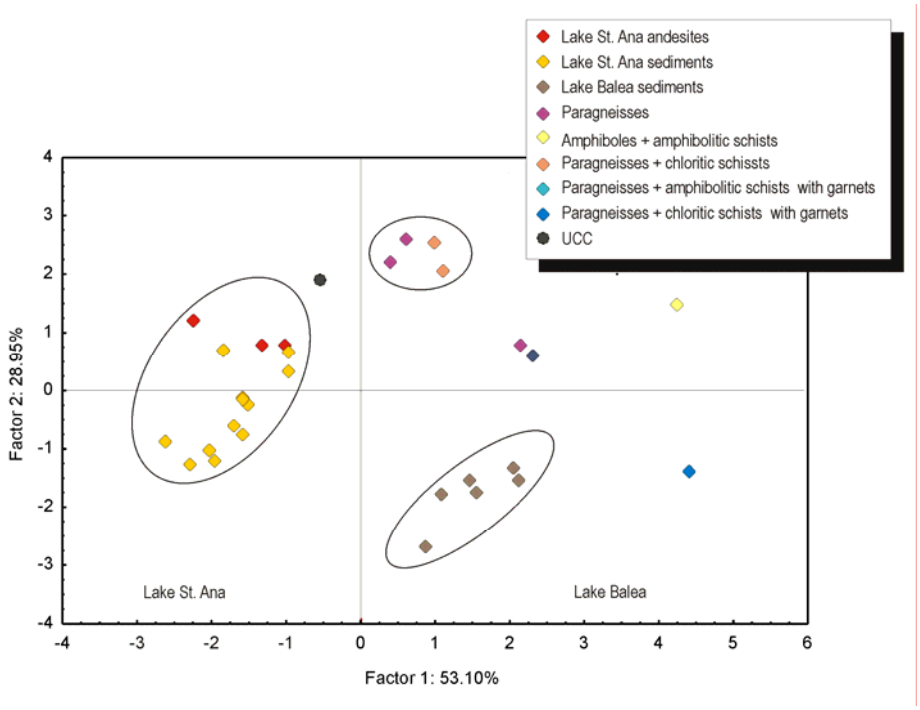

Fig. 3. A bivariate PCA plot illustrating the clusterization of the main lithologic components of investigated systems (Q – mode analysis)

Conclusion

Final results showed that the average content of these elements in sediments was close to these in rocks, and, at the same time, comparable with the normal environmental content of these elements as defined by the Romanian Regulations, such that all elements could be considered as normal, nonpolluting components of investigated lakes. A Principal Component Analysis performed in R-mode showed that Sc, Cr and Co on one hand and As, Sb, Br, and Sb on the other forms two distinct cluster, regardless the lake while Q-mode analysis pointed towards a significant difference between two lakes regarding both sediments and rocks.

Acknowledgement *This work was done within Romanian Ministry of Education and Research 31-013 Grant (Alpimp) and the University of Bucharest and JINR – Dubna, cooperation protocol # 4077-4-11/13.*

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APPLICATION OF SOME MICROORGANISMS FOR SYNTHESIS OF GOLD AND SILVER NANOPARTICLES

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Introduction

Nanobiotechnology is a rapidly advancing area of scientific and technological opportunity that applies the tools and processes of nano/microfabrication to build devices for studying biosystems. The microorganisms (bacteria, microalgae, yeasts, fungi) are often used as possible "nanofactories" for the development of clean, nontoxic and environmentally friendly methods of producing silver and gold nanoparticles (Klaus-Joerger et al., 2001; Li et al., 2011; Mohanpuria et al., 2008; Gericke and Pinches, 2006). Successful collaborative studies of the Sector of NAA and Applied Research of the Division of Nuclear Physics of FLNP with the Institute of Physics, Georgia, in the interaction of metals with microorganisms (Mosulishvili et al., 2002, 2007) were extended to nanobiotechnology (Tsibakhashvili et al., 2011; Kalabegishvili et al., 2011).

Materials and methods

Neutron activation analysis (NAA) is used among the variety of analytical and spectral methods: UV-vis spectrometry, X-ray diffraction (XRD), transmission electron microscopy (TEM), scanning electron microscopy (SEM) with energy-dispersive analysis of X-ray (EDAX), atomic absorption spectrometry (AAS) for investigation of the obtained nanomaterials. The few bacterial strains of actinomycetes *Streptomyces glaucus* 71MD and *Streptomyces* spp. 211A (isolated from the rhizosphere of soybeans grown in Georgia), arthrobacter genera – *Arthrobacter globiformis* 151B and *Arthrobacter oxydans* 61B (isolated from the basalt rocks collected from the Kazreti region of Georgia) and blue-green algae *Spirulina platensis* (strain IPPAS B-256 from the algeological collection of the Timiryazev Institute of Plant Physiology, Russian Academy of Sciences) were used for gold and silver nanoparticles synthesis. Cells of actinomycetes and *Spirulina platensis* were grown as described elsewhere (Tsibakhashvili et al., 2011; Kalabegishvili et al., 2011). The harvested mycelial mass was then resuspended in 250-ml Erlenmeyer flasks in 100 ml of 10^{-3} M aqueous $HAuCl_4$ (chloroauric acid) solution in the synthesis of gold nanoparticles, and in aqueous $AgNO_3$ (argentum nitrate) solution in the synthesis of silver nanoparticles. Time-dependence of nanoparticle formation was studied in different time intervals (several days).

Result and discussion

The gold and silver surface plasmon resonances (SPR) was observed in the UV-vis absorption spectra at ~ 530 nm for gold and at 425 nm for silver, respectively. The presence of SPRs indicate the gold and silver ion reduction and the subsequent

aggregation of nanoparticles in the solutions. The intensity of the peaks increased as a function of the reaction time. A single band in all spectra gives evidences for the spherical shape of gold and silver nanoparticles that is also confirmed by TEM images (Fig. 1).

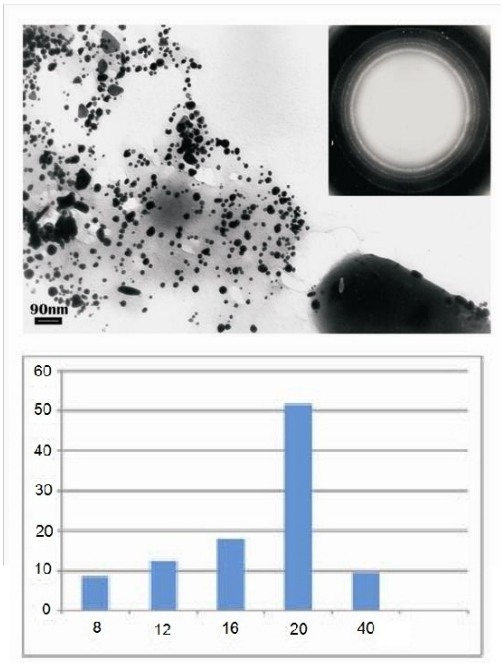

Fig. 1. TEM image and size histogram of Au nanoparticles in biomass of Arthrobacter *61B*

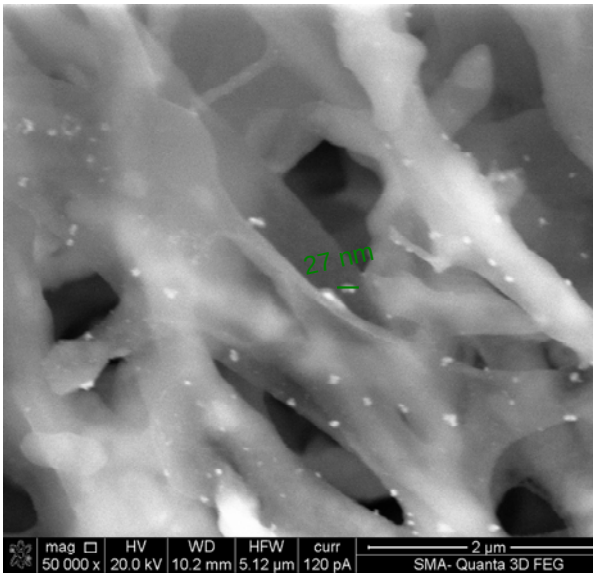

Fig. 2. SEM image of Ag nanoparticles in biomass of *Streptomyces glaucus* 71MD

In all TEM images the diffraction patterns correspond to the face centered cubic (fcc) structure of gold and silver nanoparticles. The particle size histogram for studied samples shows that the size of gold and silver nanoparticles are in the range of 5 to 80 nm, with an average of 25 nm. The XRD data for gold and silver nanoparticles confirm the presence of the fcc structure. As an example, the SEM image of *Streptomyces glaucus* 71MD cells (after interacting with $AgNO_3$ solution for seven days) is given (Fig. 2). The SEM images illustrate that most of the particles are spherical and do not create big agglomerates.

The EDAX X-ray spectra were registered proving the presence of gold nanoparticles in *Spirulina platensis* cells treated with $HAuCl_4$ for 5 days (Fig. 3). Along with the Au peaks, the signals from C, O, Cl and Fe were recorded. Neutron activation analysis (NAA) was carried out in collaboration with the South African Nuclear Energy Corporation (Necsa), Pelindaba, Pretoria, South Africa, at the nuclear research reactor SAFARI-1. The samples were irradiated for 8 s at a neutron flux density of ~ 5.10^{14} n cm^{-2} s^{-1}. Their activities were measured three times, after cooling for 3 and 30 hours and 7 days, respectively. The gold content was determined on the 411.8 keV γ-line of ^{198}Au. Genie 2000 software was used to process NAA data. The data obtained by NAA illustrates that uptake of metals includes two phases: rapid and slower uptake. In the first 'rapid' stage, the metal ions are adsorbed onto the surface of the microorganism. The concentration of gold increases rapidly. In the 'slow' stage, the metal ions are transported across the cell membrane into the cytoplasm. The total concentration of gold in the samples (extracellular and intracellular) does not change significantly. The data obtained by AAS (Fig. 4) are confirmed by NAA (Fig. 5).

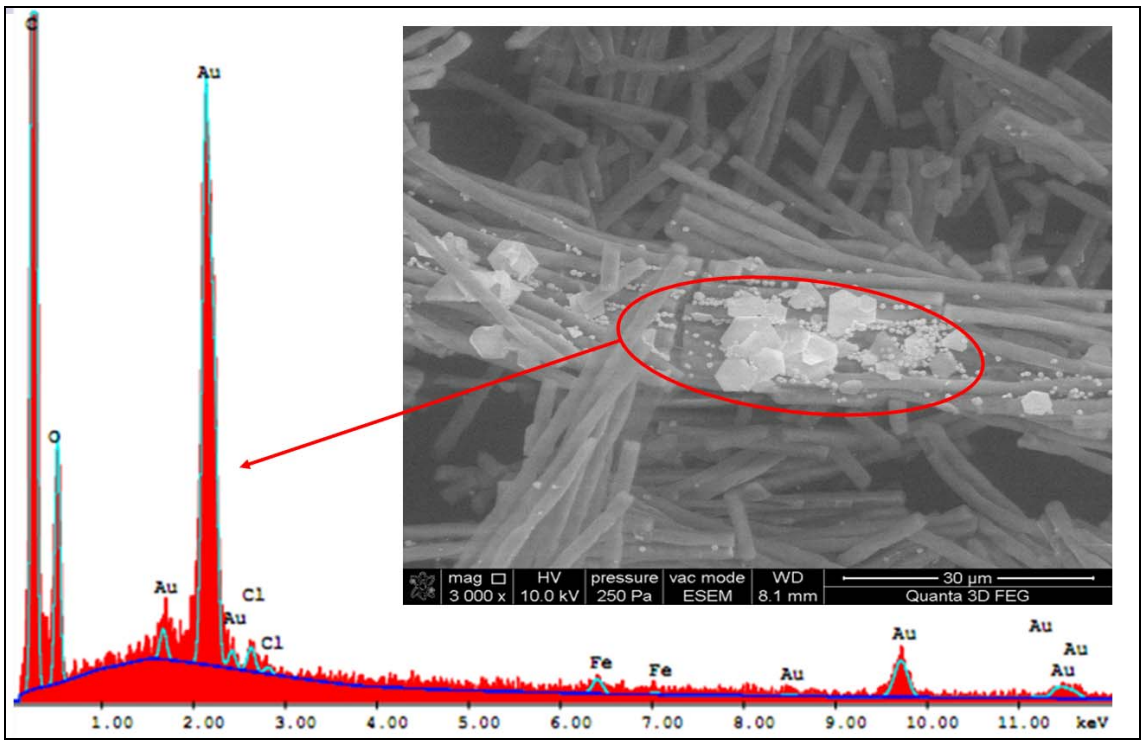

Fig. 3. EDAX spectrum of *Spirulina platensis* exposed to $HAuCl_4$ (10^{-2} M)

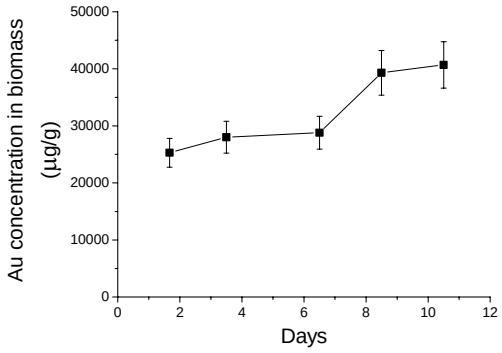

Fig. 4. The gold concentrations in biomass of *Arthrobacter globiformis* 151B versus the time of exposure gold cloroaurat determined by AAS

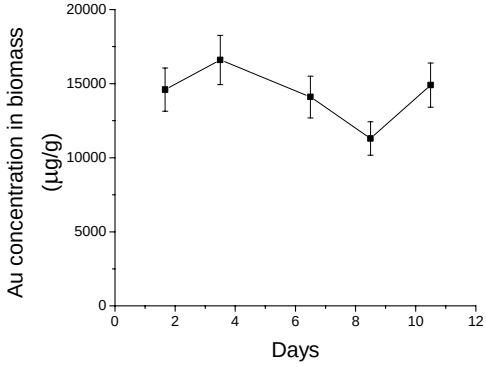

Fig. 5. The gold concentrations in biomass of *Arthrobacter globiformis* 151B versus the time of exposure gold cloroaurat determined by NAA

Conclusions

The results of the performed investigations show that the studied microorganisms are capable of producing nanoparticles extracellular when exposed to the gold and silver compounds. The shape of the majority of the nanoparticles is spherical and the average size is 25 nm. The biosynthesis of nanoparticles is simple, economically viable and an eco-friendly process.

The authors acknowledge the Ukrainian Science and Technology Centre (STCU Grant # 4744) and the Grant of the JINR-South Africa Collaborative Agreement (Order # 486, 2010).

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Editors: A.V.Belushkin, O.A.Culicov

Translated from Russian: T.V. Avdeeva

Typesetting: T.V. Avdeeva, I.N. Guseva

Cover: V. O. Tamonova

Photography: Yu.A.Tumanov, E.V. Puzynina, P.E. Kolesov

ISBN: 978-5-9530-0317-9

2011
Annual Report