Synchrotron Radiation Techniques for Catalysts and Functional Materials

ABSTRACTS

November 11 - 14, 2024,
Tomsk

Federal Research Center Boreskov Institute of Catalysis
Synchrotron Radiation Facility SKIF
Budker Institute of Nuclear Physics of SB RAS
Novosibirsk State University
National Research Tomsk Polytechnic University

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Основные темы научной программы конференции:
The collection includes abstracts of plenary lectures, oral and poster presentations.
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- Structure-Driven Design of Catalysts and Functional Materials Based on Synchrotron Diagnostics
- Synchrotron Radiation for Structural Biology
- Development of Instrumentation for Synchrotron Beamlines
- New Data Processing Algorithms, Artificial Intelligence and Machine Learning in Bulk Data Analysis

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Plenary Lectures

PL-1 ÷ PL-8

PL-1

Key activities of the Tomsk Polytechnic University related to synchrotron and neutron instrumentation development and research

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PL-2

Synchrotron X-ray radiation for porous metal-organic frameworks. Gas adsorption and separation applications

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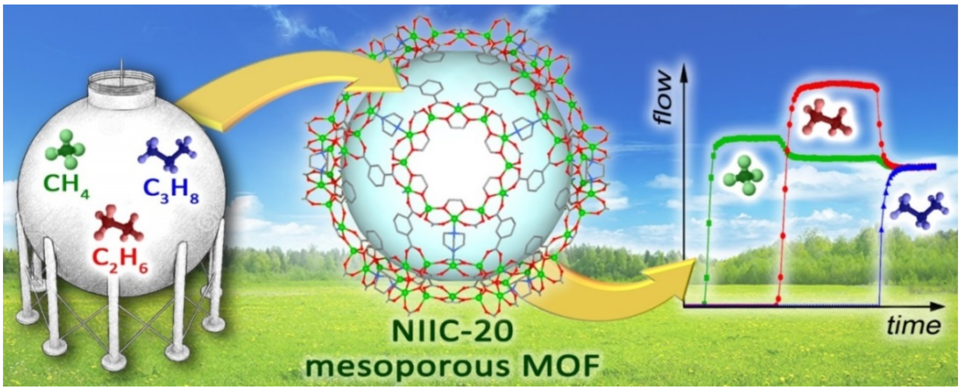
Metal-organic Frameworks (MOFs) are an important class of porous materials that have been intensively studying for the last two decades. An important advantage of MOFs is the ability to precisely design geometry of the pores as well as an arrangement of functional groups on the inner surface of the framework, which predetermines their unique adsorption properties with respect to various substrates. Synchrotron Radiation X-ray diffraction analysis is a very powerful tool, not only affording comprehensive information on the crystalline structure of the framework, but visualizing the adsorbed molecules inside the pores, thus providing valuable insights on both the nature of the adsorption centers and the type of intermolecular interactions that ultimately determine the adsorption sorption properties of the porous material.

A number of exciting examples of applications of synchrotron X-ray diffraction techniques in MOF area will be presented. Also, recent results on the design of porous MOFs with high adsorption characteristics towards gases and vapors of volatile substances will be reported [1-3]. Record-high adsorption selectivity has been achieved for a number of industrially important gas mixtures. The preparative separation of multicomponent mixtures of light hydrocarbons has been demonstrated under conditions simulating industrial technological processes.



Acknowledgement: This work was supported by the Russian Science Foundation, grant 23-13-00310.

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PL-3

SR in the study of electronic and spin structure of functional materials: capabilities of the ARPES station at the SKIF synchrotron

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The development of photoelectron spectroscopy with angular and spin resolution (SR-ARPES) has opened the way for direct measurement of the electronic structure of solid surfaces and has played a huge role in the discovery and understanding of new quantum and functional materials, whose properties are determined by collective effects or their symmetry and nontrivial topology. Currently, ARPES is successfully applied to study the electronic structure of such systems as cuprate and iron-containing high-temperature superconductors, graphene and graphene-like materials, transition metal dichalcogenides and topological quantum materials, atomic-pure surfaces with adsorbates, etc. It can be noted that historically the development of the ARPES method has been in accordance with the problems solved with its help. In particular, high-resolution electron spectrometers were created in response to the need to study sub-millivolt energy pseudo-slits in superconductors. With the discovery of topological insulators and the emerging interest in transition metal dichalcogenides, methods for electron spin detection in ARPES began to be actively developed. Further development of the ARPES method can be directly related to the development of synchrotron radiation sources to obtain previously inaccessible spatial and temporal resolution, allowing the study of systems that were previously considered unsuitable for this method due to inhomogeneities or the impossibility of obtaining an ordered surface of sufficient area for the study.

In contrast to developed countries (and not only), the ARPES method has not been practically developed in Russian Federation and is represented only by a few working laboratory installations in St. Petersburg (SPbU), Vladivostok (IACP FEB RAS) and Novosibirsk (ISP SB RAS). Figure 1 shows examples of photoemission spectra and Fermi surface maps for the surface of different systems measured at the ISP SB RAS: atomically clean Si (111) surface with surface reconstruction (7×7), epitaxial graphene on SiC surface and (0001) surface of three-dimensional topological insulator Bi2Se3.

Despite the high energy and angular resolution of modern laboratory ARPES systems, all the possibilities of this technique are fully revealed only when using radiation sources based on synchrotrons, which allow us to vary the energy of SR in the range from soft UV (10 eV) to soft X-ray (1.5 keV) with a high degree of monochromaticity and high intensity. Synchrotron radiation sources of generation 4 and 4+ with ultra-low emittance allow, in addition to increasing the intensity, to easily obtain a small diameter of the radiation spot on the sample.

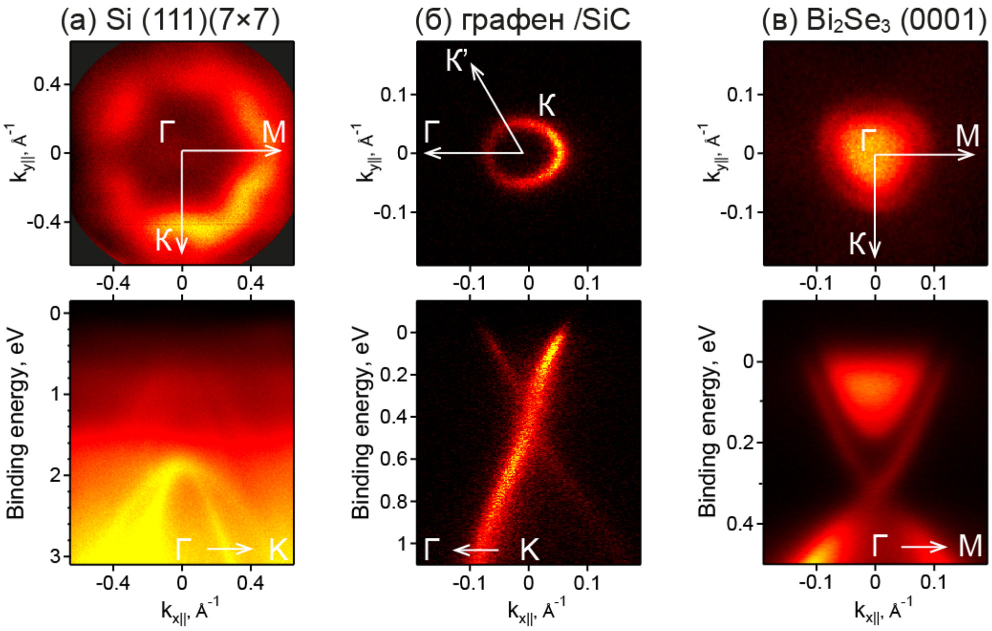

Fig. 1. Fermi surface maps and dispersion bands measured by the ARPES method on (a) Si(111) surface with (7x7) reconstruction - the bulk states of light and heavy holes and the dispersion of surface states in the forbidden gap are visible (hν = 21. 2 eV, 300K), (b) epitaxial graphene on 6H-SiC with (6√3×6√3) reconstruction at the K-point (hν = 40.81 eV, 300K), (c) cleaved (0001) surface of the three-dimensional topological insulator Bi_2Se_3 *(hν = 21.2 eV, 300K)*

At present, the key elements of the station are being designed to ensure the following parameters and capabilities:

1) The energy range of photoemission-inducing radiation is from 10 to 1500 eV, with the possibility of arbitrary choice of polarisation and photon flux not less than 10^{12} photons per second;

2) The size of the radiation spot on the sample is less than 10×10 µm, with the potential possibility of focusing into the area of 1×1 µm;

3) Electron energy analyser with a deflector, providing a limiting energy and angular resolution of the system of less than 1 meV and 0.1°;

4) Sample temperature during photoemission measurements from 7 to 400 K.

5) New type highly efficient three-dimensional photoelectron spin detector developed at ISP SB RAS.

PL-4

Advanced In-Situ methods based on microfocus synchrotron X-Ray scattering for the study of functional materials

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Modern synchrotron sources offer materials scientists a wide range of methods to study the structure and self-organization processes in materials. The high intensity of X-ray beams, achieved with third- and fourth-generation sources, along with the ability to focus them to submicron sizes, enables the collection of structural data with high spatial and temporal resolution.

One example of using nanofocused X-ray diffraction to study molecular self-assembly processes is described in the case of sector-shaped mesogens self-organizing within the pores of a nanoscale aluminum oxide template [1]. This method allows 2D scanning of the sample with submicron steps and quantitatively determines the degree of orientation of the columnar phase of the mesogens inside the nanopores, depending on the distance from the free surface of the membrane.

The combination of microfocus diffraction, grazing incidence diffraction, and in-situ ultrafast calorimetry enables a detailed analysis of the structure, texture, and thermal behavior of functional materials with complex hierarchical organization [2-3].

Acknowledgement: This work was supported by the Russian Science Foundation, grant 14-13-12345.

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PL-5

X-ray absorption spectroscopy: state-of-the-art and perspective

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X-ray absorption spectroscopy is a powerful technique that can be used to study the local spatial, electronic, and magnetic structure of a wide range of objects, regardless of their aggregate state (gas, liquid, solid). The absence of a long-range order requirement permits the study of both crystalline and amorphous materials, including glasses, nanoparticles, single-atom centers, and clusters. A significant benefit of X-ray absorption spectroscopy is its ability to discern the presence of specific elements, which can be utilized to identify impurities in alloys or trace heavy elements in biological matrices. XAS studies are required in a number of scientific fields, including solid-state physics, materials science, geology, environmental science, inorganic chemistry, colloidal chemistry, heterogeneous and homogeneous catalysis, and microelectronics.

The lecture will address the current state of the art to carry out XAS study of functional materials (catalysts, photocatalyst, batteries, etc.) with a particular focus on *in situ/operando* studies and the further development of *in situ/operando* XAS studies. The potential for combining these techniques with other analytical methods will also be discussed.

This lecture will outline the opportunities afforded by SKIF for X-ray absorption spectroscopy studies. The beamlines 1-4 “XAFS-spectroscopy and magnetic dichroism” will be observed. The concept of relocating the EXAFS spectroscopy beamline from the Siberian synchrotron and terahertz radiation center to SKIF will be presented, and the new opportunities afforded by the renovated beamline will be discussed.

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PL-6

In situ study of high-temperature ceramic coatings

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Currently, there is a constant search for the new thermal protection coatings for gas turbine engines [1-4] due to the increasing requirements for them. The temperature of gases in the turbine is directly proportional to the efficiency of the engine, so the key task in the development of aircraft engines is the creation of new high-temperature materials and methods of their protection [5-7]. One of the promising ceramic systems for thermal protective coatings today is the Y-Al-O system. [8]. For the compound of $YAlO_3$ the average coefficient of thermal expansion is 6…7*10^{-6} °C^{-1} and the density is 5,35 мг*$м^{-3}$ [9]. One of the key properties of YAlO3 oxide is the absence of phase transformations in the temperature range from room temperature up to the melting temperature of 1913°C, which allows us to suppose the stability of the coating under thermal loads and the absence of stresses and volume changes during phase transitions, that is typical for many other ceramic systems. In due to the growing interest in new coatings, in situ study of their formation on the substrate during deposition or sputtering is of particular interest, as well as testing in approximate operating conditions will optimize the application processes of such coatings.

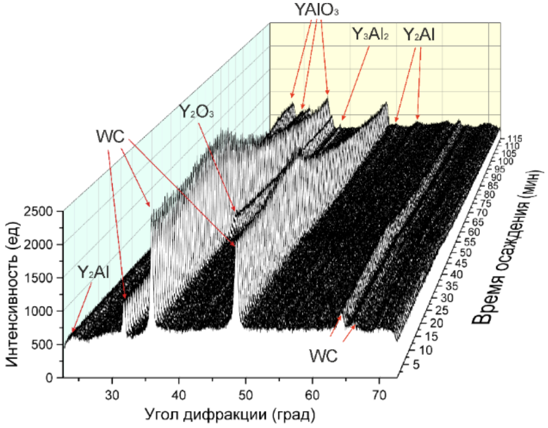

Fig. 1 Set of diffractograms obtained in situ during vacuum-arc deposition of Y-Al-O coating

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PL-7

Structural biology of DNA repair

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Our genomes are under constant threat of damage inflicted by both environmental assaults and harmful byproducts leaking from metabolic pathways. DNA repair systems remove damaged nucleotides thus preventing mutagenesis and cell death. Six major DNA repair pathways are known at present (direct reversal, base excision repair, nucleotide excision repair, mismatch repair, recombination repair, and non-homologous end joining), of which base excision repair (BER) processes the most abundant DNA lesions arising from oxidation, deamination, and spontaneous base loss. Several DNA glycosylases initiate this process, specifically excising damaged bases, followed by DNA nicking by apurinic/apyrimidinic endonucleases, nucleotide replacement by DNA polymerases, and nick sealing by DNA ligases. Based on their sequences and three-dimensional structures, all these enzyme classes are divided in several superfamilies. An interesting feature of many DNA repair enzymes is that some structurally closely related ones can have different substrate and even reaction specificity, while some unrelated enzymes can share the same substrate specificity. Additionally, all DNA repair proteins face a hard problem of finding rare DNA lesions against a huge excess of normal DNA. A combination of structural, computational and biochemical methods is required to establish the principles of lesion search, discrimination and removal by DNA repair proteins. Mechanistic understanding of the enzymes involved forms the basis of rational development of new anticancer, antibacterial, and immunoregulatory drugs.

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PL-8

Application of X-ray atomic pair distribution function analysis to structural studies of catalytic materials

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Structural diagnostics is an important step in understanding the physicochemical and functional properties of catalytic materials. The X-ray powder diffraction is widely used to obtain the information about the composition and structure of solid catalysts. Most of the reported studies in heterogeneous catalysts include XRD data on the crystalline components detected in the catalysts, their average crystal structure, and the average size of coherently scattering domains. However, the majority of the heterogeneous catalysts rely on highly dispersed nanoparticles (<3 nm in size) to maximize the availability and efficiency of active sites. Noble metal-based catalysts typically contain supported species dispersed on the support surface at the cluster or atomic level. In addition, the catalytic systems often include nanostructured and structurally disordered solids. As a result, the XRD profiles exhibit highly broadened, indistinguishable Bragg reflections or pronounced diffuse scattering. Structural characterization of such systems by traditional XRD methods, which deal with positions, intensities, and shapes of the Bragg peaks, is impossible.

Atomic-scale structure characterization methods are needed to identify ultra-disperse components, to solve their structure, and to reveal the local structural peculiarities and defects in nanocrystalline components. An efficient and valuable technique is X-ray atomic pair distribution function (PDF) analysis. The PDF is obtained by Fourier transformation of the total X-ray scattering data, which include both Bragg diffraction and diffuse scattering. The PDF is a probability function describing the distribution of interatomic distances. The positions of peaks in the PDF correspond to interatomic distances, while the amplitudes depend on the number of atoms at these distances. PDF analysis is an excellent tool for studying the atomic structure of highly dispersed or poorly crystallized particles with only short-range atomic arrangement, for estimating the average size of atomic arrangement domains, and for revealing changes in the short-range atomic arrangement in the crystalline solids. Such structural information as a set of interatomic distances, coordination numbers and the average size of the atomic arrangement region can be obtained from simple, model-free analysis. In-depth, quantitative structural analysis implies suggestion of structural models, their verification and refinement by calculating model PDFs and fitting them to the experimental PDFs.

In the context of the application of the PDF for structural studies of catalytic materials will be discussed: structure solution of supported ultra-dispersed particles and clusters in supported catalysts, identification of poorly crystallized and highly dispersed components in support materials and composite catalysts, characterization of the nanostructured materials. Available programs for calculation and refinement of PDFs, as well as modern trends in the development of the PDF-based studies will be mentioned.

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Oral Presentations

OP-1 ÷ OP-26

OP-01

Synchrotron studies of tin oxide nanolayers atomic and electronic structure

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Tin oxide is very promising material for production of the novel electronic devices such as resistive gas sensors with advanced characteristics: miniaturization and low energy consumption undergo with very developed technologies aiming at low thickness active nanolayers or other forms e.g. nanowires. This study aims at investigation of their formation specifics by several approaches: magnetron sputtering (MS), metal-organic chemical vapour deposition (MOCVD), molecular beam epitaxy (MBE) or vapor-liquid-solid (VLS) nanowires growth. In spite of tin oxides structures wide modern usage no systematic, comparative and complex studies of their morphology, local atomic and electronic structure have been performed by electron microscopy, high resolution synchrotron based X-ray Absorption Near Edge Structure (XANES), X-ray Photoelectron Spectroscopy (XPS) or PhotoEmission Electron Microscopy (PEEM) techniques in combination with first principles calculation and modelling. Such characteristics of the active surface and interfaces as the local atomic surrounding and electronic structure, physical and chemical stability are very important, especially for developing a strategy for functional application in modern engineering and technologies beyond optical transparency and gas sensing properties, such as bio-photonics.

The complex comparative study of Sn-SnO_x-SnO_2 structures deposited on planar silicon surfaces formed by MS, MOCVD and MBE together with VLS nanowires have been performed in combination with first principles calculations and linear-combination fitting modelling. Thicknesses of layers did not exceed few tens of nanometers (5 monolayers for MBE) and wires section was about few tens of nanometers. VLS nanowires found to be highly ordered crystals with the developed surface available for electronic structure engineering. For MS layers a wide range of phases in form of granular film have been shown indicating a flexibility in gap engineering. At the same time MOCVD approach demonstrated a possibility of stable low dimensional granular SnO_{2-x} core-shell film formation. Phase transformation for MBE structures included oxygen transport to silicon buffer under certain anneal. All approaches are considered as possibly prospective for real 3D developed surface functionalization.

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OP-02

Application of hard X-ray photoelectron spectroscopy in near total reflection regime for the study of nanoelectronic devices

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The performance of a wide range of modern nanoelectronic devices based on multilayered structures strongly depends on the properties of the underlying functional materials and corresponding interfaces. From this perspective, the utilization of techniques that combine sensitivity to chemical states and electric fields with high spatial resolution, especially in depth, is of great importance for the optimization of the device performance. Laboratory X-ray photoelectron spectroscopy (XPS) is a well-established method for this type of analysis, however small probe depth restricts its use for the bulk or buried-interface non-destructive analysis. To overcome this limitation, one can use hard (3-12 keV) X-rays to excite photoelectrons with much higher kinetic energies and consequently with inelastic mean free paths (IMFP), which eventually allows to probe layers up to ~20 nm deep. Yet, it comes at the cost of a dramatic reduction in the photoelectron emission cross-section, thus making the use of synchrotron radiation sources very much desirable. Even at higher incident photon energies, the photoemission signal occurring closer to the surface always prevails over the one from sub-layers below. These obstacles can be circumvented by adjusting the intensity of the incident X-ray electromagnetic field within the sample by selectively “exposing” regions of interest [1]. The latest generation of synchrotron radiation sources brings an opportunity to utilize different X-ray optic effects for this kind of structure highlighting including the generation of stable standing-waves [2, 3].

Here, we describe the use hard X-ray photoelectron spectroscopy (HAXPES) under near-total external reflection condition [4, 5] for experimental investigation of buried W/$Hf_{0.5}Zr_{0.5}O_2$ interface in a nanoscale ferroelectric capacitor being the functional part of a non-volatile memory device. Measurements were performed at the beamline P22 of PETRA III synchrotron (DESY, Germany) at the excitation x-ray energy of 6 keV, with Specs 225 HV analyzer used to register photoelectrons. The glancing incidence angles were varied in the range from 0.2 to 1.1 degrees (with an angle increment of 0.025 and 0.040 degree), with nearly normal photoelectron take-off geometry. Hf4f and Zr3p core-level lines were acquired as the main spectral regions of interest. NTR rocking curves (RC; core-level signal intensity vs. angle of incidence) were obtained based on accurate spectra fitting with Voigt function lineshape and Shirley background. Experimental NTR RC were matched with the sample structure-based photoelectron yield calculation [6] implemented on the top of TER_sl program [7]. The presence of ultrathin interfacial WO_x oxide was established from the analysis and the electronic band diagram was reconstructed at the W/HZO interface.

OP-02

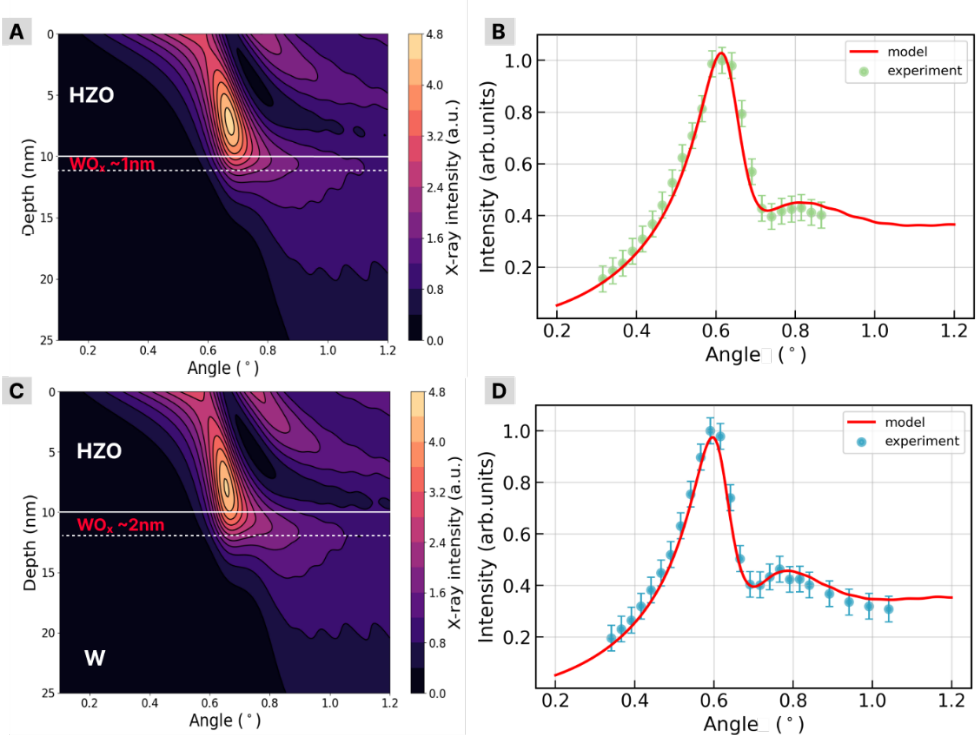

Fig. 1. (A), (C) – Corresponded to the best fit X-ray intensity distribution maps across a W/HZO structure for sample No. 1 and 2.; (B), (D) – Fitted Hf4f$_{7/2}$ angular intensity distribution (RC) for sample No. 1 and 2.

Acknowledgement: This work was financially supported by the Ministry of Science and Higher Education of the Russian Federation (agreement No. 075-15-2023-448).

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OP-03

Synchrotron study of metal-insulator phase transition in ordered manganites *R*BaMn$_2$O$_6$ (*R* = Sm, Nd, Pr) under high pressure

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Rare-earth (*R*) manganites demonstrate such functional properties as colossal magnetoresistance and magnetocaloric effect; strong mutual influence of spin, charge and phonon subsystems provide these effects. Ordered (double) manganites *R*BaMn$_2$O$_6$ are built as a sequence of *R*MnO$_3$ and BaMnO$_3$ cubic layers alternating in *c*-direction perpendicularly to the layers plane; ordering results in an increase of electric and magnetic transition temperatures and so makes double manganites even more attractive for numerous applications. Metal-insulator transition determines functional properties in studied materials, this transition is accompanied by structural phase transition with a jump of lattice volume. We have studied an influence of high pressure on this phenomenon for ordered manganites.

The measurements of physical properties (electrical resistivity, ac-susceptibility, ac-specific heat were measured in temperature range 100 – 300 K and pressure range 0 – 6 GPa in IHPP RAS. Crystal structure was studied by powder diffraction using synchrotron radiation in temperature range 300 – 523 K, pressure range 0 – 1.5 GPa at 4th beamline of the VEPP-3 storage ring using the resistively heated diamond anvils cell and wolfram gaskets, MAR345 IP detector, λ = 0.3685 Å, gold internal standard. Also diffraction experiments at low temperature 100 – 300 K, pressure range 0 – 5 GPa were performed at XPress beamline, Elettra synchrotron, λ = 0.49563(1) Å, membrane DAC (180×90 microns gasket hole), beam shaper 80 microns in diameter, PILATUS S 6M (DECTRIS) detector. Pressure and temperature values were controlled within 1 *K* and 0.2 GPa accuracy, respectively, by fluorescence of ruby and the internal lead standard. Obtained pressure influence on metal-insulator phase transition is shown in Figure 1.

Metal-insulator transition temperature decreases while the temperature of antiferromagnetic ordering T_N increases with pressure. This fact was supported by neutron diffraction for PrBaMn$_2$O$_6$ recently [1]. The pressure influence can be divided on two parts: at small pressure P < 2 GPa the reduction of lattice volume leads to suppression of lattice distortions in form of oxygen shifts and MnO$_6$ octahedra tilting; at higher pressure MnO$_6$ octahedra become compressed in *c*-direction and elongated in *ab*-plane even at high temperature for conducting state. As a result, the difference in crystal structure in high temperature conducting phase and low temperature insulating phase vanish and metal-insulator transition becomes smooth (Figure 2).

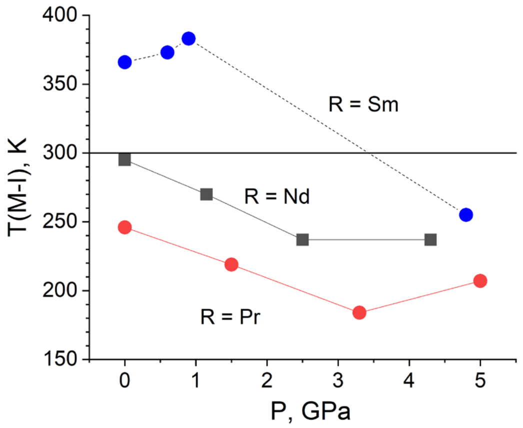
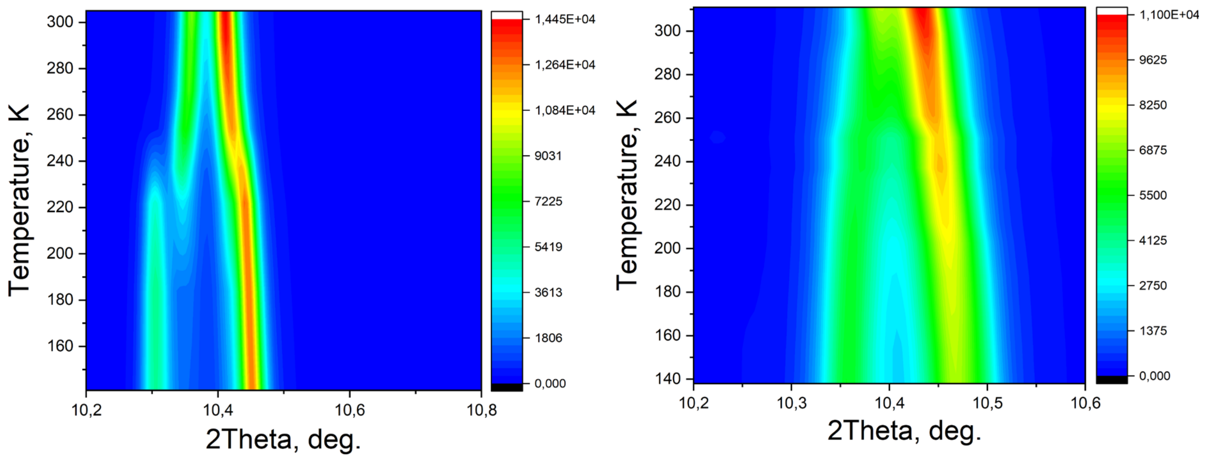


Fig. 1. Metal-insulator transition temperature as a function of the external pressure for $RBaMn_2O_6$, R = Sm, Nd, Pr



(**a**) (**b**)

Fig. 2. Diffraction lines (110) and (012) for $NdBaMn_2O_6$ at external pressure P = 2.4 GPa (a) and 4.4 GPa (b)

At small pressure $P < 2$ GPa lattice compression and suppression of lattice distortions leads to a strengthening of metallic ferromagnetic state and abrupt metal-insulator transition at cooling. Small fraction of the material remains ferromagnetic even at high pressure. Analysis of the irreducible representations at structural phase transition has been performed; it did not reveal participation of Jahn-Teller modes in metal-insulator transition.

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OP-04

Nuclear forward scattering in ultrafine ε-Fe_2O_3 nanoparticles

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The paper presents the results of nuclear forward scattering measurements at a synchrotron source on ultra-small ε-Fe_2O_3 nanoparticles ($<d>\approx 4$ nm) in the temperature range of 4.2-300 K within the external magnetic fields of 0-4 T. The fitting procedure of the experimental data allowed us to study the influence of the nanoparticle size on the features of the well-known metamagnetic transition in the range of 80-150 K [1,2]. According to our findings, we can believe that magnetic structure of the ultrafine ε-Fe_2O_3 nanoparticles do not suffer any changes in the whole temperature range.

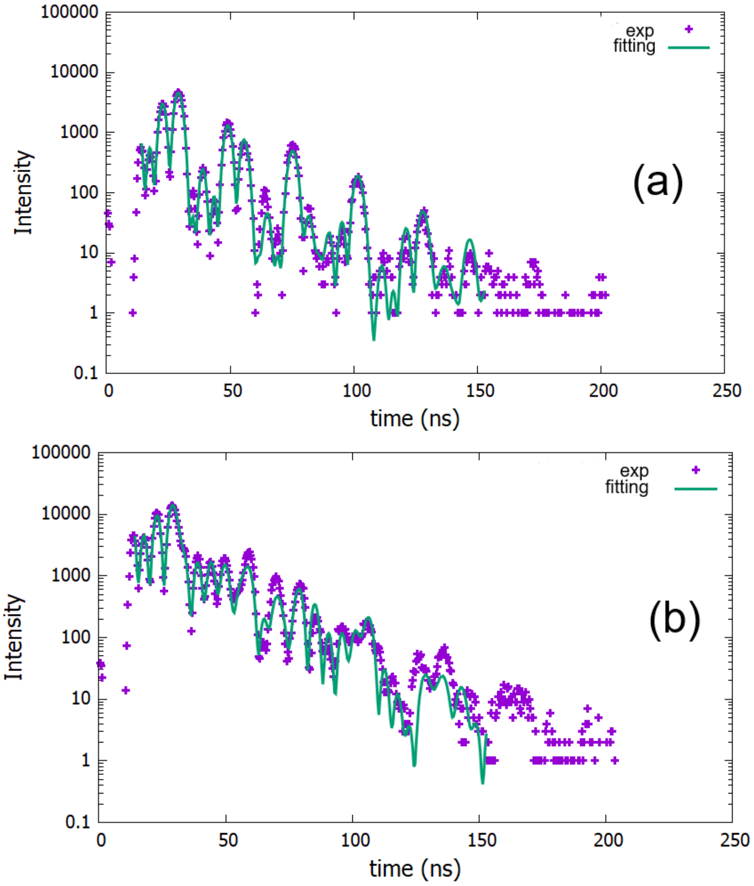

Fig. 1 Nuclear forward scattering spectra for the ultrafine ε-Fe_2O_3 nanoparticles at the temperature of 4.2K (a) without external magnetic field and (b) in a magnetic field of 4 T.

Acknowledgement: The work was supported by Russian Science Foundation, Grant No. №24-12-20011, https://rscf.ru/project/24-12-20011/), Krasnoyarsk Regional Fund of Science.

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OP-05

Development and characterization of a monocrystalline MAPbBr3 perovskite X-ray detector for ionizing radiation sensing

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Hybrid organic–inorganic lead halide perovskites are rapidly emerging as revolutionary materials in the fields of photovoltaics and radiation detection technologies. Their success is underpinned by several compelling material properties, including strong optical absorption, high mobility-lifetime products, tolerance to structural defects, and a versatile chemical composition that incorporates high atomic number (Z) elements, enhancing their interaction with ionizing radiation.

This study focuses on the development of a monocrystalline X-ray detector using methylammonium lead bromide (CH3NH3PbBr3 or MAPbBr3), grown through advanced solvent surface engineering and space-confined growth techniques[1]. These methods have been instrumental in producing high-quality, defect-tolerant monocrystalline perovskites that are ideal for high-resolution X-ray detection applications.

We have engineered a device by creating a Schottky barrier structure, utilizing ion beam sputtering to form thin layers of Nickel Oxide and Indium Tin Oxide on the MAPbBr3 substrate, followed by the addition of metal contact pads. This configuration leverages the unique properties of MAPbBr3, particularly its effective charge carrier transport and separation under X-ray illumination.

The completed device was encased and subjected to rigorous testing across a range of X-ray energies, specifically from 5 to 25 keV. Preliminary results reveal that our monocrystalline MAPbBr3 detector exhibits exceptional direct detection capabilities, characterized by high sensitivity and minimal noise, making it a promising candidate for future X-ray imaging technologies[2].

Our findings contribute to the broader understanding of perovskite-based detectors and open up new avenues for their application in medical imaging, security scanning, and scientific research, where the demand for highly efficient and accurate direct X-ray detection systems continues to grow.

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OP-06

Complementarity in synchrotron and DFT research methods for studies of the electronic structure and spin texture of materials

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Electronic structure is critical characteristic that determine the electrical, magnetic and optical properties of materials. With the capability of directly visualizing band dispersions and Fermi surfaces, angle-resolved photoemission spectroscopy (ARPES) has emerged as a powerful experimental tool to extract the electronic structures of materials and the coupling of these electronic structures to different degrees of freedom in crystal lattices [1]. In the past decades, advances in instrumentation and synchrotron light sources have significantly improved the accuracy and efficiency of ARPES experiments. These advances have enabled the application of ARPES in novel material systems to aid our understanding of their physical properties and behaviours. At the same time, the interpretation of experimental data, their deep analysis and the search for ways to modify materials to functionalize their properties requires the calling in theoretical calculation methods based on the density functional theory (DFT).

This talk will provide a brief overview of the successful combined application of experimental (ARPES) and theoretical (DFT) methods to study the bulk and low-dimensional electronic structure and spin texture of materials including semiconductors, topological insulators and magnetic metallic compounds based on the works we have been involved in resent years [2-14].

Acknowledgement: This work was supported by the Government research assignment for ISPMS SB RAS, project FWRW-2022-0001.

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OP-07

Study of phase transformations in structural and functional materials using the VEPP-3 synchrotron radiation source

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The development of new structural and functional materials involves with complex analysis of changes in the structural-phase state directly under operating conditions. The vast majority of these materials are subjected to extreme operating conditions such as aggressive environments (hydrogen, oxygen, carbon dioxide, etc.), cryo or high temperatures, low or high pressures, etc. Today, the data on structural and phase changes in materials in direct contact with aggressive environments at elevated temperatures and pressures are not only of fundamental, but also of practical interest, because on their basis it opens up the possibility of predicting and evaluating their physical and mechanical properties. Experimentally, such information can be obtained from in situ diffraction measurements using synchrotron radiation sources [1, 2].

This paper presents in situ studies of phase transformations of a wide class of structural and functional materials carried out by the staff of the Laboratory of Perspective Materials and Safety of Hydrogen Power Systems and the Experimental Physics Department of TPU over the last 10 years. In situ experiments were performed by synchrotron radiation at the "Precision Diffractometry II" station at Siberian Synchrotron and Terahertz Radiation Center of the Budker Institute of Nuclear Physics of the Siberian Branch of Russian Academy of Science. The features of the station, the main reaction chambers where the research was conducted, and the conditions of the time-resolved experiments are shown. The structural stability of laminated composites based on Nb/$Ti_3Al(Si)C_2$-TiC under linear heating in vacuum from room temperature to 1400°C followed by 60 min exposure has been demonstrated. It was found that hydrogen is able to desorb from the composite prior to the main stage of MgH2 decomposition in the temperature range (350-560) K in an argon atmosphere. It was also found that the hydrogen release peaks are asymmetric, indicating the presence of several hydrogen capture sites with close activation energies. The possibility of using TiC as protection for HfH1,66 systems, which are potential replacements for boron-based neutron absorbers, is demonstrated. The efficiency of Cr and Zr interdiffusion suppression using a Mo barrier sublayer in Cr/Mo/Zr systems at heating up to 1250 °C has been revealed.

Acknowledgement: This work was supported by the Governmental Program "Science", project No. FSWW-2024-0001.

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OP-08

XPCS study of relaxors aging

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Relaxors are one of the most interesting groups of disordered compounds. We have studied the structural relaxation in the $PbMg_{1/3}Nb_{2/3}O_3$ (PMN) relaxor. The microscopic mechanism of structural transformations in relaxors and the nature of the low-temperature state remain not fully understood despite almost 70 years of research. In the most interesting temperature range, near the freezing point, the characteristic relaxation times reach values from milliseconds to thousands of seconds, which are inaccessible to conventional methods for studying structural rearrangement processes. X-ray photon correlation spectroscopy (XPCS) seems to be the most suitable way to study slow relaxation in relaxors.

We have studied the structural relaxation in the PMN relaxor. The measurements were performed at the ID10 line of the European Synchrotron Radiation Facility (ISRF). Two-dimensional diffuse scattering patterns were measured in the region of the (110) Bragg reflection using an Andor CCD matrix. Based on the experimental data, the temperature evolution of both one-time and two-time correlation functions was followed. A series of two-dimensional scattering patterns were analyzed to extract the intensity-time correlation function, which is described by the exponentially decaying Kohlrausch-Williams-Watts (KWW) expression:

$g(2)(Q,t)=1+c\ \exp(-2t/\tau)^{\beta}$ with $\beta>1$

The obtained characteristic relaxation times β were found to be very large ($\beta \geq 700$ s). We failed to reveal a clear temperature dependence of the characteristic relaxation times, which can be explained by the existence of a pronounced aging manifested in the behavior of the two-time correlation functions.

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OP-09

Helicoidal states on atomically precise vicinal surfaces: ARPES and DFT study

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Conventional spin-generated surface electrons can be effectively manipulated using self-organizing nanomatrices. One-dimensional stepped superlattices on vicinal surfaces are of particular interest because they can be used to obtain strong surface anisotropy for electron transport devices. In this work, we present the realization of periodic resonator gratings on atomic-thick $BiAg_2$ surface alloy with atomic precision and demonstrate their potential ability to accommodate Rashba helical states with giant splitting. Using bent crystals to select local vicinal planes, two well-defined arrays of $BiAg_2$ monoatomic steps (423) and (11 7 9) with different spacing are fabricated.

Angle-resolved photoemission spectroscopy (ARPES) is used to study the Rashba helical states on such “magic” planes. Density functional theory (DFT) methods are used to interpret these experimental results. The main results of this work are shown in Figure 1

We observe coherent scattering of zones split by the Rashba spin-orbit interaction on arrays of steps, which leads to orbit-selective renormalization of the bands in the direction perpendicular to the steps and to a deep modulation of the planar orbital dichroism. The joint analysis of experimental and theoretical results allowed us to show that the interaction of Rashba electrons with the superlattice of the steps also leads to reorientation of the spin texture in both parallel and perpendicular directions. The calculations have shown that scattering on the potential of the step superlattice leads to a pronounced orbital-selective renormalization of the topology of the bands, with the effect of strong repulsive scattering on the steps observed mainly for p_Y-bands. In addition, the superlattice of the steps leads to a strong reconfiguration of the spin texture, taking the spin out of its original in-plane helical configuration. Overall, our results demonstrate the potential of certain step superlattices to create spiral bands not only in terms of energy dispersion but also in terms of their orbital composition texture. These results open the way to atomically precise coupled electron resonator arrays for the development of spin-orbit textures.

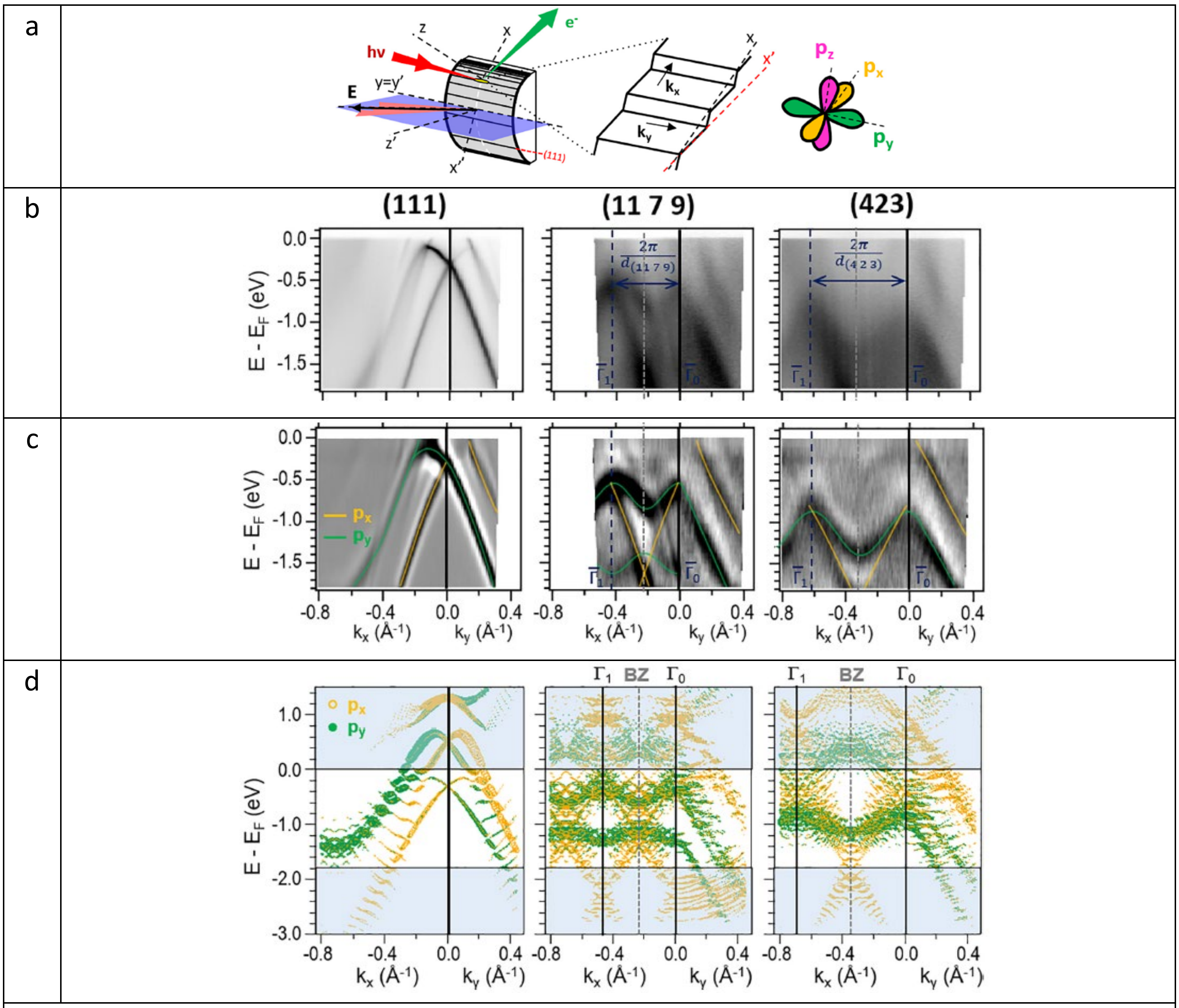

Fig. 1. Electronic structure of Rashba helical states on the BiAg2/c-Ag(111)$_{fk}$ curved surface. (a) Geometry of the ARPES experiment to determine the E(k_x) band dispersion in the direction perpendicular to the steps. The light incidence angle is 45°. For E(k_y), the sample is rotated 90° around the z axis. The x' axis is directed along the terrace perpendicular to the steps, and the z' axis is perpendicular to the terrace. (b) ARPES intensity measured near the (111), (11 7 9), and (423) planes in the direction perpendicular (x) and parallel (y) to steps. For the (111) surface, these correspond to ΓM and ΓK, respectively. The Brillouin zone edges, where bands are folded, are marked by vertical lines. (c) Second derivative of the photoemission intensity shown in (b). Green/yellow guidelines emulate the ab initio p_y/p_x bands of (d). (d) DFT-calculated p_y (green) and p_x (orange) projected electronic states. The dot size is proportional to the magnitude of the orbital projection. For clarity, data falling under 20% of the total intensity have been cut off. The energy range out of the [—1.8,0] eV explored in ARPES appears shaded.

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OP-10

Application of synchrotron radiation for *In Situ* XRD investigation of hydrogen desorption from magnesium hydride and nanosized catalysts composites

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Magnesium hydride is considered one of the most promising hydrogen storage materials, although it nevertheless has some problems, such as the high value of the activation energy of hydrogen desorption. To solve this problem, some scientists have proposed adding nanomaterials, in particular metal-organic frameworks, carbon nanotubes, nanosized metal (Ni, Al, Cr) particles to magnesium hydride. Currently, a detailed understanding of the mechanisms of obtaining composites based on magnesium hydride and nanomaterials is lacking, as is our understanding of the effect of additives on the activation energy and temperature of hydrogen desorption depending on the parameters of the composite synthesis. In addition, the data obtained at various values of milling parameters are very different, and in some works the effect of nanomaterials on the hydrogen properties of magnesium hydride was not confirmed at all. Thus, it is important to determine the effect of nanomaterials additives on the properties of hydrogen storage of magnesium hydride under various milling parameters. This work is devoted to the application of synchrotron radiation for *In Situ* XRD investigation of hydrogen desorption from composites based on hydrides and nanomaterials. *In situ* X-ray diffraction measurements of the process of thermo-stimulated decay of magnesium hydrides were carried out at the station "Precision diffractometry» at Siberian Synchrotron and Terahertz Radiation Center of the Budker Institute of Nuclear Physics. Measurements were performed using SR from the VEPP-3 storage ring.

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Registration of the dust flow under shock loading of a plate with a given geometry of the free surface

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When a strong shock wave interacts with metals, a stream of microparticles is released from the free surface. This phenomenon is called "dusting" of metals [1-3]. The size and number of particles, and as a result their velocity, depend on the shape of the loading pulse, surface cleanliness and structure of the material. In modern devices, this effect negatively affects the parameters of the mechanisms. In this paper, we consider the case when grooves (furrows) are applied to the free surface of the surface under study, which form a flow of microparticles under shock wave loading.

Studies of the release of microparticles were carried out by pulsed radiography at the VEPP-3 and VEPP-4 synchrotron radiation source at the INP SB RAS. The X-ray shadow was recorded by a DIMEX detector [4-6].

A shock wave (UV) with an intensity of ~ 40 GPa in copper and tin samples was created by a charge of pressed rdx.

The report presents experimental results on the registration of linear density (from 5 mg/cm2) and flow velocity (1-3 km/s) of microparticles from the studied plates.

The effectiveness of the technique used to study the fluxes of microparticles with low density is shown. The results of the work can be used to design shock wave loading devices.

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OP-12

Effect of Zr content on the structure and properties of $Ce_{1-x}Zr_xO_2$ mixed oxides and related $Ru/Ce_{1-x}Zr_xO_2$ catalysts for CO_2 methanation

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Ruthenium-based catalysts are recognized as highly active and selective catalysts for CO_2 methanation under mild temperature conditions [1-3]. Due to the structural sensitivity of the CO_2 methanation reaction, the structural aspects of supported ruthenium species and support particles have a great impact on the activity, selectivity and stability in the reaction. The use of easily reducible support materials, such as ceria and ceria-based oxides, not only allows to stabilize highly dispersed Ru particles [4], but also improves the catalytic activity. Such supports are believed to be involved in the catalytic reaction. It has been suggested that oxygen-deficient sites, generated on a support surface at metal/support interface under reaction conditions, act as the additional centres for CO_2 adsorption [5]. A possible way to improve the redox properties and mechanical strength of the ceria is changing its cation composition by doping it with zirconium. For example, Iglesias et. al. [6] and Ocampo et al. [7] varied the Ce/Zr ratio in a nickel-based catalysts for CO_2 methanation to obtain the optimum zirconium content. They revealed that support doping with zirconium enhances oxygen mobility in support lattice, increases nickel dispersion, resulting in a higher activity in CO_2 methanation. X. Shang et.al. [8] evaluated the effect of Ce/Zr molar ratio on catalytic performance of the Ru–Ni–Ce–Zr system. It was shown that zirconium concentration in the support has a small influence on the catalyst activity. However, there are no data on a pure Ru-Ce-Zr system without nickel, that could shed light on the influence of the zirconium dopant concentration in a $Ru/Ce_{1-x}Zr_xO_2$ system on the structural features of the catalyst and its reaction activity. To explore this issue, it is necessary to analyze the structural characteristics of in detail. It is necessary to use methods with a high sensitivity to local structural features.

This report presents the results of the structural diagnostics of supported Ru catalysts based on CeO_2 and $Ce_{1-x}Zr_xO_2$ support materials with different compositions (x=0, 0.1, 0.25, 0.5). A series of the $Ce_{1-x}Zr_xO_2$ oxides were synthesized by a reverse coprecipitation technique. The catalysts were prepared by a sorption-hydrolytic deposition technique. Attention is paid to the study of the features of $Ce_{1-x}Zr_xO_2$ crystal structure, including those at the local atomic structure level, as well as state and structural characteristics of supported ruthenium species. A wide range of methods was used, including X-ray powder diffraction, CO chemisorption, Raman spectroscopy, electron microscopy. The X-ray pair distribution function (PDF) analysis was used to study the structure of highly dispersed Ru species at the atomic level of structure and to conduct local structural analysis.

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OP-13

Structure of Ag+ complexes with amino groups in an oligooxypropylenediamine solution by EXAFS and XANES

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Nanocomposites based on silver ions in epoxyamine matrices are of interest due to the prospects for use in micro- and optoelectronics, as well as their pronounced biocidal properties. The initial stage of the formation of such composites is the interaction of a precursor (silver (I) salt, a source of Ag+ ions) with the main components of epoxyamine matrices – an epoxy oligomer and an amine hardener. In this work, the local atomic geometry of Ag+ ions complexes with amino groups of a liquid amine hardener – oligooxypropylenediamine (Mn=230) is determined. In the report will be presented the main methods of analyzing experimental data to obtain information about the local atomic geometry of organometallic complexes: the classical method of sphere-by-sphere fitting of EXAFS spectra [1], the evolutionary approach implemented in the EvAX program [2] and the fitting of XANES spectra (calculated by the FEFF10 program [3]) by varying structural parameters. Using Ag+ ions complexes with amines as the example of the structural problem, the possibilities and limitations of each method will be presented.

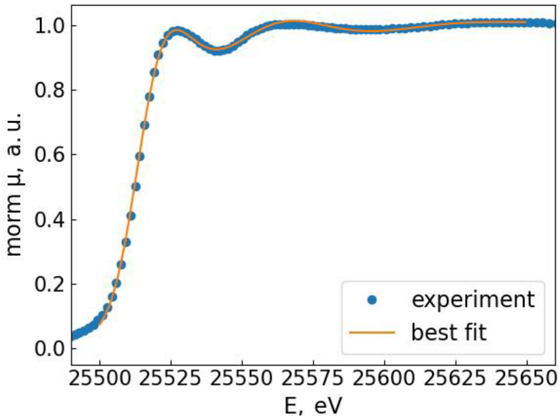

Figure 1. Experimental and optimized XANES spectra.

As a result of a consistent fitting of the extended and near edge fine structure of the X-ray spectrum, it was possible to determine that Ag+ ions are coordinated by two amino groups, the spatial arrangement of which was determined by X-ray absorption spectroscopy. The experimental and calculated XANES spectra are shown on the Fig. 1.

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OP-14

Concept and key experimental methods of the URAL beamline in the soft X-ray range for materials science research in resonance and operando mode

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The project of the universal experimental beamline "URAL" at SKIF synchrotron facility is presented. The station is intended for the study of the electronic and atomic structure, local electronic characteristics of functional materials in the soft X-ray range of 50 - 2000 eV. While subjecting samples to a variety of optical and electrical stimuli, information about charge accumulation on materials or surface structures can be obtained. These included stimuli d.c. as well as a.c. electrical and/or optical pulses covering a wide frequency range for probing charging and/or photovoltage shifts, stemming from impurities, dopants, defects, etc. It is proposed to use a bending magnet of the storage ring as a radiation source. The description of the station parameters and the main methods to be implemented on it is given. It is proposed to develop and subsequently implement a new promising method for studying local conductivity of semiconductor structures and thin films using X-ray photoemission spectroscopy with laser on/off (XPS+laser, Operando XPS) at synchrotrons in the Russian Federation. Laser-induced photoconductivity ensures fast relaxation of excited states in semiconductor structures, which is manifested in a shift in the binding energy of internal levels. This method has enormous potential and is currently used only in a few foreign laboratories where semiconductor structures and materials for solar batteries are synthesized and studied. The range of scientific, technical and applied problems to be solved by the community of station users is outlined. The functional experimental capabilities of the planned station and the methods to be implemented are shown. The requirements for the beam transport channel, monochromator, detectors, and measuring chamber are described. The technical parameters of the station are given, the experimental methods being implemented are described, and possible tasks for science and industry that can be implemented at this station are outlined.

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OP-15

PDF analysis of alumina-based catalytic materials

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Direct structural diffraction methods provide the most reliable information about the structure of materials. Conventional X-ray diffraction techniques are based on the analysis of the position and intensity of diffraction peaks, providing information about average atomic and real structures. However, for catalytic materials with dispersed and amorphous supports and active components of catalysts, the application of these methods may be challenging in terms of studying their structure. These systems require the use of special diffraction methods based on a full profile analysis of diffraction patterns, including diffuse scattering. The PDF (pair distribution function) analysis allows us to establish the local structure, phase composition, particle size when it is less than 3 nm, and possible interactions between the components of a multicomponent system. The use of a powerful radiation source, such as synchrotron radiation, enhances the accuracy of diffraction experiments and significantly expands the capabilities of this method.

The present work investigates aluminas, which are commonly employed in catalysis as supports, and the deposited catalysts based on these materials with rhodium and platinum as active components. PDF analysis were used for this study. These deposited catalysts and their components often consist of nanoscale substances, including X-ray amorphous ones, and their composition and physico-chemical characteristics depend on synthesis conditions. Therefore, we consider the characteristics of aluminas obtained from different precursors — hydroxides. When depositing the active component, its precursors varied, and the order of their deposition was adjusted in the case of bimetallic catalysts.

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OP-16

Analysis of EXAFS spectra containing superposition of L3-edge signals of Au and Pt

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One of the methods for studying the local atomic structure is X-ray absorption spectroscopy. By now, EXAFS spectroscopy has a large theoretical basis. However, the following problem may still arise when studying complex systems. When analyzing X-ray spectra of materials containing chemical elements with close atomic numbers, an overlap of the spectra of several chemical elements is observed. In this case, the EXAFS spectrum contains a superposition of different wave numbers, which makes it impossible to analyze such a spectrum using the classical Fourier transform method. Thus, there is a problem of developing a method for analyzing EXAFS spectra containing an overlap of signals from several chemical elements.

A similar problem occurs not only in EXAFS spectroscopy. Previously, an algorithm for analyzing electron energy loss spectra (EXELFS) was developed. The developed method made it possible to determine the parameters of the local atomic structure from the spectra of the Cu-Mn system.

In this paper, the possibility of analyzing EXAFS spectra containing a superposition of two extended fine structures in one energy range is considered. The test object was a solid solution of AuPt. The EXAFS spectrum of this system contains a superposition of the Au L3-edge signal on the Pt L3-edge. A series of numerical experiments were performed to isolate the normalized oscillating parts of Au and Pt from the overlapping EXAFS spectra of the AuPt system in order to determine the parameters of the local atomic structure.

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OP-17

SAXS study of the Pt precursor transformation during the preparation of the supported catalysts

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Supported metal catalysts are widely used in modern industrial processes. Traditionally, in their preparation, solutions of precursors of active particles, which can be both true and colloidal solutions, are used as the first component. Various porous supports are used as the second component. Upon contact between the precursor solution and the solid support, interaction between the precursor particles of the active component and the surface groups of the support takes place with their subsequent deposition on the support surface. Unfortunately, all the processes occurring at the contact of liquid and solid phases during the catalysts preparation often remain behind the scenes, since the main emphasis in the study of supported catalysts is placed on the already prepared metal nanoparticles before, after, or during the catalytic reaction. In such a paradigm for the study of supported catalysts, questions remain as to what still happens in solution when the precursor solution interacts with the support surface. Why in some cases in the catalysts preparation small metal nanoparticles are obtained, and in other cases large ones?

This work reports on the study of the aggregation and transform processes of platinum precursor when it is deposited on a different oxide supports by using SAXS. This method allows analyzing both the initial colloidal solution of the precursor and the processes occurring during the contact of the precursor particles with the porous surface of the supports. The role of tetramethylammonium hydroxide in stabilization of platinum nitrate solution as a precursor has been studied. It is shown that depending on the acid-base properties of the supports (SiO_2, γ-Al_2O_3 and MgO) and the presence of stabilizer in the Pt precursor solution, primary Pt-containing particles of different shapes (loose clew-like particles and compact particles) and sizes (from 2 to 25 nm) can be obtained on the porous oxide supports. Depending on their size and shape, both highly dispersed and coarsely dispersed secondary Pt metal nanoparticles can be formed on the supports during further calcination.

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OP-18

Structural versus functional properties of Y and La doped $Hf_{0.5}Zr_{0.5}O_2$ thin films: current status of understanding

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Doping of $Hf_{0.5}Zr_{0.5}O_2$ (HZO) films with rare-earth metals during atomic layer deposition growth may significantly affect their structural and subsequently functional properties. Here, we present the structural properties of Y and La doped HZO films research as revealed by synchrotron based Near-Edge X-ray Absorption Fine Structure (NEXAFS) technique, and complemented by XRD and TEM techniques for Y:HZO. In contrast to XRD and TEM, NEXAFS analysis taken near O K (1*s*)-edge can unambiguously distinguish between orthorhombic (o-) and tetragonal (t-) phases (Fig. 1) and, moreover, provides quantitative information about the phase composition. In addition, from the absorption spectra near Zr M_3 ($3p_{3/2}$)-edge (Fig. 2) also provides complete and reliable information about the presence of the monoclinic (m-) phase, even in the case of highly textured samples. The structural data are further correlated with the electrophysical properties of Y: and La:HZO when sandwiched in capacitor structures. It is found that the evolution of electrophysical properties of doped HZO samples is accompanied by the change of the o/t ratio as revealed by NEXAFS. At the same time, o/t ratio is not the only structural parameter defining their electrophysical properties of La/Y doped HZO samples, at least in the pristine state, and the amount of m- phase also plays a role. The unambiguous prediction of the electrical response of doped HZO still remains challenging despite the breakthrough in the quantitative assessment of structural composition and may require the application of *operando* NEXAFS technique which would allow to monitor possible structural evolution in such thin-film materials following the electrical stress.

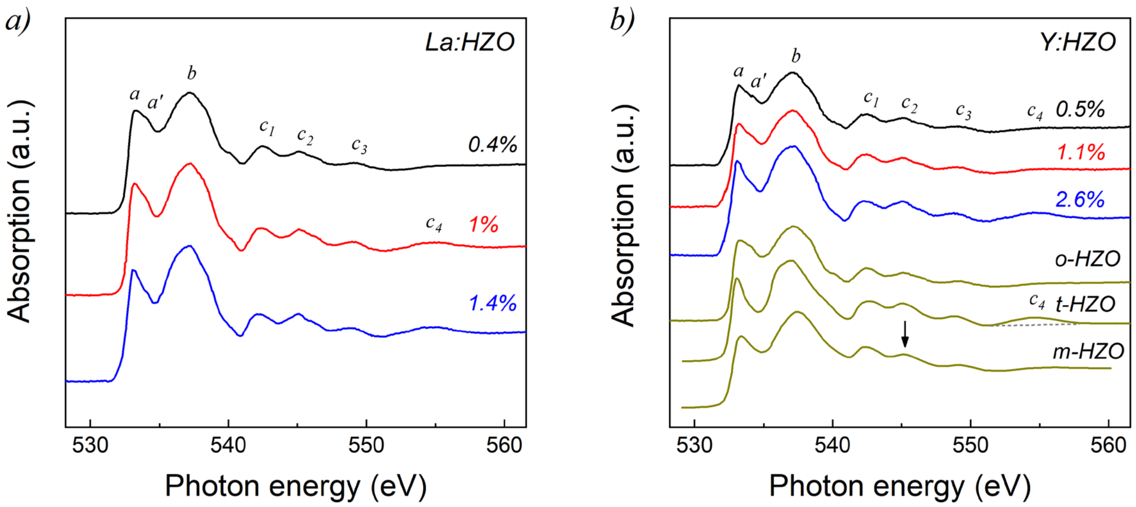

Fig. 1. O K (1s) X-ray absorption spectra of La-doped (a) and Y-doped (b) HZO films, dopant concentration is shown in mol. % (in terms of La_2O_3 and Y_2O_3). All spectra are shifted along the ordinate axis for clarity. The reference spectra of orthorhombic (o-HZO) (this work), tetragonal (t-HZO) from ref. [1] and monoclinic (m-HZO) from ref. [1] films are shown in the panel (b).

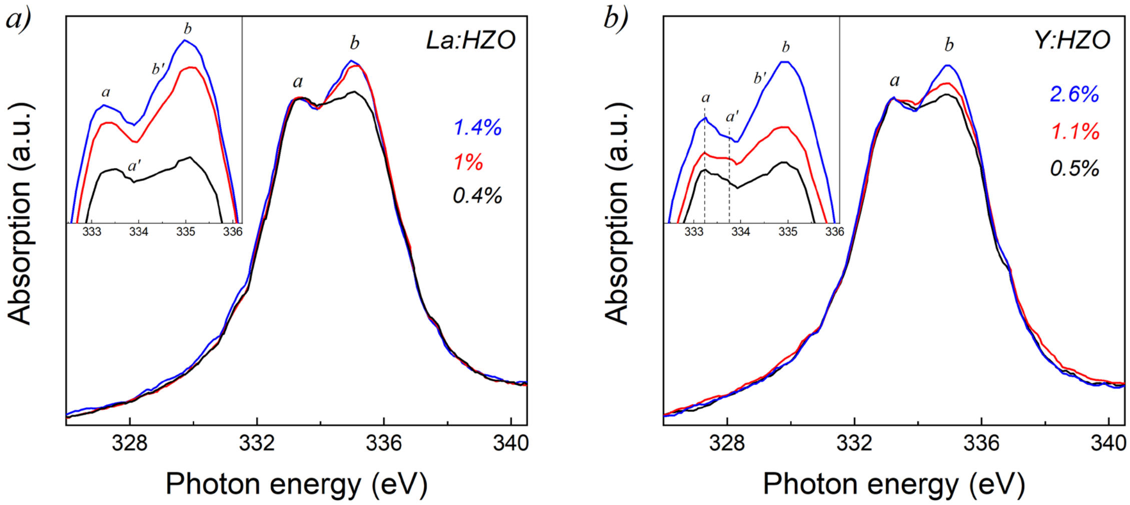

Fig. 2. Zr M_3 ($3p_{3/2}$) X-ray absorption spectra of La-doped (a) and Y-doped (b) HZO films, dopant concentration is shown in mol. % (in terms of La_2O_3 and Y_2O_3). The spectra are normalized to intensity of the first peak a for visibility. The insets show zoomed fine structure of peaks a and b, the spectra in the insets are shifted along the ordinate axis for clarity.

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OP-19

Method for quantitative XRD phase analysis of mixtures of low-temperature alumina forms

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Nanocrystalline aluminum oxides are important materials that are widely used in various technological applications. Due to their high dispersion, porosity, developed specific surface area and acid-base properties of the surface, low-temperature aluminum oxides are particularly attractive for use in catalysis as both catalysts and supports in metal and oxide catalysts.

There are several forms of low-temperature aluminum oxides (γ-Al_2O_3, χ-Al_2O_3, η-Al_2O_3) obtained from various hydroxide precursors. Despite having the same chemical composition, various low-temperature Al_2O_3 forms differ greatly in their physicochemical properties. For this reason, when developing technologies for the synthesis of alumina materials with desired characteristics, it is necessary to determine the quantitative composition of the obtained products. The main tool for distinguishing different Al_2O_3 forms is the powder X-ray diffraction (XRD) method. However, the problem of quantitative X-ray phase analysis of mixtures of different low temperature Al_2O_3 forms remains unresolved. Their powder XRD patterns are characterized by strong peak broadening, the presence of pronounced diffuse scattering effects and anisotropic broadening of a number of reflexes [2]. All this excludes the use of standard methods of quantitative XRD phase analysis intended for the diagnosis of wellcrystallized crystalline phases.

The aim of this work is to develop a method for quantitative XRD phase analysis of mixtures of the low-temperature Al_2O_3 forms. The proposed method is based on the representation of the diffraction pattern of a mixture of *N* phases by a linear combination of diffraction patterns of individual phases I_i with coefficients k_i:

$$I_{mix}(s) = \sum_{i=1}^{N} k_i I_i(s)$$

Preliminary procedures of normalization of the diffraction patterns with intensity conversion into electronic units, corrections for instrumental factors and absorption allow one to compare XRD patterns collected on different diffractometers and under different conditions. Thus, the task of quantitative analysis is reduced to finding the coefficients k_i in the linear combination using the least squares method.

Based on the proposed algorithm, a program was written in the Python programming language. This program was tested on mixtures of γ-Al_2O_3 and χ-Al_2O_3 oxides of known composition. When these mixtures were analyzed, an absolute determination errors were found to be within 5 wt. %. Fig. 1a shows an example of describing a sample with a χ-Al_2O_3 content of 50 wt. % by the proposed method. The determined content of χ -Al_2O_3 in this case was 45 wt.%. It was shown that the noise in the XRD pattern leads to an error of 1 wt.%, which is slightly smaller than the detected error. This is due to systematic errors that appear during data processing, for example, during the background subtraction procedure.

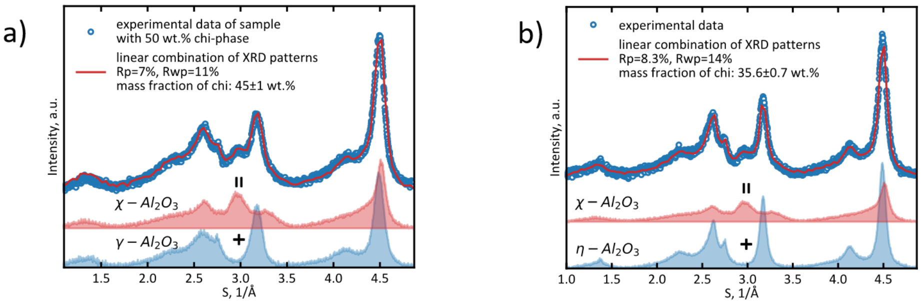

Fig. 1 – An example of a description of powder XRD patterns of mixtures of (a) χ-Al_2O_3 and γ-Al_2O_3, and (b) χ-Al_2O_3 and η-Al_2O_3 using the proposed method.

For comparison, the phase analysis was also carried out using widely accepted method of calibration plots. This approach is based on the use of dependencies of the ratio of the intensities of peaks characteristic of χ-Al_2O_3 and γ-Al_2O_3 oxides on their content, which were preliminary plotted using prepared mixtures of known different compositions. In the case of the method of calibration plots, the absolute error reached 10 wt.%, especially at low content of χ-Al_2O_3 form. The main reason is the difficulty of accurate detection and integral analysis of the reflection characteristic of χ-Al_2O_3 form due to its low intensity and overlapping with other intense peaks from γ-Al_2O_3 oxide. Thus, it was shown that the proposed method has better accuracy compared to the method of calibration plots. Moreover, its implementation does not require recording XRD patterns of a series of specially prepared mixtures of known composition, but only the diffraction profiles of individual reference phases.

The proposed method was also applied to mixtures of η-Al_2O_3 and χ-Al_2O_3 oxides of unknown composition. As can be seen from Fig. 1b, the proposed method allows for a satisfactory description of the diffraction pattern of the mixture. The residual factors are also close to those obtained when studying γ-Al_2O_3 and χ-Al_2O_3 mixtures of known composition (Figs. 1a and 1b). This indicates the reliability of the data obtained.

The aim of further research will be to minimize systematic errors associated with the background subtraction procedure. The methodology will also be improved to enable quantitative analysis for mixtures of different chemical compositions, such as mixtures of aluminum oxides and hydroxides.

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OP-20

A comparative study of the effect of high-pressure and low temperature on the crystal structure of the salts of purine nucleobases

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Crystals containing nucleotides can serve as models of the effect of pressure on nucleic acids (RNA and DNA) and oligonucleotides, as well as nucleotide-containing biological assemblies, similar to how the crystals of amino acids are used to model peptides and proteins. Up to now, however, they are studied significantly less than those of amino acids. Metal cations and water molecules were shown to play an important role in the formation of nucleotide assemblies in solutions and their response to pressure. In present contribution a number of salts were studied, namely sodium guaninate heptahydrate, potassium guaninate monohydrate, lithium xanthinate monohydrate, adeninium- 2,6-dihydrobenzoate monohydrate and adeninium- 3,5-dihydrobenzoate monohydrate [1- 4].

All compounds revealed a high stability of crystal structures to compression, phase transitions of different types at increasing pressure, preservation of crystal structure at cooling to 100 K [1-3]. Potassium guaninate monohydrate exhibits a reversible phase transition to an incommensurate phase at increasing pressure [3, 4]. The crystal structure of the incommensurate modulated phase of has been solved on the base of high-pressure single crystal x-ray diffraction data. This is a first example of high-pressure incommensurate crystal structure solved for the organic compound. In contrast to known structures of incommensurate modulated organic compounds, modulation functions of all atoms are discontinuous. This is a first example of experimental detection of incommensurate modulated crystal structure that can be modelled using the special modulation function "soliton mode" proposed in [5].

Adeninium- 3,5-dihydrobenzoate monohydrate exhibits a phase transition with reversible amorphization, whereas the structure of adeninium- 2,6-dihydrobenzoate monohydrate preserves at increasing pressure.

The ability of a single crystal of lithium xanthinate hydrate to withstand the volume decrease to -16.6% at 3.9 GPa without a phase transition, is, to the best of our knowledge, up to now, the largest value achieved for crystals of nucleotides, DNA and RNA fragments and complexes [1, 3, 6, 7]. Also, the ability of the crystal to withstand a reversible structural rearrangement without any crystal fragmentation or damage at higher pressures is quite remarkable. For a comparison, a pressure-induced phase transition in a sodium guaninate hydrate resulted in crystal fragmentation to powder [1], and also DNA crystals collapsed at much lower pressure values – less than 1.6 GPa [6, 7]. ThetaToTensor software was used to calculate compressibility tensor and to represent graphically its characteristic surface [8].

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OP-21

The structures of L- and D-phenylglycine: topological analysis for understanding the mechanical properties

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Amino acids are of special interest for research since they serve as "building blocks" for peptides and proteins, and play a crucial role in numerous processes within living organisms. The properties of amino acids are generally determined by the intermolecular interactions that their functional groups can participate. A comparative analysis of the structures of amino acids with similar side chains can help to identify general patterns in the formation of intermolecular interactions in crystals.

It has already been established that pure enantiomers of hydrophobic amino acids, such as valine, leucine, and isoleucine, crystallize in $P2_1$ space group, $Z' = 2$. Two molecules in asymmetric unit help to optimize the energy of hydrogen bonds. This motif is known as L2-L2 [1]. However, for amino acids with larger aromatic substituents, the situation is different. Phenylalanine can form four different polymorphic forms under ambient conditions [2]. These polymorphs have unusual L1-L1 patterns of hydrogen bonding. Additionally, π-π interactions have large contribution in crystal lattice energy of all phenylalanine polymorphs. This assumption can be made based on the conformation of side radicals, as neighboring phenyl rings are aligned "knobs-into-holes".

Crystal structure topology of phenylglycine is an intermediate between the structure topologies of leucine/isoleucine and phenylalanine. Previous studies suggested its structure belongs to $P2_1$ space group, $Z' = 2$ and an contains L2-L2 hydrogen bond motif [3]. The phenyl groups have been described as disordered. In our study, we redetermined and reanalyzed the structures of L-phenylglycine and D-phenylglycine and found that the aromatic rings have no disorder. Each molecule in the independent part of the structure was characterized by two conformations that optimize π-π interaction between phenyl groups, similar to those in phenylalanine. Taking into account the aromatic interactions, structure was described in $P1$ space group, $Z' = 8$. Thus, an inaccuracy was found in the previous structural data. The possibility of describing the structure as commensurate modulated has been shown. We also performed a topological analysis of the structure, analyzing the relationship between slip planes, hydrogen bonding motifs, and mechanical properties. It follows from the topological analysis that a large number of formula units in an asymmetric unit is probably caused not only by the optimization of aromatic interactions, but also by very weak hydrophobic interactions.

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OP-22

Real-time combination of synchrotron X-Ray scattering and ultrafast chip calorimetry to study thermal properties of polymers

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Combining synchrotron X-ray diffraction (XRD) with ultra-fast chip calorimetry provides a comprehensive understanding of the structural and thermal behaviors of materials during rapid phase transitions. Synchrotron XRD offers high-resolution insights into the atomic and crystalline structures, allowing scientists to track subtle changes in real time. When paired with ultra-fast chip calorimetry, which measures heat flow during rapid thermal events, this technique can elucidate how materials respond to extreme temperature changes on a very short timescale.

This combination is particularly valuable for studying polymers, metals, and other materials undergoing fast crystallization or melting processes. The structural information from XRD complements the thermal data from chip calorimetry, allowing researchers to correlate specific structural changes with thermal events. This dual approach enables a deeper understanding of material behavior under non-equilibrium conditions, which is crucial for optimizing their properties in applications such as high-performance materials and advanced manufacturing processes.

The methodological and scientific developments driven by this research will be beneficial for a broad scientific community working in the field of functional nanomaterials and polymer nanocomposites. We believe that this work will enhance the interest of a growing user community for in-situ combinations of novel emerging characterization techniques, thus contributing to the possible development of a future lab-on-a-chip platform.

Acknowledgement: The authors acknowledge the Russian Science Foundation (grant 23-73-30005).

OP-23

Composite for synchrotron radiation visualization based on a diamond with embedded YAG:Ce particles

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Introduction

Composite X-ray luminescent films based on diamond with embedded particles are promising materials for detecting and visualizing the high-intensity X-ray beams for using in synchrotrons and free-electron lasers. Such composites consist of luminescent rare-earth (RE) particles embedded into an X-ray transparent diamond matrix by overgrowing them with CVD-grown layer of diamond [1, 2].

The single crystalline diamond is most suitable materials for composite material preparing, since it has significantly higher transmission in the visible range of the spectrum than polycrystalline diamond. Impurity active colour centres in diamond are poorly excited by X-ray and synchrotron radiation. Significantly better excitation occurs through cerium-doped yttrium-aluminum garnet nanoparticles. The goal of study was to develop and test a composite visualizer based on single-crystal diamond with YAG: Ce nanoparticles.

Experiments and results

$Y_3Al_5O_{12}$: Ce (YAG:Ce) particles were synthesized at room temperature by dropwise addition of a dilute aqueous chlorides salt solution into dilute precipitate of ammonia water with ammonium sulfate as a dispersing agent. The synthesized precursor powder was dried at 60 °C in an oven and then annealed at in a high-temperature furnace Nabertherm 40/17(Germany) at 1600 °C in air. After annealing, the ceramic powders were ground in a mortar.

The composite diamond films were done in two steps. At the first stage, a suspension of YAG: Ce particles in DMSO was deposited onto a single-crystal diamond, followed by drying at a temperature of 360 °C. At the second stage, the deposited particles were grown into diamond using the CVD process in microwave plasma reactor ARDIS-100. The thickness of the built-up layer was about 15 microns. The photo of the composite film surface presented on Fig.1.

Successful visualization of the beam was realized by exposing the obtained composite film to synchrotron radiation (Fig.2). The experiments were carried out on the “Microscopy and Tomography” beamline on the VEPP-3 storage ring (Novosibirsk, Russia). For visualization, a polychromatic beam from a 2 T shifter was used. The composite diamond film was installed at a distance of 13 m from the irradiation source. The beam was collimated by a rectangular slit 2x2 mm2 with peak photon energy (8.869 keV) and beam power 0.2 W.

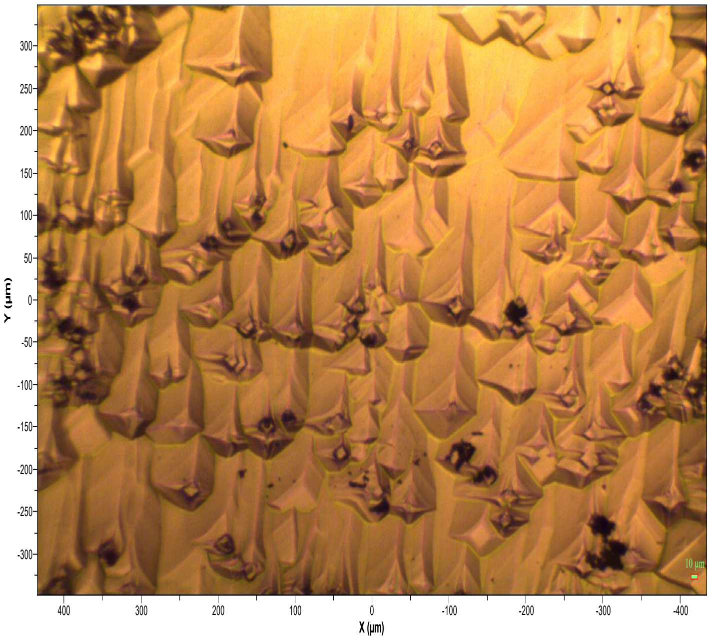

Fig. 1. Microphoto of composite diamond surface.

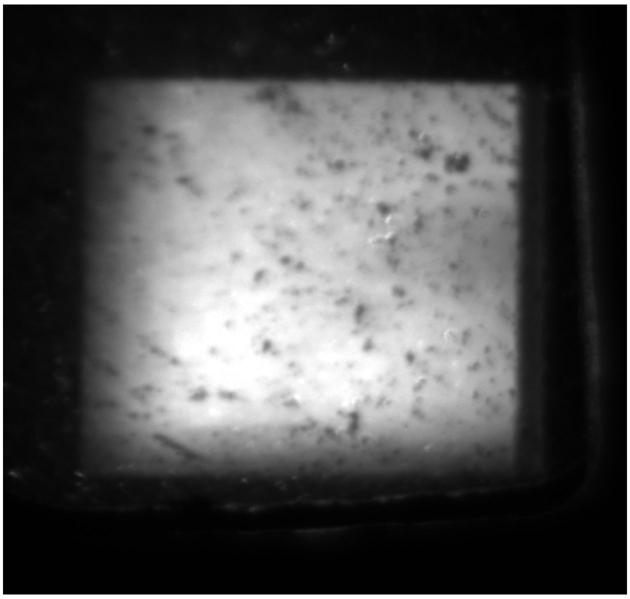

Fig. 2. Photo of the beam visualization by diamond-particle composite.

Acknowledgement: This work was supported by Russian Science Foundation, project 22- 13-00401, https://rscf.ru/en/project/22-13-00401/.

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OP-24

Synchrotron X-ray sources for the study of hybrid functional materials

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Perovskite solar cells are a class of thin-film solar cells first described in 2009, which demonstrated explosive growth in efficiency and stability. If in 2009 for the first laboratory prototypes they were only 3.9% and 15 minutes, respectively, then in 2024 they reached values of more than 27% and 6000 hours, competitive in comparison with existing PV technologies. The unique physico-chemical properties of hybrid perovskites, such as high mobility of charge carriers, record extinction coefficients, high tolerance to defects and impurities, as well as the smallest of all thin-film PV device thickness of less than 2 microns, coupled with the ability to create them using inexpensive solution methods from available reagents, make them an extremely attractive object of research.

The report will provide an overview of laboratory methods for the production of thin films of hybrid perovskites, the results of the study of intermediate phases formed during the crystallization of hybrid halogenplumbates from various solvents, as well as highlight the still unresolved scientific problems in this field, which require the use of synchrotron research methods.

OP-25

Development of combined methods for analyzing the structure and properties of “smart” functional materials

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Polymeric and low molecular weight materials capable of changing their properties under external influences in a controlled pattern are called “smart” materials. Such materials have attracted great interest in recent years because they combine the ease of obtaining and a wide range of parameters characteristic of synthetic materials with the adaptive behavior of living systems. Changes in physical and chemical properties due to heating, UV irradiation, changes in humidity, pH, etc. are associated with the presence of a variety of 2D and 3D nanostructures formed at different hierarchical levels in “smart” materials. Their study requires new in situ experimental approaches with high temporal and spatial resolution.

The presented paper provides several examples of integrated methods for studying the structure and properties of adaptive materials using a synchrotron source. Such methods allow us to determine the mechanisms of formation of multilevel morphology of thin films of wedge-shaped amphiphilic compounds to produce organic zeolites and homeotropic-oriented films on their basis [1,2]. Analysis of the dynamics of structure formation of thermoplastic polyurethanes during deformation allows us to draw important conclusions about the nature of the shape memory effect in such polymers [3]. The presented results can be used to develop original approaches to obtaining new materials.

Acknowledgement: The authors acknowledge the Russian Science Foundation (grant 23-73-30005).

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OP-26

Synchrotron radiation methods for determining the fractal dimension of clusters in nanocolloidal gels and nanostructuring parameters in polymer films

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Recently, there has been great interest in the creation of materials that are natural analogues. Outstanding mechanical characteristics, sensitivity to external influences, optical and self-healing properties of natural materials are largely determined by the spatial arrangement of their structural and functional elements [1]. Natural materials are extremely difficult to reproduce due to their complex three-dimensional structure, which in turn may consist of various ordered substructures (natural silk fibers, Bouligand structures, layered structure of the eyeball, etc.). Self-organization and 3D printing are promising production strategies that allow the production of materials with predefined complex shapes, architectures and compositions. At the same time, a separate task is to characterize the obtained nanostructured materials. Synchrotron research methods are a powerful tool for obtaining information about the dimensionality and structuring of materials.

It is shown that the methods of synchrotron small-angle X-ray scattering make it possible to determine the fractal dimensions of clusters of latex nanoparticles and determine the mechanism of interaction between them [2]. Self-organization in colloidal particles is an important model system for studying self-organization in biological colloids. Synchrotron grazing-incident small-angle scattering was used to demonstrate the gradient in the refractive index in polymer lenses imitating the lens of the human eye. The nanostructure in perfluorinated sulfide-containing membranes, in which the self-organization of hydrophilic and hydrophobic blocks creates ion-conducting channels, was also studied using synchrotron scattering methods [3].

Acknowledgement: This work was supported by Ministry of Science and Higher Education of the Russian Federation (Goszadaniye) 075-03-2024-117, project No FSMG-2022-0012.

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Poster Presentations

PP-1 ÷ PP-65

PP-01

XPS study of molybdenum isopolycompounds

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Methods of studying polyoxo compounds of Group VI metals, including molybdenum, can be conditionally divided into two groups depending on the aggregate state of the sample at the time of analysis - solid-state (crystalline and amorphous) and solution [1]. Since the results of XRD (powder diffractography) of samples demonstrate their X-ray amorphous character, the list of possible methods to be used is rather strongly reduced. therefore, high-precision structural physicochemical methods are usually used for primary studies of the structure of isopolymolybdate compounds. In particular, X-ray photoelectron spectroscopy (XPS) is used to confirm the presence of the reduced form of molybdenum Mo^V in the analyzed isopolycompounds. In addition, this method can be used to confirm the presence of particle hydration, which according to literature sources is characteristic of molybdenum isopoly compounds [2]. The overview spectrum of molybdenum blue particles is shown in Figure 1.

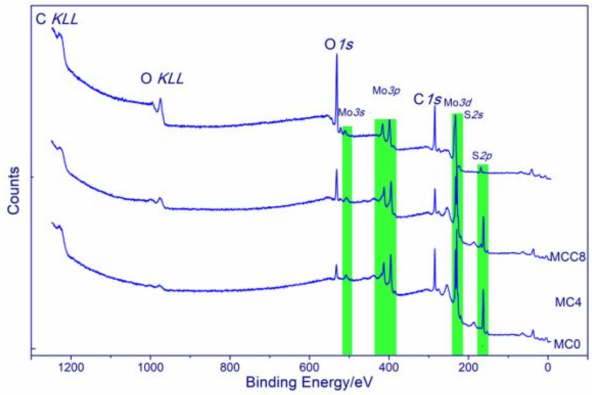

Fig. 1. XPS spectra of the obtained isopolymolybdate compounds

It should be noted that in order to obtain the most complete result, not a single method is usually used, but a complex of different physicochemical methods confirming and complementing the target analytical method**.**

The aim of this work is to investigate the physicochemical properties of the obtained isopolymolybdate compounds using the XRDES method.

Acknowledgement: The work was carried out within the framework of the state task of the Institute of Chemical Sciences of the Siberian Branch of the Russian Academy of Sciences, funded by the Ministry of Science and Higher Education of the Russian Federation.

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PP-02

Experimental investigations and density functional theory simulation of phase stability in Ti(Fe, Al)$_3$ alloy with L1$_2$ crystal structure

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Titanium trialuminide is an attractive structural material for the aerospace industry. This is due to the combination of its properties, such as low density, excellent heat resistance, oxidation resistance, high specific strength and Young's modulus [1, 2]. However, the $TiAl_3$ application is limited by its high brittleness arising from tetragonal D0$_{22}$ crystal structure [3]. The method of solving this problem is the alloying of titanium trialuminide with the following transition metals: Cr, Mn, Fe, Co, Ni, Cu, Zn, Rh, Pd, Ag, Pt and Au. This makes it possible to transform the tetragonal D0$_{22}$ lattice into the cubic L1$_2$ [4]. However, the field of homogeneity of the L1$_2$ phase is very narrow in ternary systems. Therefore, the L1$_2$-based alloys often contain second phases, which can reduce the fracture toughness and ductility of the material. To avoid the appearance of the minor phases, it is necessary to adjust the composition through numerous experiments, because the literature data on the homogeneity range is often not very precise. The other approach to formation of a single-phase alloy is selection of the appropriate heat treatment which allows for the dissolution of the second phase. In this work, the second approach was examined.

Titanium trialuminide based alloy stabilized with iron in the amount of 9 at. % was obtained by arc melting in a Büehler ArcMelter AM furnace. The alloy has the $Ti_{26}Fe_9Al_{65}$ stoichiometry. According to the Ti-Fe-Al ternary phase diagram [5], the composition of the alloy corresponds to the L1$_2$ single-phase field. However, structural investigations have shown that in addition to the cubic modification of titanium trialuminide, the $TiFeAl_2$ inclusions were formed in the alloy. The presence of this phase did not allow for the significant increase of fracture toughness of the alloy; it varied withing the range from 1.8 to 7.0 $MPa \cdot m^{1/2}$.

To reveal the possibility of obtaining the L1$_2$ single-phase alloy, the relative phase stability of Ti(Fe, Al)$_3$ and $TiFeAl_2$ was simulated using density functional theory (DFT) and in-situ synchrotron X-ray diffraction (SXRD) analysis during heating of the alloy was performed. Diffraction patterns were obtained at the 8A beamline of the VEPP-4 source at the «Siberian Synchrotron and Terahertz Radiation Centre». The energy was 69.5 keV, which corresponded to a wavelength of 0.178 Å. The sample was heated by a laser beam.

The results of DFT simulations showed that the $TiFeAl_2$ phase has a lower Gibbs energy and, therefore, more stable compared to L1$_2$-$TiAl_3$ phase. Thus, homogenizing would not be able to change the formed structure. However, SXRD measurements revealed total dissolution of the second phase at a temperature close to the melting point of the alloy (Fig. 1).

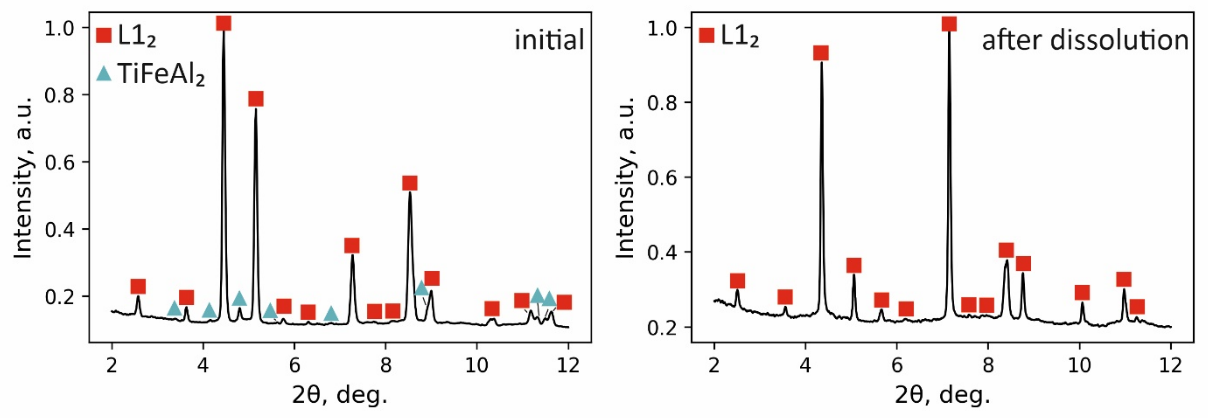

Fig. 1. Diffraction patterns of $Ti_{26}Fe_9Al_{65}$ alloy in the initial state and after dissolution of the second phase

Based on the results obtained by SXRD and density functional theory simulation, the alloys were quenched at 1200 °C. The holding time at this temperature was 2 hours. As can be seen in Figure 2, heat treatment contributes to the almost total dissolution of the $TiFeAl_2$ second phase, which was replaced by pores.

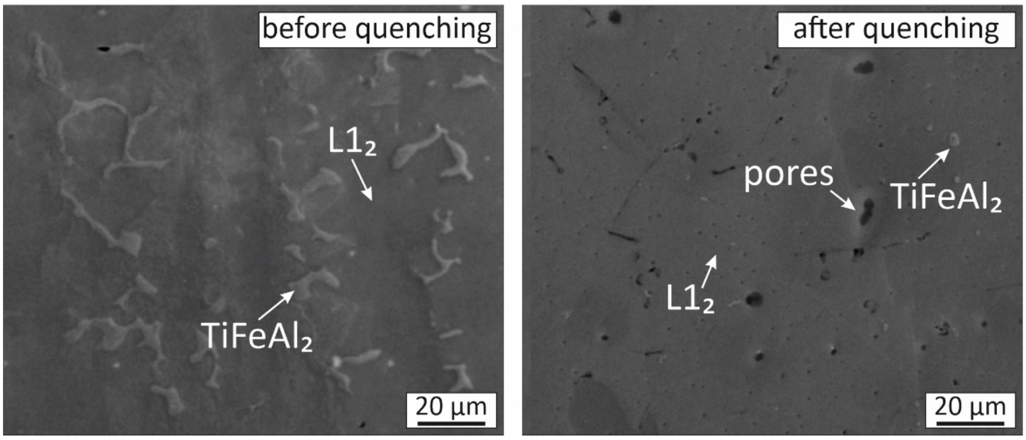

Fig. 2. Structure of $Ti_{26}Fe_9Al_{65}$ alloy before and after quenching

Thus, it can be concluded that by quenching at 1200 °C, it is possible to form a single-phase $L1_2$ structure in $Ti_{26}Fe_9Al_{65}$ alloy. However, a longer holding time is required for the total dissolution of the minor phase.

Acknowledgement: The study was supported by the Ministry of Science and Higher Education of the Russian Federation as a part of the state task FSUN-2024-0005 «Structural transformations in surface layers of metal alloys under extreme thermal and deformation impacts».

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PP-03

The technique of synchrotron studies of explosive processes

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The basic properties of synchrotron radiation (SR) allow measurements to be carried out in dynamic experiments with very small exposures (less than 1 ns), in which the results of interaction with matter are recorded at successive points in time (i.e., you can make a "film"). In the described experiments, the SR source is the VEPP-4 storage ring (electron energy – 4.5 GeV, wiggler with 9 poles).

In this work, a series of explosive experiments was carried out with simultaneous registration of relative intensity changes along the axis of structural materials (foam, fluoroplast, 3D structural aluminum) by the SR method. The registration of the transmitted radiation was carried out by the DIMEX X-ray detector, which was located parallel to the axis of the material. On the basis of experimental data, the velocities of the shock wave front in foam, fluoroplast and 3D structural aluminum are obtained. In experiments with aluminum, it was possible to obtain the value of the mass velocity in the sample. When conducting a series of similar experiments with 3D structural aluminum, it will be possible to build a shock adiabate for this substance.

A relative change in intensity along the fluoroplast axis was obtained after compression by a strong shock wave (Fig. 1)

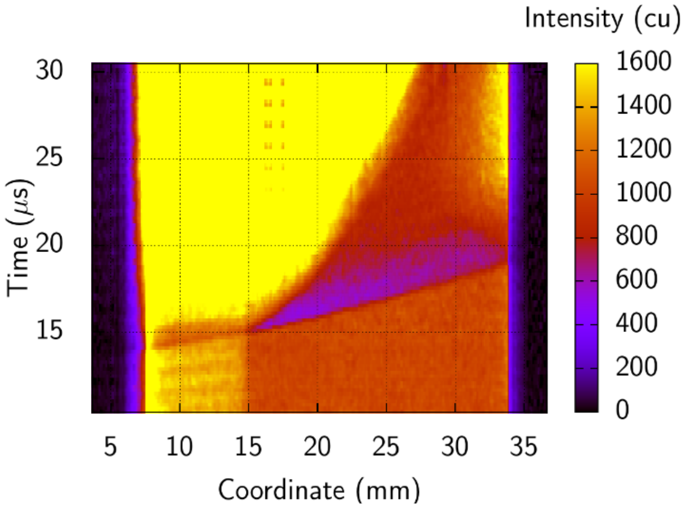

Fig. 1. A relative change in intensity

Acknowledgement: This work was carried out with the financial support of the Priority 2030 program

PP-04

In situ/ operando study of MoOx/TiO$_2$ catalysts for selective oxidation of alcohols

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One widely used method for producing aldehydes and carboxylic acids is the selective oxidation of alcohols using heterogeneous catalysts. While the application of alcohol oxidation processes has been successfully implemented, certain fundamental factors that influence the activity and selectivity of the catalytic reaction remain poorly understood, due to the multicomponent nature of the catalysts or the transformation of active centers during the reaction. To elucidate the mechanisms of a catalytic process, it is essential to investigate the relationship between physicochemical properties and catalytic behavior. Therefore, the development of advanced methods for the systematic study of catalysts, including in situ and operando techniques, is of a significant challenge.

Catalysts based on molybdenum oxide (MoOx) deposited on titanium dioxide (TiO$_2$) were chosen for this study, as they are considered highly promising for the selective oxidation of alcohols at temperatures between 150°C and 200°C, according to literature reports.

To determine the reaction mechanism, the structure and composition of the MoOx/TiO$_2$ catalyst were characterized by X-ray diffraction analysis, electron microscopy, *in situ* infrared spectroscopy, X-ray photoelectron spectroscopy (XPS), and *in situ/ operando* X-ray absorption spectroscopy (XAS). The effect of temperature and reactant ratio on catalyst activity was investigated, and the catalytic activities of samples with different molybdenum oxide contents were compared. The results of this study allowed us to propose a mechanism for the selective oxidation of methanol and ethanol on MoOx/TiO$_2$ catalysts.

Acknowledgement: This work was supported by the Ministry of Science and Higher Education of the Russian Federation (Agreement No. 075-15-2022-263). This work was supported by the Priority 2030 program.

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PP-05

Synthesis and study of Bi_2Se_3 derivatives using photoelectron spectroscopy

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Topological insulators (TIs) represent a new quantum state of matter. TIs are insulating in the volume while strong spin orbital coupling on the surface creates topologically protected surface states with Dirac cone dispersion [1,2]. TIs have unique electronic properties and are considered as promising materials for use in spintronics and quantum electronics [3, 4].

In this work the electronic properties of bismuth selenide single crystals doped with 3d elements (Mn, Co) and In have been investigated. The single crystals were synthesized using Bridgman vertical technique. These materials are of interest because magnetic impurities create an internal magnetic field that can break the time reversal symmetry which can lead to anomalous quantum Hall effect and magneto-optic effect. The electronic structure of the compounds has been investigated by XPS and ARPES methods including the use of synchrotron radiation source. Based on a series of photoemission spectra obtained at different incident photon energies, the dominant valence partial densities of electronic states for the compounds $Bi_{1.8}Mn_{0.1}In_{0.1}Se_3$ and $Bi_{1.8}Co_{0.1}In_{0.1}Se_3$ were reconstructed by the method of statistical Bayesian inference considering the known photoionization cross sections of valence electronic states of ions.

Acknowledgement: This work was supported by the Russian Science Foundation, grant 23-72-00067.

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PP-06

Incommensurately modulated structure of synthetic nyerereite under high-pressure

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Nyerereite, a carbonate mineral of chemical composition of $(Na,K)_2Ca(CO_3)_2$ has been attracting attention since 1962 due to its incommensurately modulated crystal structure. Its structure at ambient conditions was described by Bolotina et al. [1] using (3 + 1)-dimensional symmetry group $Cmcm(\alpha00)00s$ with modulation vector $\mathbf{q} = 0.383(1)\mathbf{a}^*$ and three orthorhombic components related by threefold rotation about [001] direction. Here we present the high-pressure study of a synthetic nyerereite ($Na_{1.64}K_{0.36}Ca(CO_3)_2$) structure up to 8 GPa, based on single-crystal X-ray diffraction data collected at ESRF ID15B. Data reduction was performed separately for each orthorhombic twin component. The value of modulation vector **q** is independent from applied pressure and retains value of about 0.374**a***. No irregularities in pressure behavior was found for unit cell parameters up to 8 GPa; 3-rd order Birch-Murnaghan equation of state has $V_0 = 559.2(8)$ Å^3, $K_0 = 30.0(11)$ GPa, $K'_0 = 6.4(5)$. At ambient conditions incommensurate modulation manifest itself in: (1) drift of Ca atoms along *c* axis with an amplitude about ±0.4 Å (Fig.1); (2) similar drift of perpendicular to crystallographic *c* axis CO_3 groups with an amplitude about ±0.5 Å; (3) variation of Na/K ratio between 0 and ~1.

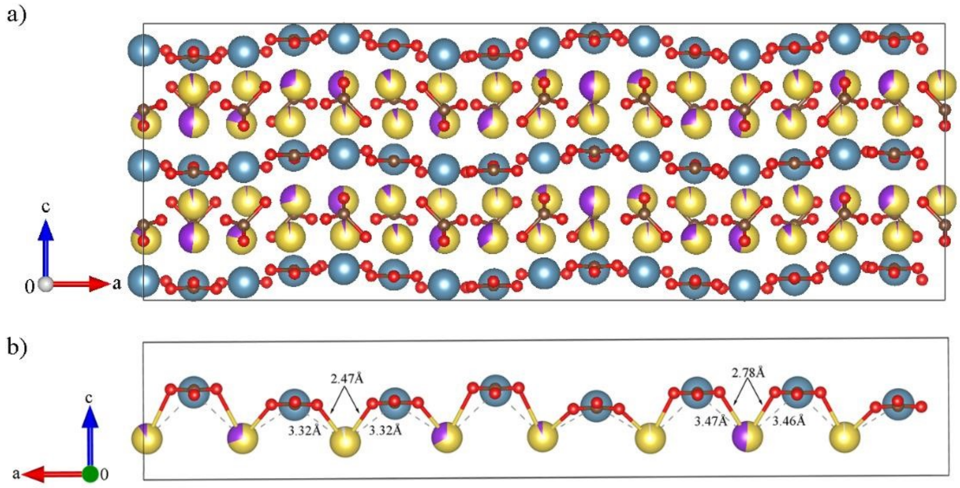

Fig. 1. (a)The approximant (8×1×1 unit cells) of incommensurately-modulated crystal structure of synthetic nyerereite. Ca atoms are shown in blue, O atoms – in red, C atoms – in brown, Na – in yellow, and K – in purple (filled according to the Na/K ratio), (b) – changes in local coordination of Na/K mixed position along modulation vector

The obtained data in the pressure range from 1atm. to 8.2 GPa show the stability of the incommensurately-modulated structure of synthetic nyerereite under hydrostatic compression. The relative values of the modulation vector are almost independent of pressure which is related to chemical nature of modulation. The unit cell and the position modulation parameters show a change in structural behavior at pressures around 2 and 5 GPa, which is correlated with Raman spectroscopy observations. The obtained bulk modulus value is significantly lower than those of other carbonates with similar compositions.

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PP-07

Current status of “Electronic Structure” Beamline 1-6 at the SRF SKIF

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The report will present the progress of the development of the synchrotron beamline 1-6 «Electronic structure» which is allow to realize the methods using the Soft X-Rays range (10-2000 eV) with two end-stations, located in independent beamline branches, for Near Ambient pressure X-Ray Photoelectron Spectroscopy (NAP XPS) and Spin-Angle Resolved Photo-Emission Spectroscopy (Spin-ARPES). The NAP XPS end station will allow carrying out *in situ* and *operando* studies of a wide range of catalytic systems; *in situ* studies of regularities of deactivation/poisoning processes for catalytic systems depending on different conditions; also *in situ* study of innovative functional materials. Another important technique which will be realized on a beam line (the other branch line) is Spin-Angle Resolved Photo-Emission Spectroscopy (Spin-ARPES) end station which will allow to study the electronic and spin structure of solids for applications in nanoelectronics and spintronics.

The beamline is based on the use of an electromagnetic undulator with ability to change beam polarization. It will allow generating the synchrotron radiation in VUV and Soft X-Ray range (10-1800 eV). Two endstations at the "Electronic structure" will work alternately. The optical scheme is based on a plane grating monochromator and set of focusing mirrors with total external reflection.

The report will outline the development status of each component of the beamline, as well as its key parameters. In addition, it will describe the methods and equipment that will be available to users in the powder diffraction and macromolecular crystallography sections.

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PP-08

Development of the confocal X-ray microscope module for studying the elemental composition with high spatial resolution

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μ-XRF is a perspective technique for elemental analysing of natural environment objects and for technological objects. This method allows one to obtain the information about the spatial distribution of a wide range of chemical elements (from S to U) within its surface and sub-surface layers with relative simplicity and without destroying the sample. Combination with μ-XAFS makes it possible to supplement the obtained information with the structural features of inclusions.

A unified module confocal X-ray microscope (CXM) based on polycapillary x-ray optics has been developed and assembled at the INP SB RAS for μ-XRF and μ-XAFS investigation. Spread function and the factors of its dependences has been determinated in detail [1, 2], the using spread function allow the size of confocal volume to be changed controllably in range 15-100 mkm.

The developed approaches allow studying natural or technological samples in 2D and 3D investigations. The method is used to search for micro-inclusions with a size of about ten microns and study them by micro-XAFS spectroscopy. The spatial resolution reaches 10 μm, and the limits of detection (units ppm) are limited by the capabilities of the synchrotron radiation source used, X-ray optics, and quantitative methods for analysing the obtained spectrometric data. The use of mathematical data processing methods being developed by us, for example, various deconvolution approaches [3], allows one to increase the spatial resolution by several times, and the use of the method of fundamental parameters can improve the detection limits significantly.

The success of CXM using in experimental researches at VEPP-3 (BINP SB RAS), for example, with micro-aerosol particles [4], Pd and W particles [5, 6] allows us to offer the same module for realization at SRF SKIF “Microfocus” beamline.

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PP-09

Synchrotron XANES and XPS studies of the SnO_2 wire-like crystals

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Currently thin-film structures based on tin oxides are prospective as a gas-sensitive material. Nanoscale materials based on tin dioxide such as wire-like crystals with a high surface-to-volume ratio are really promising in terms of applied properties for various applications. Knowledge about the local atomic surrounding specificity of the wire-like crystals developed surface, its physico-chemical state will allow the controlled formation of such objects with specified properties.

In present work tin oxide wire-like crystals obtained by gas transport synthesis technique by vapor-liquid-solid (VLS) strategy were studied. Additionally the thin 30 nm tin oxide films obtained by magnetron sputtering and annealed in air as well as the films obtained by metal-organic chemical vapor deposition, and the natural cassiterite SnO_2 crystals were also studied. The research was carried out by X-ray Absorption Near Edge Structure Spectroscopy (XANES) and X-ray Photoelectron Spectroscopy (XPS) techniques using highly brilliant synchrotron radiation.

Synchrotron XANES and XPS studies have shown that a significant quantitative contribution of the surface compared to the volume of tin oxide wire-like crystals (with a minimum cross section of ~ 20 nm and with a length of up to several micrometers) leads to the influence of surface imperfections on the electronic spectrum, composition and physico-chemical state. The lack of oxygen atoms in the layers of the wire-like crystals developed surface leads to the formation of the peak in the synchrotron XANES spectra corresponding to the free electronic states in the band gap of tin dioxide close to the conduction band bottom. In the case of conditions ensuring the formation of an ordered (crystalline) structure of tin dioxide the specificity of morphology does not significantly affect the distribution of the main features of the electronic spectrum, including for wire-like crystals. Additional oxidation of tin metal films to SnO_2 (SnO_{2-x}) leads to the formation of a two-phase system consisting of orthorhombic and tetragonal tin dioxide. Variations in the wire-like tin oxide crystals formation conditions can make it possible to control their atomic and electronic structure, physico-chemical state prospective for modern electronic devices.

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PP-10

Synchrotron methods for studying thermoplastic polyurethanes: evolution of methods and development prospects

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The theoretical foundations of synchrotron research methods were developed at the turn of the 19th and 20th centuries. Synchrotron radiation was discovered in the 1940s. This was initially viewed as a side effect. Since then, the branch of research methods using accelerators has been rapidly developing. Second generation of synchrotrons were purposely built to produce synchrotron radiation for scientific research. By the end of the 1990s, synchrotron methods such as small-angle X-ray scattering (SAXS) and wide-angle X-ray scattering (WAXS) began to be actively used to study the structure of polymers, including materials based on thermoplastic polyurethanes (TPU). Nowadays, synchrotron research methods provides wide analytical capabilities. For example, synchrotron radiation was used to study the structure and deformation relationships of polyurethane elastomers. The knowledge gained from this analysis has played a significant role in the development of advanced TPU-based materials with improved performance characteristics. Thus synchrotron-based research has led to the development of thermoplastic polyurethanes with improved thermal stability, durability and mechanical properties, which are used in various application areas, including medical materials. Although synchrotron techniques provide powerful tools for studying the structural and mechanical properties of polymers, their effectiveness is often limited by material standards, difficult-to-validate data, and practical limitations of experimental setups. Looking ahead, the integration of synchrotron methods with new technologies such as machine learning and data analytics is expected to further improve the understanding of material structures. This may facilitate the development of next-generation TPU-based materials with optimized properties for specific applications, which is relevant in various industrial sectors. Thereby, the history and development of synchrotron methods in thermoplastic polyurethane research reflects a trajectory of technological progress that has significantly improved our understanding and created prospects for the discovery of new polymer materials.

PP-11

Novel complexes of Nd(III) and Gd(III) with 2-ethyl-4-oxo-4H-pyran-3-olate ligands: preparation and study of the crystal structures

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2-Ethyl-3-hydroxy-4H-pyran-4-one (ethyl maltol, Hema), known as the flavor enhancer E637, is considered a promising chelating ligand for the preparation of complexes, including those for biomedical applications [1]. Ethyl maltol is reported to enhance the membrane permeability and overall *in vitro* anticancer activity of the different transition metal complexes [2, 3]. Single crystals of a number of complexes with common formula $X[Ln(ema)_4]\cdot 2H_2O$, where X = Li, K; Ln= Nd, Gd, were obtained by crystallization in agarose gel. For all compositions, two polymorphic modifications were found. The crystal structures of the compounds were determined using single-crystal X-ray diffraction. The complexes $Li[Nd(ema)_4]$ and $Li[Gd(ema)_4]$, as well as $K[Nd(ema)_4]$ and $K[Nd(ema)_4]$ are isostructural to each other, while $Li[Ln(ema)_4]$ and $K[Ln(ema)_4]$ have significant differences in crystal packing.

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PP-12

Study of shear instability of piroxicam-succinic acid cocrystal: quantum chemical approach

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Modelling of materials properties is of great importance for understanding the patterns of their behavior upon change of external conditions, including mechanical impacts.

Multi-component crystals that contain medicinal chemicals, known as cocrystals if the components stay neutral and do not turn into salts, present great interest for pharmaceuticals. Such cocrystals are often synthesized in a mechanochemical way, as well as being subject to shear and impact mechanical action on different commercial production steps, including dispersion and tableting. Earlier it was reported that cocrystals (**PS**) of piroxicam (**P**) and succinic acid (**S**) show an interesting synthesis behavior: the mixture of **S** and **P** turns into cocrystal if treated with impacts, but shear action leads to decomposition of **PS** to **P** and **S** [1].

Quantum chemical modelling of crystals and functional materials allows to explain and predict mechanical properties of crystals [2]. In this work, density functional theory (DFT) and orbital-free (OF) crystallography approaches are used for mechanical properties investigation to determine the causes of shear instability of cocrystal **SP** and its coformers. Hidden features of the electron density (ED) distribution and its connection with the elasticity of crystals are revealed. The ground-state wavefunctions of electrons in **S, P,** and **SP** crystals are obtained using CRYSTAL17 program package [3]. The periodicity of the electron wavefunctions is taken into account. Elasticity moduli are calculated and analyzed in Voigt, Hill, and Royce approximations. Quantum topological analysis of electron density is performed in TOPOND14 sofware, full set of critical points is found. Poincare-Hopf relationship is established. Inner descriptors of chemical bonding such as quantum electronic pressure [4] are calculated and analyzed.

The correlation between the directional features of crystal packing compressibility and the intermolecular interactions directions was established via analysis of the most rigid and most compliant axes of elasticity tensor. Shear instability of cocrystal **SP** is discussed and the connection to spatial organization of intermolecular contacts in the crystal is studied. Elasticity of electronic continuum is studied. The link between macroscopic elastic properties and microscopic stresses of electron density is established.

Calculations were performed with the usage of NUSC NSU cluster resources.

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PP-12

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PP-13

The effect of planar defects on the diffraction patterns of tetragonal Ruddlesden-Popper structures of the $A_{n+1}B_nO_{3n+1}$ series

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Structures of the RP series with the general formula $A_{n+1}B_nO_{3n+1}$ arouse a growing interest of researchers in the field of materials science and technologies because materials based on these structures can potentially be used as superconductors, catalysts and thermoelectrics, and in other physicochemical applications. Variations of the chemical composition of these oxides (A = La, Sr, Ca, Ba and others, B = Ti, Sn, Nb, Cu and others) determine the possibility of controlling their functional properties. This is widely used in materials science to search for the optimal compositions intended for different applications. Other potentialities for varying the properties of materials based on the oxides of RP series are determined by the features of their crystal structure. The crystal-chemical formula presented as $AO(ABO_3)_n$ adequately reflects the fact that structures of the RP series can be considered as the sequences of alternating layers with the perovskite-type ABO_3 and rock-salt AO structures (Fig. 1).

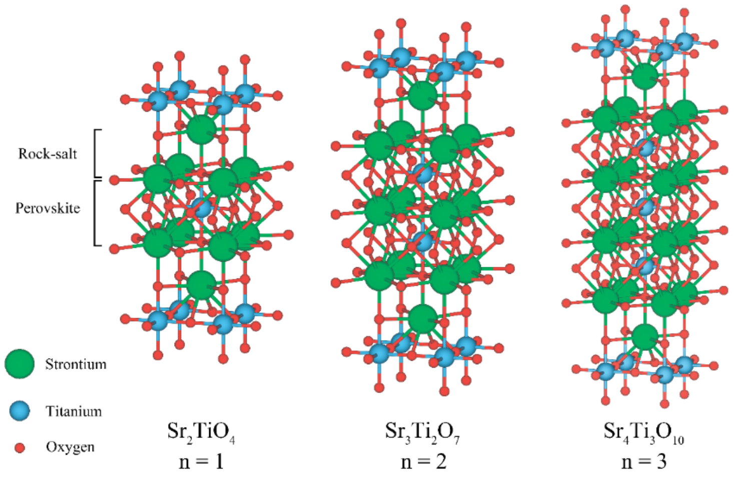

Fig. 1. Crystal structures of the first three terms of the Ruddlesden–Popper series $Sr_{n+1}Ti_nO_{3n+1}$ (n = 1-3).

In such structures, the alternation of layers can be easily disturbed, which is associated with changes in consecutive coupling of different number of ABO_3 blocks. As a result, fragments with the content of perovskite-type layers differing from the specified stoichiometry can emerge in phases of the RP series (for example, fragments of the A_3B2O_7 structure can appear in A_2BO_4 particles, etc.). Violations of the layer alternation in these oxides and also the local structures like inclusions of the fragments of one term of RP series into the matrix of another term of the series were repeatedly observed by many authors in electron microscopy studies [1, 2]. However, it is quite difficult to estimate quantitatively from electron microscopy data the content of defects and deviations of stoichiometry of the formed phases from the nominal composition of samples, if the entire sample is considered instead of local regions. X-ray diffraction is a classical method for analyzing the crystal structure of chemical compounds. Diffraction patterns can be quite sensitive to violations of the periodic structure. The presence of defects in the structure of crystals may affect the intensity ratio of diffraction peaks, their position and broadening, also it may lead to pronounced diffuse scattering effects. For specific types of defects in specific structures, using methods for simulation of imperfect crystals and calculation of the corresponding diffraction patterns, one can reveal the most typical features of diffraction, which make it possible to qualitatively identify the presence of certain defects and quantitatively estimate their content. Thus, the analysis of the effect exerted by RP faults for tetragonal A_2BO_4 structures of the RP series (using Sr_2TiO_4 as an example), which was performed for the first time in our study, allowed us to relate these structural violations to the shifting and broadening of some diffraction maxima in the powder diffraction patterns [2]. This effect was used to develop a simple technique for estimating the content of RP faults from the ratio of interplanar distances with certain indices hkl for the particular case of low ($\delta \leq 0.1$) content of such defects in the absence of a correlation in their distribution [3]. However, the problem of defectiveness in oxides of the RP series deserves more attention. Other terms of the series should also be considered, as well as the cases of local ordering (correlation in the distribution of defects) leading to the formation of nanostructured coherent systems (inclusions of structural fragments of one phase into the matrix of another phase with the retained orientational order).

This study considers the effect of randomly distributed RP faults on the diffraction patterns of the first and second terms of the RP structural series $A_{n+1}B_nO_{3n+1}$ for tetragonal modifications of these phases (using Sr_2TiO_4 and $Sr_3Ti_2O_7$ as examples). To use the methods of estimating the content of RP faults described in this work, high-quality diffraction data (high signal-to-noise ratio and minimum reflection width) are required. The results of this work can be used to develop express methods for determining the content of planar defects in tetragonal (and in the future, perhaps, not only) Ruddlesden-Popper structures at diffraction stations of synchrotron X-ray sources.

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X-ray absorption and resonance photoemission studies of rare earth magnetic alloys

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The electron and magnetic states in multicomponent rare-earth (RE) intermetallic of the RNi_4Cu and RNi_2Mn_x type (R=Gd, Tb, Dy, Er) were studied by X-ray absorption (XAS) and resonance photoemission spectroscopy (RXPS) methods using synchrotron radiation. Unlike direct photoemission (PE) of valence electrons, the resonant PE process occurs in two stages. If the photon energy reaches the excitation edge of the core level, for example Gd M5 with a binding energy of 1184 eV, then the 3d electron from this level goes into unfilled valence 4f states, then it can return back, transferring energy to one of the valence electrons, which will be counted by the detector. The intensity of the resonance process can be many times greater than the intensity of direct photoemission of valence electrons. The weak photon interaction (Fourier transform) with a well-localized core-electron state is much greater than with a delocalized valence state, and the reverse transition has a higher probability, since it occurs due to the strong Coulomb interaction. It is known that the resonant PE "highlights" the partial contribution of the excited atom in the total valence band of the compound, since the Coulomb interaction works (screening) at short distances.

The elastic process described above has competitors. The core-level hole can be filled not by the excited electron, but by one of many other valence electrons, and energy will be lost in the formation of an additional electron-hole pair. Finally, the excited electron can simply leave the parent atom, and the inner hole will be filled with the emission of an Auger electron with a different kinetic energy. We draw attention to the very strong dependence of the PE on the value of the magnetic moment of the RE atom. The conservation of the electron spin projection during X-ray and Coulomb transitions imposes a strong limitation on the number of possible intermediate and final states. In the case of a half-filled 4f shell (7↑ and 0 ↓ electrons), the elastic channel has no competitors (if limited to only 4f states). As electrons with spin "down" are added, inelastic losses increase and the intensity of resonant photoemission decreases.

A very strong dependence of the resonant enhancement of the PE from the valence band on the magnitude of the magnetic moment of the RE atom at the M5 excitation edge (3d – 4f) has been experimentally established (Fig. 1). The PE amplitude increases at the 3d-4f resonance by 41 times on Gd, by 17 times on Tb and only by 8 times on Dy. The XPS increases at the resonance much more strongly than the absorption. Moreover, only the magnitude of the moment is important, and its direction in space or magnetic order are of no importance. Being a second-order process, the resonant photoemission at the first stage creates a spin 3d probe, which reads the spin moment of the valence shell at the second stage.

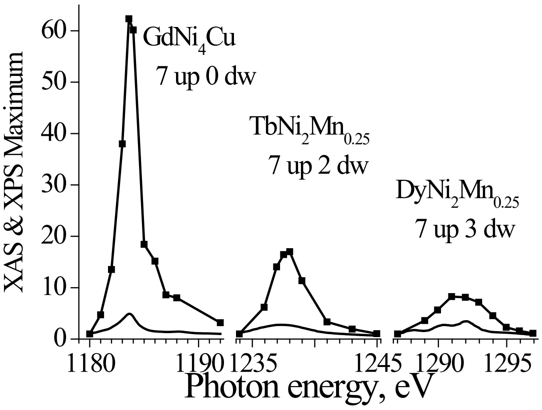

Fig. 1. Maximum photoemission value (dots) and M5 edge of absorption spectrum (solid line) versus photon energy.

A striking distinctive feature of the absorption and resonance at the N edge is the transition of the 4d electron to the so-called "giant resonance" state. It is located 7-20 eV above the absorption edge in Fig. 2. It is this state that determines the energy of the maximum of the resonance FE. Note that the power of the state decreases as the 4f shell is filled and the atomic magnetic moment decreases. In erbium (7↑ and 5 ↓ electrons), the absorption into two unfilled 4f states and into the "giant resonance" state become approximately equal in magnitude.

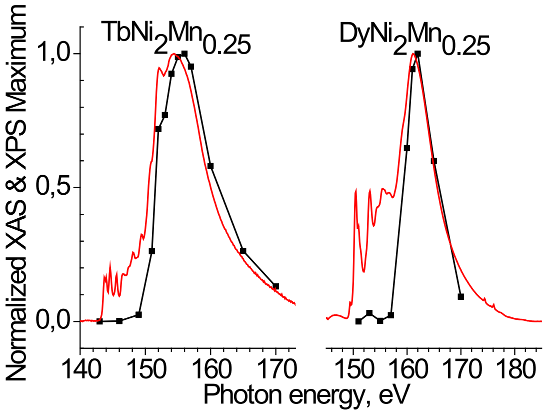

Fig. 2. Absorption spectrum of the N edge (red line) and the maximum value of photoemission from the valence band (dots), normalized to (0, 1).

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PP-15

Disclosing the mechanism of uranium(VI) solvent extraction by polydentate ligands in a polar solvent: the role of ion pairs

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Worldwide, new solvent extraction systems for the separation of the transuranium elements from spent nuclear fuel are under development. Polydentate N,O-donor extractants are promising for extraction and separation of f-elements. Understanding the extraction mechanism is key to controlling the separation system. The composition of the complex in the organic phase can change significantly with the macro quantities of the extracted component, as well as the type and especially the polarity of the solvent used. In this work, we synthesized 2-ethylhexyl substituted pyridine-2,6- and 1,10-phenanthroline-2,9- diphosphonates (Figure 1) for high solubility in organic solvents and phase stability of the systems in contact with high concentrations of uranyl nitrate. To establish the extraction mechanism we used a combination of spectroscopic methods (UV-vis, ^{31}P NMR and Raman spectroscopy) and synchrotron based techniques – EXAFS. The experimental data obtained were supported using quantum mechanical calculations both in terms of the thermodynamics of possible extraction processes and in terms of modeling spectroscopic data (UV-vis and EXAFS).

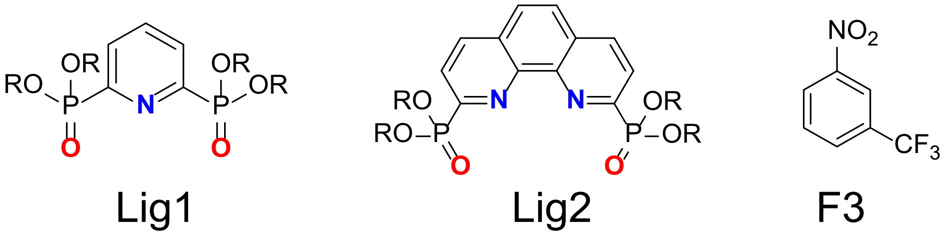

Fig. 1. Structures of ligands and solvent (F3) used in the research

First of all an isotherm for the extraction of uranyl nitrates were constructed both ligands exhibit high loading capacity, making them promising candidates for industrial use as U(VI) extractants. The coordination structures of uranyl nitrate complexes with both pyridine- and phenantroline-based ligands formed during extraction were further studied in detail. Through a combination of experimental (loading isotherms, UV-vis and EXAFS) and theoretical methods we have shown that UO_2^{2+} forms complexes with the structure of tight ion pars $([UO_2\mathbf{L}NO_3]^+[UO_2(NO_3)_3]^-)$ with tetradentate phenanthroline-based ligands and a 1:1 mixture of complexes with different stoichiometry, supposedly, $[2LUO_2](NO_3)_2$ and $[2LUO_2][UO_2(NO_3)_3](NO_3)$, with tridentate pyridine-based ligands.

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PP-16

Investigations of coating growth from gas discharge plasma in real time by X-ray phase analysis using synchrotron radiation

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The aim of this work was to study the process of coating formation based on a highentropy alloy of the TiNbZrTaHf composition, carried out ***in situ*** by X-ray phase analysis using synchrotron radiation. Metal and nitride coatings based on HEAs were formed in the form of thin (2-2.5 µm) films by deposition of a multi-element gas-metal plasma created in an environment of ionized argon (metal film) or nitrogen (nitride film) by electric arc plasma-assisted sputtering of a TiNbZrTaHf cathode composition close to equiatomic. The structure of thin films formation process ***in situ*** with high time resolution was studied using a synchrotron radiation source - the VEPP-3 electron storage ring of the Institute of Nuclear Physics SB RAS. To confirm the results of X-ray diffraction in the ***in situ*** method, electron diffraction was used to study the resulting films using transmission electron diffraction microscopy.

The multistage, multiphase nature of the growth process of metal and nitride films has been revealed.

It has been established that at the first stage of a metal film deposition, the β-phase (bcc crystal lattice; a = 0.34748 nm) is formed. At the second stage, the formation of an amorphouscrystalline phase is recorded, as evidenced by a rise in the background of the diffraction line in the angular range 2θ = (24-25) degrees. At the final stage of deposition, in this angular range, diffraction lines of the ω-phase (hexagonal crystal lattice; a = 0.46636 nm, c = 0.27872 nm) and α-phase (hcp crystal lattice; a = 0.31261 nm and c) are detected = 0.47846 nm). Relative content of phases after completion of the deposition process: β-phase – 84 wt.%; α-phase – 14 wt.%; ωphase – 2 wt.%. It has been shown that by alloying a metal film with copper by simultaneous sputtering of two cathodes (TiNbZrTaHf and a copper cathode), the resulting film has an amorphous-crystalline (X-ray amorphous) structure.

The deposition of a metal sublayer located in the contact zone of the coating and the substrate (the first stage of coating deposition) is accompanied by the formation of a three-phase system represented by metal phases β-phase, α-phase and ω-phase, which are in amorphous- crystalline state was established during studying the process of formation of a nitride coating. The nitride layer formed on a metal substrate is represented by two (MeN and MeN*) phases, differing in the type of crystal lattice. It was found that the diffraction lines of the nitride phases are blurred, which may be due to the nanocrystalline (0.7 - 1.2 nm) state of the coating.

Thus, the use of ***in situ*** studies using X-ray phase analysis methods using synchrotron radiation made it possible to identify the time intervals for the formation and restructuring of the detected phases.

Acknowledgement: The work was supported by the Ministry of Science and Higher Education of the Russian Federation under project No. 075-15-2021-1348).

PP-17

Preparation and diffraction studies of solid phases of tecovirimat

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The abolition of mass vaccination at the end of the 20th century led to an increased likelihood of human infection with zoonotic infections - orthopoxviruses transmitted from animals to humans and posing a potential biological threat. A total of 99 176 laboratory-confirmed cases of monkeypox virus infection, including 208 deaths, were reported to World Health Organization (WHO) (data of access: 17th august 2024). Due to the current epidemiologic situation, the development and improvement of vaccines and diagnostic tests, as well as the design of drugs, became an urgent task.

Currently, one of the most effective drugs against orthopoxviruses is tecovirimat (fig. 1). Tecovirimat inhibits cowpox virus (CPXV) replication at a half-maximal effective concentration (EC50) of 0.01 μM, with an index of selectivity (IC50) of 160 000 units. The antiviral activity of tecovirimat is associated with inhibition of enveloped virus formation and prevention of virus spread in vitro and in vivo [1].

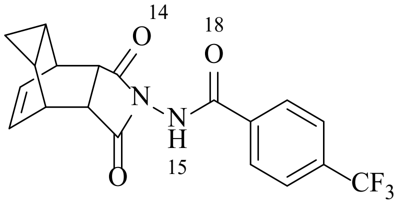

Fig. 1. Chemical formula of tecovirimat (N-((4R,4aR,5aS,6S,6aS)-1,3-dioxo-3,3a,4,4a,5,5a,6,6a-octahydro-4,6-ethenocyclopropa[f]isoindol-2(1H)-yl)-4-(trifluoromethyl)benzamide

From the patent applications it is known that six solid phases have been identified for tecovirimat. There are 2 monohydrates (**I**, **III**), one hemihydrate (**V**), and three anhydrous phases (**II**, **IV**, **VI**). The crystal structure is known only for the monohydrate phase **I** (CSD Refcode: UPUDOZ), and it is this hydrate that is found in the currently used dosage forms. Determination of the conditions for obtaining phases **II**-**VI** and their crystal structures is an important fundamental link that may be useful for the development of new active dosage forms of tecovirimat. [2].

Series of crystallization experiments were carried out (Table 1), and single crystals tecovirimat phases **I**, **III**, **IV**, and **V** were obtained. The crystal structures for the phases were determined by X-ray diffraction analysis. Theoretical powder diffraction patterns were calculated from the obtained crystal structures and compared with the experimental powder diffraction patterns given in the patents. Using X-ray phase analysis, it was established that during crystallization of tecovirimat from THF, a mixture of phase **I** and phase **III** crystals is obtained.

Table 2. Relation of crystallization conditions and obtained during experiments tecovirimat phases

Experiment number	Solvent	Crystal shape	Phase
1	Acetone	Prism	**I**
2	Acetonitrile	Prism	**I**
3	Benzene	Prism	**III**
4	Benzene (dry + molecular sieves 3Å)	Needle	**IV**
5	Benzene (dry)	Plate	**V**
6	DMF	Prism	**I**
7	Ethanol	Prism	**I**
8	Ethyl acetate	Prism	**I**
9	Ethyl acetate (dry)	Prism	**III**
10	n-butanol (dry)	Prism	**III**
11	Nitromethane	Prism	**I**
12	THF	Prism	**I, III**
13	Toluene	Prism	**III**
14	Water	Needle	**I**

It is found out that to obtain single crystals corresponding to anhydrous phases **II**, **IV** and **VI**, it is required to add molecular sieves with 3 Å pore diameter in the tecovirimat solution for its dehydration.

Acknowledgement: This work was supported by the Ministry of Science and Higher Education of the Russian Federation within the governmental order for SRF "SKIF" Boreskov Institute of Catalysis (FWUR-2024-0040).

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OP-18

Effect of temperature, hydrostatic pressure and irradiation on photosensitive complexes of $[Co(NH_3)_5NO_2]XY$

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Noncovalent interactions have a significant impact on chemical transformations in solids. By influencing noncovalent interactions one can control the structure and reactivity of a substance. Noncovalent interactions are sensitive to external influences, such as heating, cooling and pressure. They determine the crystal structure, anisotropy of crystal deformation, possibility of phase transitions and solid-state chemical reactions. Information about impact of external influences on the crystal structure allows understanding how changes in external conditions affect the course of chemical reactions. Also this information helps to understand relationships between anisotropy of deformation of the structures and noncovalent interactions.

Convenient objects for studying such solid-state transformations are cobalt coordination compounds $[Co(NH_3)_5NO_2]XY$. In such compounds a mechanical response during the photoisomerization reaction $[Co(NH_3)_5NO_2]XY \rightarrow [Co(NH_3)_5ONO]XY$ is observed[1-3]. The convenience of using such cobalt complexes for studying the relationship «composition-structure-properties» arises from the fact that in most of them no destruction of the crystal is observed during the photoisomerization reaction. This allows solving their crystal structure and geometry of intermolecular interactions using X-ray diffraction analysis and also comparing changes arising from the photoisomerization reaction and from external influences for both irradiated and non-irradiated compounds.

Despite said convenience, sometimes it`s rather complicated to study such compounds in laboratory setup, because there is not enough speed of measurement or resolution. For example, study of kinetics of photoisomerization reaction could reveal important details about the course of the reaction and help in understanding the relationship between crystal structure and chemical reaction. However, it was shown that reaction proceeds fast, depending on the source of irradiation – one hour or less, which makes it impossible to study it using laboratory X-ray diffractometers. Other obstacle is phase transitions, which occur in several compounds under pressure. In all of the studied compounds phase transitions occur through solid solution, which results in formation of many misoriented crystal domains and sometimes in fracture of the crystal. This is another obstacle to understanding the relationship between intermolecular interactions and anisotropy of deformation, which are connected to the course of the reaction, since it is not always possible to solve crystal structure of a new phase using laboratory X-ray diffractometer.

In this work previously unknown cobalt complexes $[Co(NH_3)_5NO_2]XY$, XY = $BrNO_3$, INO_3, $C_3H_4O_4$, $(NO_3)_2$ and $[Co(NH_3)_5NO_2]_2I_3Cl$ were studied. The goal of this work is establishing the relationship between the possibility of photoisomerisation reaction, the outer-sphere anion, crystal structure and anisotropy of its deformation under external influences. In this work crystallochemical analysis was performed: structural changes during photoisomerisation reaction for studied complexes were compared and main structural factors influencing the course of photoisomerisation reaction and the presence of photomechanical response were revealed. It was shown that all studied compounds react differently to irradiation: for some of them complete isomerization with preservation of the crystal is observed, for some isomerization is accompanied with destruction of the crystal, some crystal show partial isomerization in some crystallographic positions and negative feedback arising in the structure during reaction. Such differences are associated with different intermolecular interactions and crystalline environment of nitro-groups in studied complexes. Voronoi-Dirichlet polyhedral were used for analysis of crystalline environment of the nitro-groups, also special attention was paid for voids and cavities in the structures. Structures of some complexes were studied upon cooling to 100 K and hydrostatic pressure up to 6 GPa in diamond anvil cells. For $[Co(NH_3)_5NO_2]BrNO_3$ a phase transition was observed at pressure of 3 GPa, when pressure was removed, reverse phase transition occurred. In the $[Co(NH_3)_5NO_3]C_3H_4O_4$ complex phase transition also was observed approximately at 3 GPa, during this phase transition new intermolecular interactions formed due to a significant shrinkage of the structure and some changes in coordination of malonate-anions. Moreover, for all studied complexes some anomalies in changes of unit cell parameters upon cooling were observed. These anomalies were not associated with phase transitions; the absence of phase transitions upon cooling was further confirmed by DSC-analysis.

Acknowledgement: This work was supported by Russian Science Foundation, grant № 24-22-00293, https://rscf.ru/en/project/24-22-00293/.

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PP-19

The system for detecting X-ray spectra using a detector based on microchannel plates

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Detectors based on microchannel plates are widely used in laboratory X-ray sources, but also in synchrotron sources. They have not only high spatial resolution and efficiency, but also a long operating time. At the same time, they have a relatively low cost [1].

This work presents an X-ray image registration system including a two-dimensional detector, camera and software [2]. Various detector and camera options were considered. Cameras vary in resolution, photosensitivity and the presence of cooling. The main difference between the detectors is the degree of amplification of the original signal, otherwise the design has not changed. The influence of the presence of defects on the quality of the received data is demonstrated.

Acknowledgement: This work was supported by the Russian Science Foundation, grant 23-72-00067.

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PP-20

Electronic structure of La0.5Pr0.2Ca0.3MnO3 single crystal manganite using high resolution synchrotron radiation resonant photoemission spectroscopy

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The work is devoted to the study of the electronic structure of the multicomponent manganite compound La0.5Pr0.2Ca0.3MnO3 by resonant photoemission spectroscopy (RPES).

Manganite unique characteristics allow us to consider them as promising functional materials with giant magnetostriction, large magnetoresistance and magnetocaloric effect [1-3]. The study of the valence band of compounds containing more than four elements is difficult due to the fact that there is an overlap of energy bands. Therefore, a method with sufficient resolution for a detailed study of the valence band is needed.

The method of RPES It allows us to determine the partial contributions of electronic states in the valence band, which is very important in the study of the electronic structure of multicomponent systems. Analysis of the data obtained in the resonance mode will be useful for calculations of the electronic structure of multicomponent systems containing 3d elements.

Acknowledgement: This work was supported by the Russian Science Foundation, grant 23-72-00067.

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OP-21

Investigation of the effect of copper content on the atomic and electronic structure of Cu-Si ion beam films by USXES and XPS methods

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Cu-Si nanocomposite films have a wide range of technological applications in microelectronics, catalysis, silicon bronze technology, and the production of a new type of lithium-ion batteries. A number of studies have reported that copper deposited on a silicon substrate contributes to the intensive growth of the oxide layer at the Cu-Si interface [1]. In addition, a number of phases in the Cu-Si system have still not been largely studied, and the patterns of phase formation vary depending on the ratio of elements and the method of obtaining composite films. Therefore, the study of the effect of copper content on the atomic and electronic structure of Cu-Si films obtained by ion beam sputtering is an important task.

In this work, Cu-Si nanocomposite films of various compositions (Cu ~15, 51, 65 wt. %) were obtained by ion beam sputtering of a composite target in vacuum at a pressure of $1*10^{-5}$ torr on Si(111) silicon substrates of the KDB-12 brand. X-ray phase analysis was performed on a DRON-4-07 $CuK\alpha_{1,2}$ diffractometer (λ = 1.5406 Å). The study of the elemental composition was carried out on a JEOL JSM-6380LV RAM with an INCA 250 microanalysis system. The study of the electronic structure of Cu-Si films was carried out using ultra-soft X-ray emission spectroscopy (USXES) on the RSM-500 monochromator X-ray spectrometer [2] and X-ray photoelectron spectroscopy (XPS) on the SPECS spectrometer using $MgK\alpha$ (1253.64 eV) X-ray source.

According to the results of X-ray diffractometric studies, γ-Cu_5Si phases are formed in Cu-Si films of various compositions (in a film with a low copper content ~15 wt. %) and η-Cu_3Si (in copper-rich films Cu >51 wt. %). In films with a high copper content, Cu ~65 wt. % of the copper is oxidized to form Cu_2O oxide. The presence of Cu_2O copper dioxide in Cu-Si films is confirmed by the XPS Cu *2p* spectra. In addition, according to the XPS data in the Si *2p* spectra, it is seen that some of the silicon atoms are in an oxidized state with an oxidation state of Si^{1+} and Si^{4+}, which corresponds to silicon-oxygen tetrahedra of the Si-Si_3O and Si-O_4 types, respectively. The strong oxidation of silicon may be related to the catalytic properties of the Cu_3Si phase.

X-ray emission Si L2,3 spectra of Cu-Si films of various compositions (Cu ~15, 51, 65 wt. %) were obtained at an analysis depth of 60 nm. The analysis of the phase composition was carried out by computer simulation of experimental spectra. According to the simulation results, in a film with a low copper content ~ 15 wt. % most of the silicon atoms (70%) are in the amorphous silicon phase and about 30% form $SiO_{0.47}$ suboxide. An increase in the copper content in the film leads to an increase in the content of SiO_x phases and to an increase in the degree of oxidation of "*x*" in SiO_x, as well as to the formation of Cu_3Si copper silicide.

	a-Si	SiO0,47	SiO0,8	SiO2	Cu3Si
Cu ~15%	70%	30%	-	-	-
Cu ~51%	40%	15%	-	10%	35%
Cu ~65%	-	-	30%	25%	45%

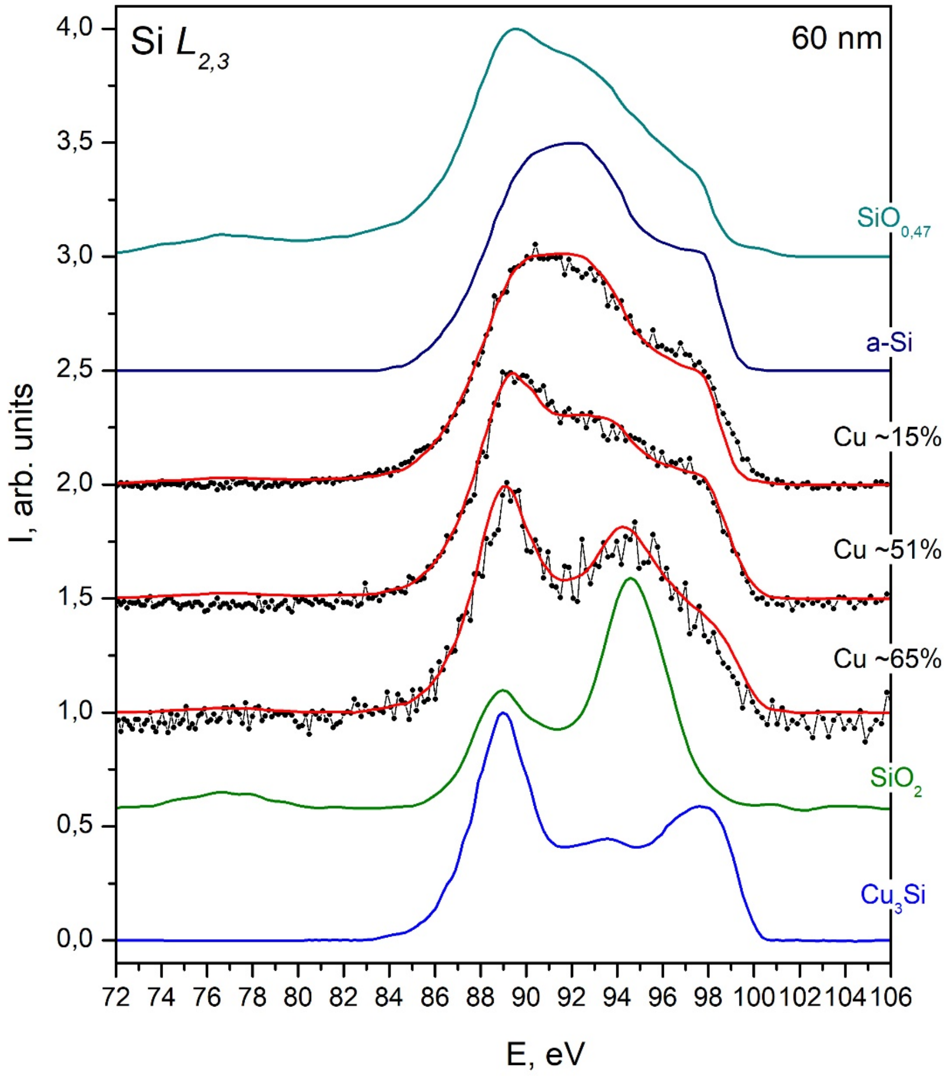

Fig. 1. X-ray emission Si $L_{2,3}$ spectra of Cu-Si films of various compositions (Cu~15,51,65%) obtained at an analysis depth of 60 nm. Simulated spectra (red line). Reference spectra of $SiO_{0,47}$, SiO2, a-Si u Cu_3Si.

Acknowledgement: This work was funded by the Russian Science Foundation, under grant number 23-79-10294, https://rscf.ru/project/23-79-10294/.

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PP-22

Comparison of elastic properties of polymorphic glycine modifications by DFT calculations

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Quantum chemical modeling makes it possible to study various properties of solids, such as elasticity, brittleness, and piezoelectricity. Approaches of density functional theory (DFT-density functional theory) and experimental X-ray diffraction methods simultaneously allows to obtain the most complete information about the relationship between the structure and properties of crystals. Synchrotron radiation sources utilization makes it possible to obtain the most precise structural data that can be used as a starting point for DFT calculations. In the case of molecular crystals, namely glycine, elastic properties are associated with changes in various non-covalent intermolecular contacts, for example, hydrogen bonds or van der Waals interactions.

The purpose of this work is to study the relationship between changes in macroscopic elastic and piezoelectric properties and the behavior of the electron continuum (microscopic properties) in the intermolecular space for all currently known polymorphic modifications of glycine (α, β, γ, ζ), including high -pressure phases (β’, δ).

Wave functions of crystal structures (α, β, γ, ζ, β’, δ) of polymorphic modifications of glycine were obtained using the Kohn-Sham method, taking into account their translational symmetry. The results of neutron powder diffraction were used as initial data. Elasticity tensors and, for non-centrosymmetric phases, piezoelectricity tensors are calculated. The regularities between the features of the distribution of electron density in the intermolecular space and the functional effect of various polymorphic modifications are revealed.

The work was carried out under the scientific supervision of Dr. Prof. Boldyreva E.V. and under the technical supervision of Khainovsky M. A.

Calculations were carried out using the resources of the computing cluster of the NSU Computing Center (NSU ICC).

PP-23

Computation, synthesis, and crystal structure investigation of new tecovirimat analogies

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The smallpox virus causes the disease known as smallpox, which was one of the most feared infections in human history, but has been eradicated through vaccination and global eradication efforts. However, the threat of its re-emergence remains, and research continues to prevent potential outbreaks. The p37 protein of the smallpox virus is an important element that contributes to the pathogenesis of infection and the interaction of the virus with the host immune system. Inhibition of this protein stops the development of the virus in the cell and reduces the viral load on the human body [1].

One of the modern antiviral drugs developed for the treatment of smallpox infections is tecovirimat. It inhibits p37 protein needed for viral replication. This prevents the virus from reproducing and spreading throughout the body. Recently, it has been approved in some countries, including the United States, to combat the threats posed by viruses. Despite its effectiveness in treating infections caused by smallpox viruses, it has several disadvantages, namely high cost of the drug and low solubility in water and low oral bioavailability [2].

In this work structures of 9 new structures (that are similar to tecovirimat) were predicted. Molecular docking allowed to determine how a smalls molecule binds to a target protein (p37 smallpox protein) and the energy of complexes. Obtained complexes were used for molecular dynamic simulation 100 ns.

The predicted leader compound was synthesized by following scheme (Fig.1):

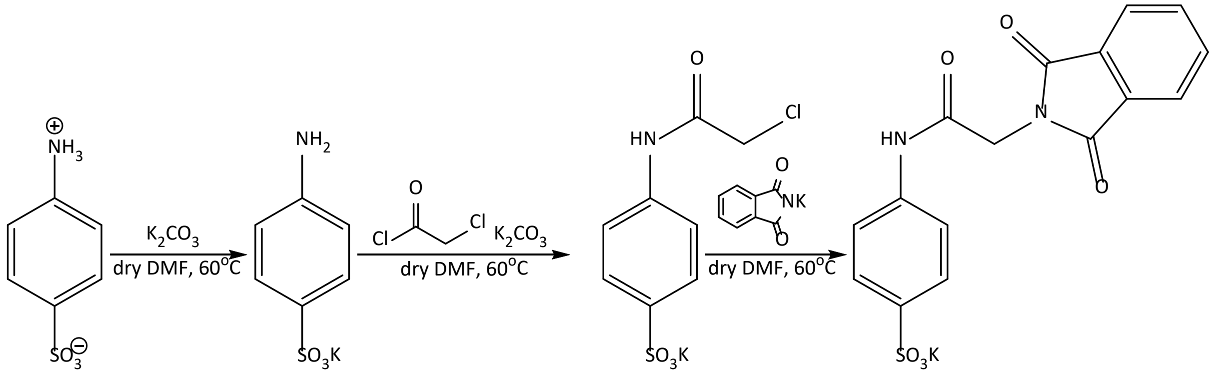

Fig. 1. Scheme of preparation of potassium 4-(2-(1,3-dioxoisoindolin-2-yl)acetamido)benzenesulfonate

Obtained compound was crystalized from methanol/2,2,2-trifluroethnol in 1:1 rate. Needle crystal was used for XRD analysis. In order to influence the crystal habit, the potassium ion was replaced by hydrophobic organic cations - tetraethylammonium and tetrabutylammonium (Fig.2).

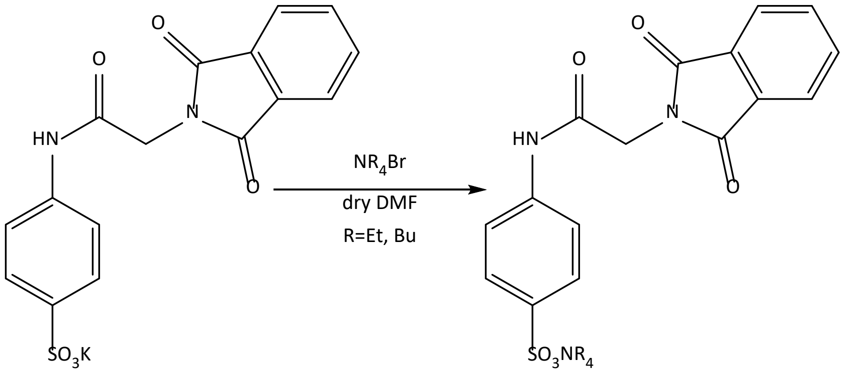

Fig. 2. Scheme of preparation of quaternary ammonium salts of leader compound

Crystals of tetraethylammonium were obtained by slow evaporation saturated water solution, crystals of tetrabutylammonium were obtained by slow evaporation or slow cooling hot dimethylformamide solution. Prism crystal was used for XRD analysis.

Acknowledgement: This work was supported the Ministry of Science and Higher Education of the Russian Federation within the governmental order for SRF SKIF Boreskov Institute of Catalysis (project FWUR-2024-0040).

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PP-24

Development of the first-priority beamlines and associated engineering infrastructure of the synchrotron radiation facility 'SKIF': experience of cooperation network formation and efficient coordination

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Construction of the 4th-generation synchrotron radiation source SKIF in the science city of Kol'tsovo in the Novosibirsk region is a complex large-scale project with no comparable analogues in Russia for several recent decades. Its successful accomplishment requires concerted efforts of many institutions.

Presently, the project enters the final stages of its implementation. Civil construction works at the construction site in Kol'tsovo are underway. Most out of 30 buildings are at a high level of constructive readiness. BINP (Budker Institute of Nuclear Physics) specialists have started assembly of the linear accelerator equipment in the Injector building. Technological equipment of six first-priority beamlines is being manufactured with the readiness degree above 70% (as of September, 2024) and scheduled date of 100% readiness by the end of November, 2024. Four institutions, viz., Tomsk Polytechnic University, Institute of High-Current Electronics SB RAS, Lavrentyev Institute of Hydrodynamics SB RAS, and Lavrentyev Institute of Hydrodynamics SB RAS, coordinate the works on the design, manufacture, assembly, and commissioning of six first-priority beamlines of SKIF. In total, 14 state contracts are concluded to put the beamlines into operation. And the number of subcontracts and thus the number of diverse institutions from all around Russia and friendly foreign countries involved in the beamline development process exceed 100. In addition to main scientific equipment, the beamlines should be equipped with technical supply systems (cooling water, compressed gases, electricity, etc.) as well as with signal and control circuits (entrance and access control, machine safety, fire safety, and so forth). The beamline subsystems should be seamlessly integrated into the respective systems of the storage ring building. This requires additional efforts for coordination and reconciliation of technical documentation and equipment specifications.

This report summarizes our positive and negative experience on all aspects devoted to the SKIF beamline creation. Such a critical analysis is crucial for the next stage of the SKIF development regarding second-priority beamlines. It could be also of importance for other synchrotron projects in Russia.

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PP-25

Synchrotron In situ investigations of multilayer Cr/Ta and Cr/Mo coatings on Zr-1Nb alloy, subjected to LOCA conditions

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The main material of fuel elements of modern water-water reactors is zirconium-based alloys. This choice of material is caused by the properties of zirconium alloys, especially their corrosion resistance and low thermal neutron absorption cross-section. Despite all the advantages of such alloys, an important disadvantage is their possible uncontrolled exothermic reaction with water vapor in accidental conditions, which results in the formation of explosive hydrogen [1].

To prevent or at least slow down this reaction, many scientific groups have proposed an approach involving the application of barrier coatings to fuel cells. The most common coating material is chromium. The oxidation of chromium results in the formation of Cr_2O_3 oxide film, which acts as a diffusion barrier [2]. In addition, chromium has good mechanical and adhesion properties. The only, and at the same time critical, disadvantage of chromium coatings is the interdiffusion of chromium and zirconium at high temperatures, which leads to increased diffusion rates into the zirconium alloy [2].

One of the ways to solve this problem is to develop a new type of chromium-based protective coating with an intermediate barrier layer preventing Cr-Zr mutual diffusion [3]. It is important that the material of this sublayer has no influence on the reactor economy and has a small thermal neutron absorption cross-section [4,5]. In addition, this material should not form compounds with either chromium or zirconium. A number of studies suggest that refractory metals are promising materials for the role of the intermediate layer. For example, tantalum has a relatively small thermal neutron absorption cross section, as well as a small solubility limit in betta-zirconium. Meanwhile, with a molybdenum thickness of 12-15 µm, using a protective coating thicker than 100 µm was found to effectively prevent mutual diffusion at 1200 °C for several tens of minutes [6]. However, such thick coatings would severely reduce the reactor economy. However, until now, the oxidation behavior of the Cr/Ta- and Cr/Mo- coated Zr-1Nb alloy has not been sufficiently investigated.

High temperature oxidation of Zr-1Nb alloy samples with Cr/Ta and Cr/Mo coatings was carried out under LOCA conditions. A comparative analysis of samples with Cr/Ta and Cr/Mo bilayer coatings was carried out on the basis of the data obtained from weight gain, SEM, XRD and in situ XRD. In the case of the Cr/Ta coating, a short-term prevention of Cr-Zr interdiffusion is observed. The Cr_2Zr phase is formed after two minutes of oxidation at 1250 °C according to in situ XRD (Fig.1, left). The interdiffusion of Cr and Mo is less pronounced as the Cr_3Mo phase was not observed during in situ XRD studies (Fig.1, right). The formation of Cr_3Mo phase was found only after HT oxidation. According to SEM analysis the thickness of the Cr_3Mo layer increased with oxidation time. During the investigations it was found that enhanced oxidation of the Mo-Zr layer can cause local oxidation of the coated Zr alloy samples when oxygen penetrates up to the Mo layer/Zr alloy interface.

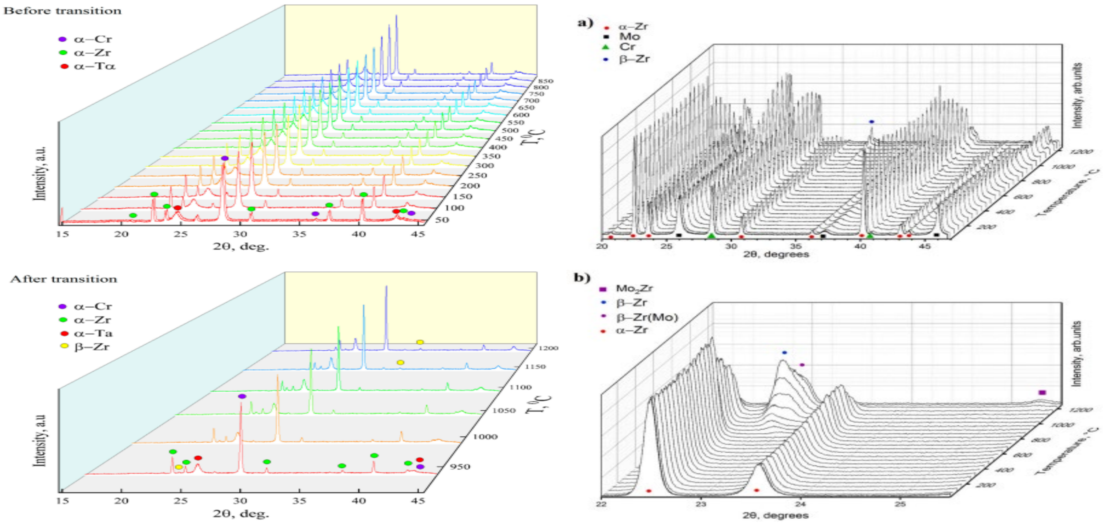

Figure 1. Diffraction patterns 0f Cr/Ta (left) and Cr/Mo (right) coated zirconium alloy during linear heating from room

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PP-26

Synthesis and structural elucidation of Pu, Ce, and Th double phosphates using synchrotron radiation

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The widespread use of atomic technologies has made the issues surrounding the disposal of radioactive waste a concern for nearly every country today. One of the promising methods for the long-term management of radioactive waste is deep geological disposal in stable geological formations, utilizing both engineered and natural barriers. To ensure the safety of disposal sites, it is essential to understand the migration behavior of radionuclides at a molecular level. Phosphate anions and alkali metal cations are integral components of natural environments. Therefore, the investigation of the structure and properties of double phosphates of actinides is one of the key tasks.

The chemical behavior of Pu is complex as it possesses seven oxidation states, four of which (Pu(III), Pu(IV), Pu(V), and Pu(VI)) may coexist under environmental conditions and can easily transform from one to another depending on the conditions. Here, we investigate the formation patterns of Pu-containing particles in aqueous media, focusing on their interactions with vital inorganic phosphate ligands. Given Pu complex redox chemistry and high radiotoxicity, our study also includes their less radioactive monovalent thorium (Th) analogue and non-radioactive cerium (Ce) for comparison. We aim to establish the relationship between actinide particle formation conditions, size, structure, and thermodynamic properties.

A traditional method for determining the structure of compounds is X-ray diffraction (XRD). Synchrotron X-ray diffraction allows for the investigation of small quantities of substances, which is particularly important in the context of working with radionuclides. In addition to X-ray diffraction, X-ray absorption spectroscopy (XAS) enables the determination of the oxidation state of actinides as well as their local environment. Together, these methods provide comprehensive data on the structure of double phosphates of actinides, allowing for a thorough description of new compounds that were previously unknown in the literature.

Double phosphates of Pu, Th and Ce were synthesised by by aging process of PuO_2, ThO_2 or CeO_2 nanoparticles in Na_2HPO_4/NaH_2PO_4 media for up to 4 years. Additionally, the dioxide's aging process was studied under mild hydrothermal treatment (HT) conditions (< 150 °C) in K_2HPO_4/KH_2PO_4 or $(NH_4)_2HPO_4/NH_4H_2PO_4$ solutions.

The structure features and phase composition of the samples were investigated by synchrotron XRD and absorption spectroscopy (XAS) and electronic microscopy (SEM and HRTEM). Under long-term storage and HT treatment conditions in a phosphate buffer medium, PuO_2, CeO_2, and ThO_2 nanoparticles are crystallized into the phosphates phase. Their second-generation phase structure depends on the pH value and the composition of the initial phosphate buffer. The HT treatment of dioxides in a NaH_2PO_4/Na_2HPO_4 buffer solution leads to the formation of a nanocrystalline Ce(IV) phase. In contrast, thorium and plutonium generate a phase characterized by the local structure of $NaTh_2(PO_4)_3$. Additionally, Pu(IV) double phosphates with K^+ or NH_4^+ can be synthesized when PuO_2 undergoes HT treatment in potassium phosphate or ammonium phosphate buffers. Using synchrotron radiation techniques enable to decode the structures of $K_2Pu(PO_4)_2$ and $(NH_4)_2Pu(PO_4)_2$ for the first time.

Acknowledgement: This work was supported by the Ministry of Science and Higher Education of the Russian Federation, grant number 075-15-2021-1353.

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PP-27

Development of extrusion parameters of fasteners based on polylactides

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The use of metal fasteners, such as screws, plates, pins, in traumatology and orthopedics is not always justified due to the need for repeated surgery to remove them. In modern medicine, metal products are increasingly being replaced by various biodegradable materials, among which polyesters based on lactic acids are the most effective. Fasteners based on polylactides are absorbed in the body during the process of bone fusion [1,2].

The aim of this work is to study the mechanical characteristics of fasteners obtained with different extrusion parameters to achieve the highest values of mechanical characteristics. For tests of fasteners were varied parameters such as temperature, screw rotation speed, mixing time, casting mold temperature and annealing time. To study the influence of the degree of crystallinity and mechanical properties we measured the mechanical properties using the dynamometer method. According to DSC data, the degree in crystallinity of the obtained fastener samples ranges from 9% to 40%: the higher the mold temperature during casting and the longer the mixing time, the lower the degree of crystallinity. The surface of fasteners was examined by SEM (Fig 1, 2). the surface of the screws is heterogeneous: there are areas with cavities - defects that are the result of incomplete filling of the mold with the melt. No cracks were observed on any of the samples.

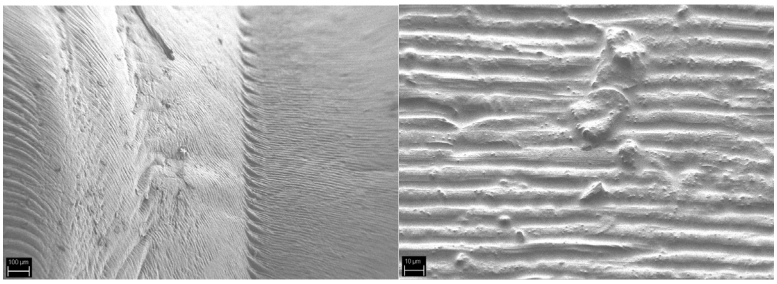

Fig. 1, 2. SEM image of the surface of samples 15 (left) and 16 (right)

Injection molds were created for casting fasteners, the influence of mixing parameters and casting on the structure and properties of screws were studied. The best in terms of obtaining screws without cavities and cracks was the following mixing mode: 50 rpm, 15 min, extruder zones temperature 190-200-210 °C, hammer temperature 180 °C, mold temperature 100 °C and feeding conditions melt: 6 bar, 20 s.

PP-27

Acknowledgement: This work was supported by the Ministry of Science and Higher Education of the Russian Federation, (Agreement 075-10-2021-093).

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PP-28

The influence of N- and Ni-doping on the sensory properties of MoS_2

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Air pollution has become a serious problem all over the world. It is extremely important to monitor the levels of pollutant gases using gas sensors. Currently, metal oxide gas sensors are widely used [1], but they have several disadvantages such as high operating temperature, slow recovery, and deterioration in performance under environmental conditions [2, 3]. Therefore, the development of highly efficient gas sensors operating at room temperature is currently a pressing task.

Recently, 2D layered MoS2 has been reported for high performance gas sensing applications due to their high surface to-volume ratio and excellent electronic properties [4, 5]. Currently, MoS2 is widely studied for the detection of nitrogen dioxide (NO2) at room temperature. But, these sensors have insufficient response, dynamics and selectivity. Doping MoS2 with heteroatoms of nitrogen and transition metals allows changing the physical and electrical properties of MoS2, thereby increasing the sensitivity and selectivity of the sensor material.

In this work, a technique for fabrication of a sensor material from MoS2 powder obtained by thermolysis of (NH4)2MoS4 aerogel in an inert (Ar) or reducing (NH3) atmosphere at 400 °C and 600 °C is developed. The effect of N- and Ni- doping on the sensor properties of N-/Ni- MoS.2 is studied using XPS spectroscopy. The effect of MoS2 synthesis temperature on its structure is also revealed by XPS. MoS2 powders synthesized at 400 °C contain more edge dimeric sulfur compared to samples synthesized at 600 °C. Whereas, the state of Mo in the sample does not depend on the synthesis temperature. In nitrogen-doped N-MoS2-600 powders, electron density is transferred from S and Mo atoms to N built into the lattice. It has been shown experimentally that the introduction of nitrogen heteroatoms improves the sensory responses to NO2 gas, both for sensors made from powders synthesized at 400°C and 600°C. Ni-dosing improves the sensory properties to NH3, and in the case of 600°C samples the sensor becomes selective to ammonia.

Acknowledgement: This work was supported by the Russian Science Foundation, grant 23-73-00048.

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PP-29

A new approach to polymer-assisted mechanochemistry: 3D printed polymer inserts for control of mechanochemical processes

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Mechanochemistry considers chemical transformations under mechanical treatment. Control over the mechanochemical reactions can be carried out in different ways, which include changing the type of mechanoactivator (mill), varying the sample mass-to-ball ratio, applying additives (liquids, polymers), cryogenic grinding, etc. The analysis of the results of mechanochemical experiments can be carried out both in *ex-situ* mode, by interrupting the process and analyzing the sample, and *in-situ* mode using synchrotron radiation sources or Raman spectroscopy. Milling jars for the *in-situ* experiments are often made of different polymers (for example, PMMA), in order to increase the transmittance of X-ray or laser radiation and improve signal-to-noise ratio. However, the effect of the jar wall material on the mechanochemical process was studied only in a few recent publications [1-4].

Here we present the results obtained in a few case studies using a 3D printing to manufacture special inserts placed inside the milling jar, in order to study the effect of various polymer materials on mechanochemical reactions. Polymorphic transitions in glycine and CBZ went faster in a steel container compared to jars with polymer inserts (PETG, PLA, ABS); also, different polymers had different effect on these transformations. The CBZ III → IV transformation was supposed to be faster in steel compared to polymers because the higher hardness of the metal facilitated the amorphization of CBZ III, which is necessary for the subsequent formation of a metastable form IV. Dehydration of CBZ dehydrate, in contrast, was accelerated in containers with polymer inserts, presumably due to the higher hygroscopicity of plastics compared to steel.

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PP-30

Synthesis and investigation of properties of linear block copolymers based on styrene and n- and tert-butyl acrylate

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This work reports on the efficient performance of block copolymers. In recent years, the field of controlled synthesis of macromolecules with tailored molecular weights, topology, composition, and functionality has been actively developing, particularly in the study of self-assembly processes into nanodomain structures [1]. It is known that the morphology of these materials can vary significantly depending on molecular weight, volume fraction of blocks, and interaction parameters between chemically distinct segments. The architecture and sequence of blocks also play a crucial role in the formation of the observed microphase-separated structures. By optimizing these parameters, new functional materials can be produced for various applications in science and technology.

In this work, di- and triblock copolymers were synthesized by varying the temperature and the ratio of reagents using atom transfer radical polymerization (ATRP), and their physicochemical properties were investigated. During the experiments, homopolymers with functional end groups were obtained, which were used as macroinitiators for the synthesis of block copolymers.

To optimize the synthesis methodology, several series of experiments were conducted, varying the temperature from 70°C to 100°C and the quantitative ratio of ligand/catalyst/monomer in the reaction mixture. As a result, successful polymerization reactions were carried out, yielding diblock copolymers PS-P(t)BA-Br, P(t)BA-PS-Br, and ABA-type triblock copolymers based on styrene and n- and tert-butyl acrylate.

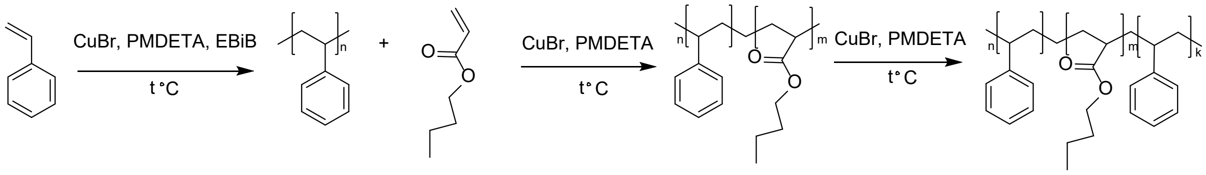

Fig 1.Scheme of synthesis of block copolymer PS-PBA-PS-Br

All samples were analyzed using gel permeation chromatography (GPC). It was shown that, as a result of optimizing the synthesis methodology, samples with a narrow distribution and a polydispersity index below 1.3 were obtained. Additionally, the systems were characterized using infrared spectroscopy, nuclear magnetic resonance (NMR) spectroscopy, differential scanning calorimetry (DSC), and thermogravimetric analysis (TGA). The supramolecular structure of thin films of block copolymers was investigated using small-angle X-ray scattering (SAXS).

In the future, the synthesized diblock and triblock copolymers will be modified through polymer-analogous transformations, which may significantly alter their morphology, physical, and chemical properties. Due to the diversity of the resulting structures and domain sizes, such systems may be promising for applications in lithography or photonics.

Acknowledgement: The authors acknowledge the Russian Science Foundation (grant 23-73-30005).

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PP-31

Thermoresponsive supramolecular nanocontainers from ionic complexes of amphiphilic wedge-shaped mesogens and polybases

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Multi-responsive polymeric nanocontainers attract significant attention for their potential applications in biotechnology, drug delivery, catalysis, and other fields. By incorporating a liquid-crystalline (LC) mesogenic ligand with an alkyl tail length ranging from 8–12 carbons, ionically linked to the polymer backbone, we generate vesicles with walls significantly thinner than those of conventional polymersomes, approaching the thickness of a lipid bilayer. These LC vesicles, ranging in size from 50–120 nm, are designed to be mechanically robust due to the alignment of the hydrophilic polymer backbone within the plane of the vesicle wall. Additionally, incorporating a temperature-sensitive block into the polymer structure imparts thermoresponsiveness to the nanocontainers, enhancing their functionality and adaptability for various applications. Ionic complexes of hydrophilic polybases, specifically poly(2-(dimethylamino)ethyl methacrylate) (PDMAEMA) and PDMAEMA-*b*-PNIPAM (poly(N-isopropylacrylamide)) block copolymers, with amphiphilic wedge-shaped mesogens bearing a sulfonic acid group at the focal point were synthesized. The designed nanocontainers, in the form of either vesicles or nanotubes, exhibit a well-defined wall thickness of 5 nm, dictated by the organization of a smectic LC phase.

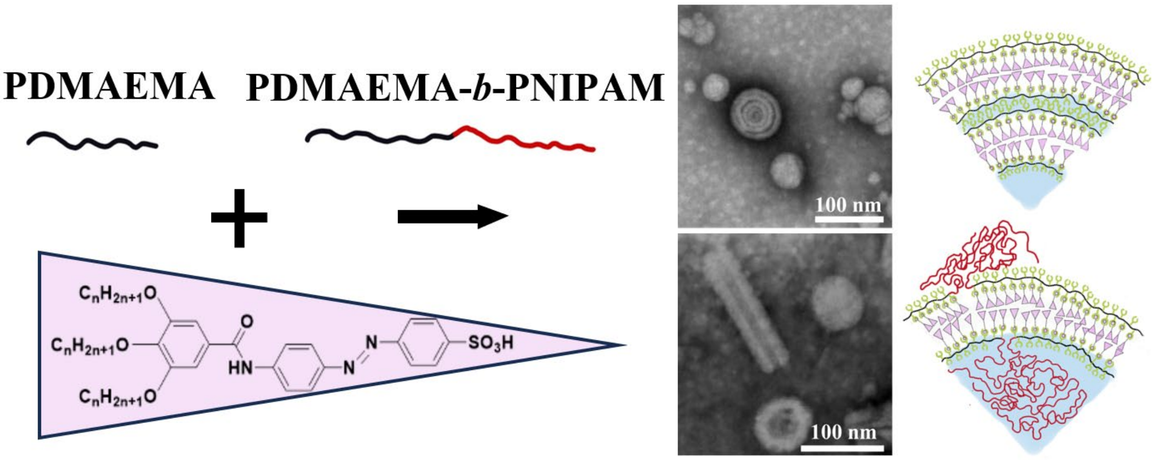

Fig. 1. STEM images of the supramolecular morphologies and schematic illustration of the possible bilayer structure of supramolecular complexes based on homopolymers (top panel) and block copolymers (bottom panel).

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PP-32

Investigation of irradiated and additively coloured CaF_2 single crystals by XPS and Raman spectroscopy

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CaF_2 single crystals are a common material in laser optics for information acquisition, processing and storage [1]. Various methods of modification of the crystal structure are used to extend the optical range of operation and optimize crystal properties. Irradiation with accelerated electrons or additive coloration can be used to form defects in ionic crystals.

In this study X-ray photoemission spectra and Raman spectra of CaF_2 samples before and after modification by additive dyeing and accelerated electron irradiation methods with different irradiation doses were obtained.

The paper discusses the comparison of the results of X-ray photoemission spectroscopy (XPS) of additively colored and irradiated CaF_2 samples. Electron irradiation is some analog of additively coloring and the coloring centers also appear.

The Raman spectra of pure CaF_2 show a characteristic peak at 320 cm^{-1}. For colored and irradiated crystals, the main peak becomes broader with a distinguished shoulder at 329 cm^{-1}. Furthermore, additional modes appear at 140, 217, 280 and 413 cm^{-1}, which may characterize the appearance of corresponding defects in the modified crystals. A colloidal image was also obtained for the additively colored sample by the mapping method.

It was found that irradiation of massive single crystals of CaF_2 by electrons with energy of 10 MeV leads to the formation of defects on the fluorine sublattice. Experimentally, a large covalent contribution to the Ca-F chemical bonding is observed from the XPS spectra of Ca 2*p* and F 1*s*.

Acknowledgement: This work was supported by the Russian Science Foundation, grant 23-72-00067.

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PP-33

Effect of Cu intercalation on the morphology and electronic structure of the $Zr_xTi_{1-x}Se_2$ mixed crystals

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The morphology of the single crystals of the TCh_2-based (T – 3,4d-transition metal, Ch = S, Se, Te) solid solutions appears to be inhomogeneous, with the formation of the structural fragments of varying natures [1-4]. The $Zr_xTi_{1-x}Se_2$ compounds were preliminarily studied using soft X-ray spectroscopic methods including X-ray scanning photoelectron microscopy (SPEM), and the inhomogeneous morphology was found to be typical for their single crystals. At the same time, both the X-ray photoelectron spectra and the parameter of the crystal structure indicate gradual Ti by Zr substitution. Cu intercalation into $Zr_xTi_{1-x}Se_2$ was aimed to understand the difference in the electronic structure of the binary and substitution intercalation compounds.

The electronic structure of the $Cu_{0.08}Zr_{0.07}Ti_{1.01}Se_2$ and $Cu_{0.19}Zr_{0.11}Ti_{1.03}Se_2$ compound has been studied using soft X-ray spectroscopy methods (XPS, XAS, SPEM) at the NANOFES beamline at the KISI-Kurchatov synchrotron facility (Moscow) and ESCA Microscopy beamline at the ELETTRA synchrotron facility (Trieste, Italy). Figure 1 shows the SPEM image acquired at the ESCA Microscopy beamline.

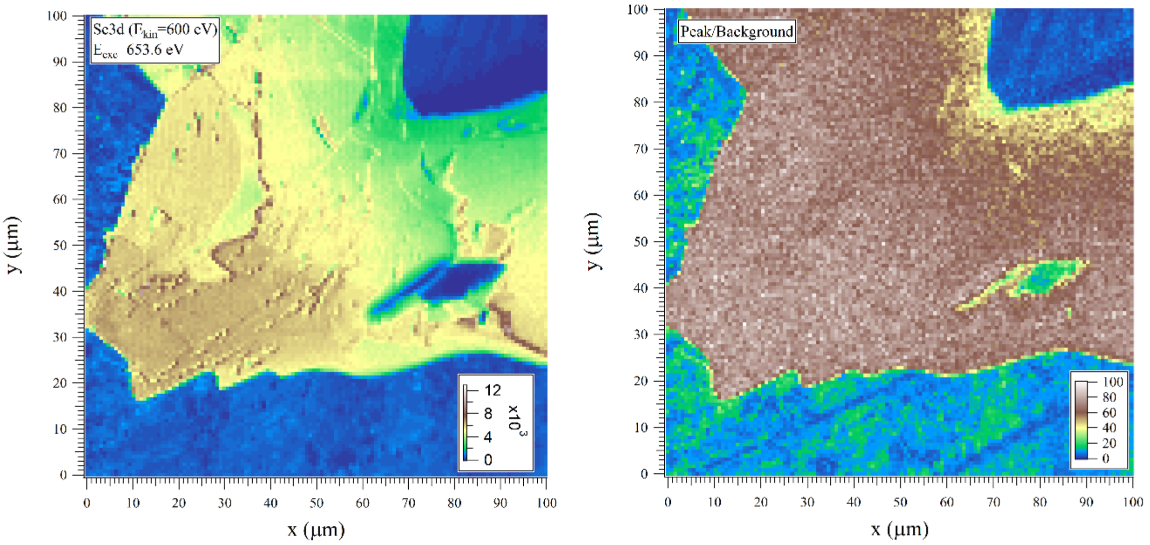

Fig. 1. SPEM image of the $Cu_{0.19}Zr_{0.11}Ti_{1.03}Se_2$ single crystal obtained at area corresponding to the Se 3d core-level spectrum. The gradient scale shows the intensity of the Se 3d line (left panel) and the Se 3d/background ratio (right panel).

Cu intercalation into $Zr_xTi_{1-x}Se_2$ was found to increase the covalent contribution to the chemical bonding of Ti with the environment. The positive shift of the core levels to the higher binding energy implies the increase in the Fermi energy due to the charge transfer from the Cu atoms to the lattice. At the same time, the shift differs for different elements, which implies additional changes in the electronic structure. The additive character of the Cu $L_{2.3}$ XAS spectrum indicates the occupation both tetrahedral and octahedral crystallographic sites in the lattice. The increase in the Cu concentration results in the formation of the Cu/Ti/Zr hybrid bands. This hybridization results in the destruction of the structural fragments and homogenization of the material.

Acknowledgement: This work was supported by the Russian Science Foundation, grant 24-23-20136.

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PP-34

Band structure features of the (111) $Pb_{1-x}Sn_xTe$:In/BaF_2 films surface studied by angle-resolved photoemission spectroscopy

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The band gap energy value of a rocksalt cubic lead-tin telluride $Pb_{1-x}Sn_xTe$ varies strongly with the tin content x and temperature [1]. At room temperature it decreases from 0.3 eV for PbTe to 0 eV at x ~ 0.35 and then increases again to -0.3 eV for SnTe. At x > 0.3, due to the strong spin-orbit interaction, the inversion of bands order occurs and a topological crystalline insulator (TCI) phase is formed, followed by the appearance of spin-nondegenerate Dirac surface states. These states have a Dirac cone dispersion with helical spin structure, so the spin-polarized charge carriers are protected from backscattering by the time-reversal symmetry. Thus, topological insulators are promising materials for realizing spintronics devices, storing and working with quantum information. The real band structure of $Pb_{1-x}Sn_xTe$ depends on many factors, including effects of surface reconstruction, external doping and strain, but is still poorly investigated experimentally. The aim of this work was to study in detail the band structure features of the (111) surface of indium doped lead-tin telluride films with compositions close to the band inversion epitaxially grown on BaF_2 substrates.

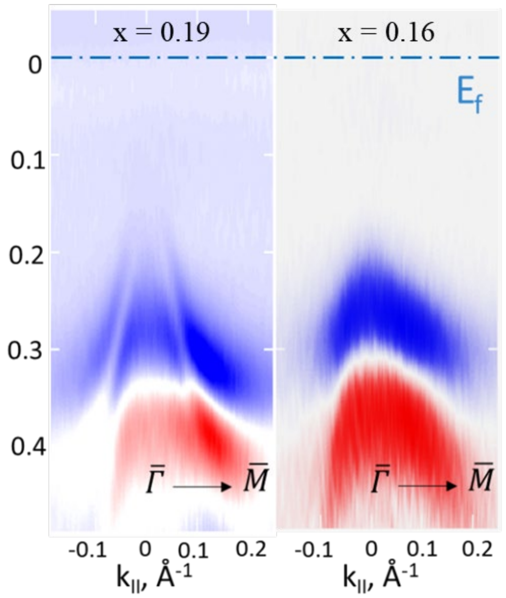

Fig. 1. The second derivative $\frac{d^2I}{(dE)^2}$ of the ARPES spectra, measured on (111) $Pb_{1-x}Sn_xTe$ surface for samples with x=0.19 and x=0.16 at T=77 K.

The (1×1) reconstructed surface of (111) oriented $Pb_{1-x}Sn_xTe/BaF_2$ epitaxial films with compositions in the range of x ~ 0.16 – 0.5 was investigated using angle-resolved photoemission spectroscopy, X-ray photoemission spectroscopy and low-energy electron diffraction. The measurements of the electronic structure of the sample surfaces were performed using SPECS ProvenX-ARPES system in the temperature range of 77 – 300 K. In order to obtain the structurally ordered and atomically clean surface of the studied films prior to the ARPES measurements, a chemical treatment including HCl-isopropanol dipping to remove natural oxides and subsequent annealing in UHV was applied [2].

It was found that at T = 77 K the transition of $Pb_{1-x}Sn_xTe$ from the trivial insulator phase to the TCI phase with the appearance of topological surface states occurs in composition range from x = 0.16 to x = 0.19 (see Fig.1). At the same time, a gapped TCI surface states are observed at higher temperatures for films with x > 0.19. Along with the energy difference of the valence band maxima in the G- and M-points of the surface Brillouin zone this indicates the presence of tensile stresses in the studied structures plane. These mechanical stresses may arise due to the difference in the thermal expansion coefficients of the PbSnTe films and the BaF_2 substrate. As a result, (111) $Pb_{1-x}Sn_xTe$ transitions from the weak topological insulator phase to the strong topological insulator phase.

It was found, that annealing of $Pb_{1-x}Sn_xTe$:In films at the temperatures above 350°C leads to diffusion of indium from the films volume to the surface with the formation of a hexagonal InTe tetra-layer, which is expected to reveal superconducting properties [3]. In this case, reconstruction of the surface occurs with the formation of a moire (7×7) superstructure and an electronic structure is formed that combines the states of the original surface (111) $Pb_{1-x}Sn_xTe$ and the InTe layer [4].

Acknowledgement: This work was supported by the Priority-2030 program.

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PP-35

Experimental and theoretical XANES study of UOx and Np oxide systems at the L_3 edge

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X-ray Absorption Spectroscopy (XAS) is currently one of the leading methods for studying the structure of substances and electronic states. Both structural and electronic properties of actinides are of fundamental interest for describing intra-molecular interactions. Using laboratory spectrometers, it is possible to obtain not only information about the oxidation state of the studied materials but also structural. (Fig. 1)

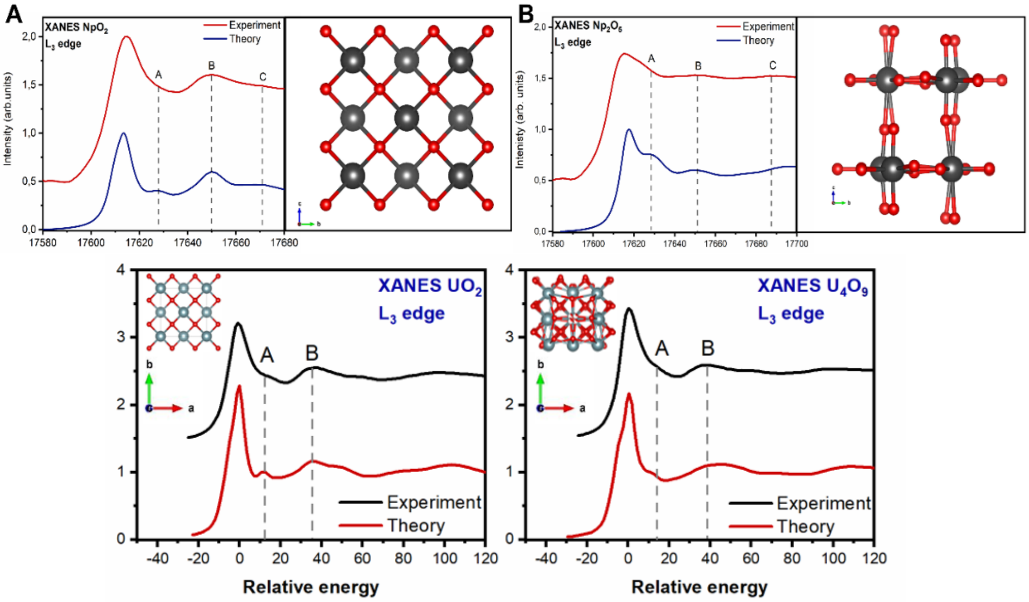

Figure 1. *Experimental and theoretical X-ray absorption spectra of U and Np L_3 edge*

Using theoretical approaches to model XANES spectra of various uranium oxides allows for the estimation of local density of electronic states (DOS). DOS helps identify potential transitions and spectral lines observed in experiments, which is crucial when considering the behavior of U and Np systems and their potential use as nuclear fuel for energy production.

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OP-36

Investigation of crystal structure and morphology of thermoplastic polyurethanes with shape memory effect under thermomechanical treatment

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Polymers with shape memory effect (SME) demonstrate change of dimensions and mechanical properties under various factors, have attracted the attention of scientists in recent years. Such materials are the basis for design of “smart” medical devices, such as plasters, orthodontic arches, heart patches, etc [1-3]. Semi-crystalline polyurethanes and composites based on them are the example of such thermo-responsive SME-materials. The key advantages of them are biocompatibility, processibility, good thermal and mechanical characteristics, as well as the ability to customize properties for a particular application.

Polyurethane is a linear block copolymer built of alternating soft and rigid blocks, the composition of which has a direct influence on the SME characteristics. The aim of this work is to study the mechanical behavior of a series of multi-block thermoplastic polyurethanes differing in soft block structure.

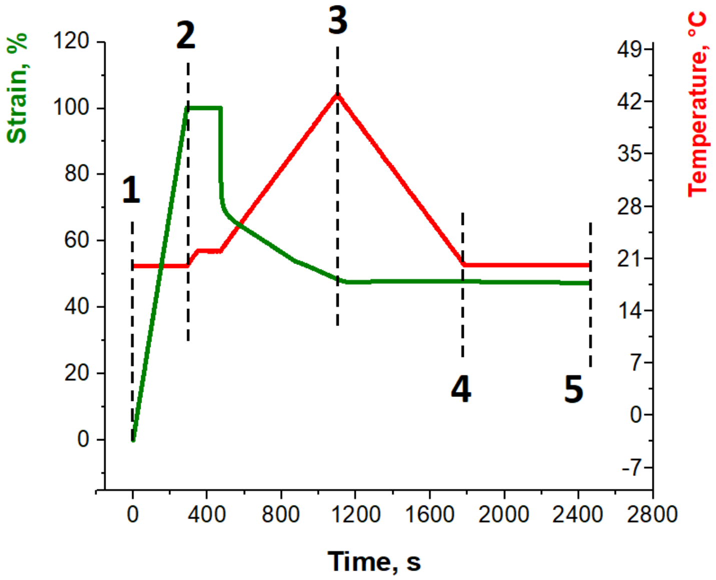
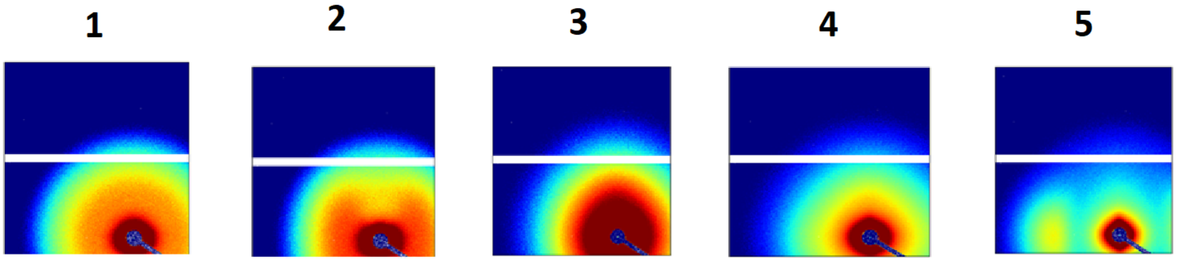


Fig. 1. Variation of shape during thermomechanical experiment of the studied polyurethanes with the key points: initial state (1), uniaxial drawing to 100% (2), heating of free-standing film to 43 °C (3), cooling to RT at zero stress (4), crystallization at RT (5)

OP-36



ε = 0%, T = 21°C

ε = 100%, T = 21°C

ε = 52%, T = 43°C

ε = 46%, T = 21°C

ε = 46%, T = 21°C
after 20 minutes

Fig. 2. 2D SAXS patterns of the studied polyurethanes in key points of the thermomechanical experiment

Using dynamic mechanical analysis, differential scanning calorimetry, IR-spectroscopy, and synchrotron small- and wide-angle X-ray scattering during deformation and heating, we have investigated the relationship between the SME and structure and morphology of polyurethanes. It is shown that the main stages of SME: fixation of temporary shape, switching of mechanical properties and restoration of permanent shape are determined by phase separation processes as well as by the degree of crystallinity of hard and soft blocks. The samples show high Young's modulus (up to 200 MPa), a shape fixing ratio of 80% and a strain recovery ratio of 50-60%. The mechanical properties depend on the density of the hydrogen bonding network in the phase-separated domains of the hard blocks. The processes of stress and strain relaxation are also investigated, correlation with the crystallization kinetics of soft block is shown, and the SME phenomenon is studied during several cycles of deformation and shape recovery. The results obtained allow us to better understand the behavior of materials with complex mechanical response and optimize their chemical structure and fabrication conditions.

Acknowledgement: This work was supported by the Ministry of Science and Higher Education of the Russian Federation, (Agreement 075-10-2021-093)

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PP-37

High-coercivity ferrimagnet $Co_2FeO_2BO_3$: XMCD insights into charge-ordering and cation distribution

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The transition metal oxyborates with the ludwigite crystal structure (space group *Pbam*) have been extensively studied due to their diverse electrical and magnetic properties. However, the local electronic and magnetic characteristics of transition metals in these crystals remain largely unexplored. In this study, we report XAFS/XMCD investigations on $Co_3O_2BO_3$ and $Co_2FeO_2BO_3$ oxyborate single crystals at the *K*- and $L_{3,2}$-edges of cobalt and iron. These compounds exhibit ferrimagnetic behavior with uniaxial anisotropy: $Co_3O_2BO_3$ (T_C = 42 K) and $Co_2FeO_2BO_3$ (T_{N1} = 115 K, T_{N2} = 70 K), showing a remarkable increase in coercive field [1].

XAFS/XMCD experiments were performed at several beamlines (ID12 at ESRF, UE46_PGM-1 at BESSY II, K1.3b at NRC KI) under high magnetic fields and low temperatures. By comparing the XMCD results with bulk magnetizations, element- and orbital-selective magnetizations of Co^{2+} and Fe^{3+} ions were determined [2,3]. The Co^{2+} ions exhibited negative element-selective magnetizations, while Fe^{3+} ions showed positive values, maintaining an antiferromagnetic interaction between them even in the paramagnetic phase. Interestingly, the field dependence of the XMCD signals did not replicate the macroscopically observed high coercive field.

A tentative explanation suggests that magnetic moments in the Co and Fe sublattices are ordered antiparallel, with a skewing of the Co sublattice. The potential correlation between bulk and element-selective magnetizations is discussed.

Acknowledgement: This work was supported by the Ministry of Science and Higher Education of the Russian Federation (Agreement No. 075-15-2022-263). The investigations were carried out using large-scale research facilities such as the "EXAFS Spectroscopy Beamline" at the Siberian Synchrotron and Terahertz Radiation Center in Novosibirsk, Russia.

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PP-38

Synthesis and research of wedge-shaped molecules based on gallic acid for the production of self-organizing complexes

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The complexation of polymer chains by low molecular weight amphiphiles (mesogens) through non-covalent interactions is an effective method for creating nanostructured macromolecular materials. Obtaining new mesogens that serve as building blocks for the synthesis of new functional materials, as well as establishing the relationship between their structure and the adaptive properties of nanoobjects, is an important task for creating new ion-conducting membranes, drug delivery systems into the cell, and others.

During the experiments, a series of wedge-shaped mesogens based on gallic acid 3-hydroxy-4,5-octanoxy-N-(pyridine-4-ilmethyl) was synthesizedbenzamide and 3,4,5-dodecoxyoxy-n-(pyridine-4 ilmethyl)benzamide.

At the first stage, alkyl tails were "sewn" using a weak K2CO3 base in order to avoid premature hydrolysis of the ester and bind excess water in the reaction mixture.

In the second stage, alkaline hydrolysis of the ester was carried out under intensive boiling in the presence of a 10-fold excess of KOH in ethanol.

During the third stage, aminolysis of the obtained acids was carried out using the 1-ethyl-3-(3-dimethylaminopropyl) catalytic systemcarbodiimide (EDC) and 4-dimethylaminopyridine (DMAP).

The structure of the compound and the absence of impurities of the obtained compounds were confirmed using 1H and 13C NMR, IR spectroscopy, and elemental analysis. The dependence of the structural-phase state on temperature was also studied using synchrotron radiation.

Acknowledgement: The work was carried out with the financial support of the Ministry of Science and Higher Education of the Russian Federation (Agreement No. 075-15-2022-1117 dated June 30, 2022).

PP-39

Determination of 3*d*-4*f* electronic states contribution to the valence band of $GdCo_2Ni_{0.1}$ rare earth intermetallic compound by resonant photoemission

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The non-stoichiometric intermetallic RCo_2Ni_x compounds with Laves phase structure of the $MgCu_2$-type (space group $Fd\bar{3}m$) attract attention with their changing magnetic properties depending on transition metal amount and the choice of rare earth element, which allows expanding the scope of their application [1-2]. In addition to the magnetic properties, the electronic structure also changes, which currently requires clarification, in particular, the contribution of the rare earth *f*-states to the valence band (VB).

The electronic structure of $GdCo_2Ni_{0.1}$ compound with Laves phase structure was studied by resonant X-ray photoemission spectroscopy (RPES) using synchrotron radiation at the NANOPES beamline of the Kurchatov accelerator-storage complex of a specialized synchrotron radiation source (“KISI-Kurchatov”). Using synchrotron radiation as a source of photons for recording photoemission allows one to select the exact excitation energy (photon energy range 25-1500 eV) and the high beam intensity allows one to obtain high-resolution spectra. To determine the excitation energies near the Co $L_{2,\,3}$, Ni $L_{2,\,3}$, Gd $M_{4,\,5}$ and Gd $N_{4,\,5}$ edges of $GdCo_2Ni_{0.1}$, the corresponding absorption spectra were taken.

RPES makes it possible to study not only the ground state, but also the lifetime of the excited state (a core level-hole – VB electron), determine the energies of VB single-particle states and two-hole states on selected atoms see reactions to the sudden appearance of the core-level photo-hole. Resonant photoemission in narrow-gap materials was described by the sum of first- and second order transitions, their quantum-mechanical interference leads to an increase in the spectrum from the VB and the appearance of an asymmetric dependence on the photon energy. Recently, these effects were studied experimentally on the example of ternary intermetallic compounds with nickel as the main transition metal $ErNi_2Mn_x$ [3] and $DyNi_2Mn_x$ [4]. The competition between the elastic and inelastic photoemission channels leads to different dependences of the photoemission spectra in our case from cobalt and nickel on the photon energy. The appearance of the second transition metal and its concentration affect the intensity of the rare earth peaks in the VB, which confirms the need to determine *d-f* interactions in such systems.

Acknowledgement: This work was supported by the Russian Science Foundation, grant 23-72-00067.

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PP-40

Thermal stability of $CuCrSe_2$

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Modern research into multiferroic materials has significantly advanced materials science. These materials exhibit ferroelectric order simultaneously with magnetic order. By increasing the ordering temperature and enhancing the coupling between the electric and magnetic systems, we can find practical applications for these materials.

One promising material that shows potential in this area is $CuCrSe_2$. It has a structure similar to delafossite ($CuCrO_2$), which can be described as a lattice of $CrSe_2$ layers intercalated with copper. However, unlike delafossite, in $CuCrSe_2$, the copper occupies a specific set of tetrahedral positions shifted from the center of the interlayer gap, rather than being evenly distributed over octahedra in the interlayer space.

The $CuCrX_2$ lattice has no center of symmetry and is prone to ferroelectric ordering. The magnetic subsystem is made up of layers of chromium atoms arranged in a triangular lattice. The magnetic interaction between chromium atoms within each layer is strong, while the interaction between chromium layers is weak. This results in a spiral magnetic ordering with an incommensurate wave vector. The temperature at which the magnetic ordering occurs coincides with the temperature at which the electrical ordering occurs, making these materials considered multiferroics.

One of the problems that arise when working with such materials is the emergence of a secondary spinel phase during synthesis. The influence of this phase greatly affects the properties of the overall material, significantly distorting the data from the main layered phase.

In this work, we investigated the crystal structure of $CuCrSe_2$ by heating in situ in the temperature range of 25-975 ^oC. The high-temperature diffraction experiment was performed at the MCX beamline [1] of the Elettra synchrotron.

The temperature of the spinel phase formation was determined, and a complete profile refinement of the crystal structure was carried out. A fragment of the phase diagram for $CuCrSe_2$ was constructed for the temperature range 25-1000 °C.

Acknowledgement: This work was supported by the Russian Science Foundation grant No. 22-13-00361.

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PP-41

XPS-based restoration of partial densities of valence states of chalcogenides using Bayesian inference

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Information about the partial densities of valence states (PDOS) of materials plays an important role in understanding their physical properties. A synchrotron radiation source can be used to "illuminate" the different partial (orbital) states of atoms in XPS (X-ray photoelectron spectroscopy) spectra of valence band. The synchrotron radiation source allows the energy of the X-ray photons incident on the sample to be gradually varied, thereby obtaining XPS spectra at different X-ray photon energies. However, the resulting set of XPS spectra often provides only a rough estimate of the energy distribution of the valence states. In this work, we propose a method using statistical Bayesian inference that can be applied to restore the valence PDOS of chalcogenides from a series of XPS spectra obtained at different X-ray photon energies (50 – 700 eV). The restored total and partial densities of states with the dominant contribution for chalcogenides GaSe, $CuGaSe_2$, $Bi_{1.8}Mn_{0.1}In_{0.1}Se_3$ and $Bi_{1.8}Co_{0.1}In_{0.1}Se_3$ are compared with the corresponding ones calculated from the density functional theory.

After background subtraction, the XPS spectrum is given by the sum of the products between the PDOS and the photoionization cross section for the corresponding partial electron states of the atoms at a given photon energy (taking into account the renormalization of the terms to eliminate the influence of the photon flux). In the case of a set of XPS spectra, we obtain a system of equations where the unknowns are the PDOS. Finding the PDOS is an inverse problem, which in general may be an ill-posed problem. Estimating parameters for parameterized PDOS based on their posterior distribution is a good way to solve such a problem, since the posterior distribution should typically be unique and robust to the input data. Therefore, the maximum and/or mean of the distribution yields a unique set of parameters for specifying the PDOS. Bayesian statistical inference using replica exchange Monte Carlo simulations allows one to sample the posterior distribution of parameters.

In some cases, it is possible to reconstruct the PDOS even in the presence of sufficiently noisy spectra or when the number of XPS spectra is less than the number of PDOS (the number of equations is less the number of unknown variables). Note that for successful restoration it is necessary that the values of the photoionization cross-section of the orbital contributions of atoms in the material differ sufficiently well from each other in the used range of photon energies.

Acknowledgement: The study was supported by the Russian Science Foundation (project no. 23-72-00067, https://rscf.ru/project/23-72-00067/) with use of the unique scientific facilities of the Kurchatov synchrotron radiation source.

PP-42

Beamline for studying fast-flowing processes at the synchrotron radiation facility SKIF

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“Fast Processes” is one out of 1st priority beamlines of SRF SKIF (Synchrotron Radiation Facility - Siberian Circular Photon Source "SKIF") synchrotron which will be created under a state contract with LIH SB RAS at the end of 2024.

The beamline would include two independent instruments installed at a wiggler source, i.e., Dynamic processes and Plasma. The beamline is designed to meet a wide range of research and technological challenges related to processes occurring in nano- and microsecond timescales. The current conceptual design of the beamline aims at a complex approach to structural studies of various objects relying on high-brightness synchrotron radiation beams.

The beamline would implement X-ray diffraction, small-angle scattering, and radiography techniques with a high temporal resolution, with a typical delay between frames down to 2.8 ns and exposures of about 50 ps.

The report presents the current state of work on the manufacture of the main technological equipment of the “Fast Processes” beamline, as well as the characteristics of the main planned techniques.

Acknowledgement: This work was performed within the framework of a budget project of the Ministry of Science and Higher Education of the Russian Federation for Synchrotron radiation facility SKIF, Boreskov Institute of Catalysis (FWUR-2024-0042).

PP-43

X-ray absorption and X-ray emission spectroscopy study of a molybdenum disulfide and Ni-doped molybdenum disulfide

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Molybdenum disulfide (MoS_2) is a 2D nanomaterial sought after for its exceptional mechanical and chemical stability. To enhance its properties, researchers often doped MoS_2 with heteroatoms by replacing Mo and S with other cations and anions. This technique allows for the creation of nanostructures with improved electrocatalytic activity and electric conductivity. The doping Ni in MoS_2 lattice significantly alter its structural and electronic properties. This results in improved catalytic activity, particularly in hydrogen evolution reactions and oxygen reduction reactions, making it a promising material for energy storage and conversion applications.

The aim of this work was to establish the effect of Ni doping on the structure and electronic states of MoS_2. Changes distribution of the partial density of unoccupied (conduction band) and occupied (valence band) S, Mo and Ni in MoS_2-based nanomaterials was studied were studied using X-ray emission spectroscopy, X-ray photoelectron spectroscopy and Near Edge X-Ray Absorption Fine Structure in comparison with density functional theory calculations.

The doping of Ni into MoS2 results in a change in the size of the band gap. Ni atoms are located in a quadrupole environment of S atoms. X-ray measurements revealed a shift towards lower energies of 3d, 4d Mo levels and towards higher energies of 3p, 2p S levels. The DFT calculation showed a shortening of the bond between the metal atom and sulfure during Ni doping. We detected a transfer of electron density from S atoms and Mo atoms of the first environment of Ni to Mo atoms of the second environment and doped Ni atoms.

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PP-44

Development a techniques complex of single crystals unit cell parameters precision determination at synchrotron radiation stations

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Determination of single crystal unit cell parameters (UCP) may be required when a significant line overlap on polycrystalline diffraction patterns makes it impossible to qualitatively process profiles and reliably determine their centres. Such situation usually occurs when studying the low symmetric phases with UCP greater than 10 Å; determining the UCP on temperature dependencies for medium and low symmetric phases; characterizing solid solutions of phases with close UCP; determining the uniformity of extended synthesized crystals. The Rietveld method is widely used in such cases, but to estimate the actual UCP determination accuracy for it can be quite difficult, due to the correlation of the contributions of a large number of parameters to the positions and shape of overlapping diffraction lines. As we have shown earlier, this problem cannot always be solved even with use high-resolution synchrotron radiation [1]. The study of individual single crystals, for which there is no problem of lines overlapping, seems to be a solution in such situations.

The determination of single crystal UCP with high accuracy is a difficult independent task. Conventionally, its solution requires the separate devices with special characteristics and parameters of radiation [2]. The majority of the techniques focused on standard diffractometers requires a point detector, while modern devices are equipped mainly with two-dimensional ones.

In this work, a new technique complex for diffraction studies of functional materials is proposed, focused on application at synchrotron radiation stations equipped with a goniometer and a 2D detector [3-4]. The complex includes two techniques based on calibration according to an external or internal standard: the entire plane of the detector for setups equipped with a one- or two-circle goniometer; individual sections of the equatorial circumference of the goniometer if it has three or four degrees of freedom. The first approach can be implemented directly on the main 2D detector. The SearchXY-2θ data processing program [5] based on the PyFAI library [6] has been developed for its application. The second technique involves the use of high-angle diffraction peaks placed in a reflective position on the equatorial circumference of the goniometer. To implement it, the James program [7] that analyzes the accessibility of the studied single crystal reflexes and calculate the conditions for their moving to reflective position (goniometer installation angles) was developed. In addition, a mathematical apparatus for processing the data obtained and scheme for using an additional detector (if there are significant restrictions on the rotation angle of the main one) were proposed. Preliminary calculations of the scheme technical characteristics for using at the SKIF station 1.2 "Structural Diagnostics" have been performed. According to the estimates, it will be possible to achieve a level of relative error in determining the UCP of no more than 5×10^{-6} with the scheme use, which will also allow metrological studies to be carried out at the station.

At the first stage, before the commissioning of the SKIF stations, these techniques were implemented and tested on Bruker D8 Venture laboratory single-crystal diffractometer (MoKα-radiation, Incoatec IμS 3.0 microfocus tube, monochromatization and focusing with multilayer Montel mirrors, three-circle goniometer, PHOTON III detector with 768×1024 resolution and 135×135 μm pixel size, Oxford Cryosystems Cryostream 800 plus open-flow nitrogen cryostat). The technique of the entire detector plane calibration is demonstrated by refining the UCP on temperature dependence for $Na_6Mo_{11}O_{36}$ monoclinic crystals. Some methodological issues related to the choice of indices *hkl* when refining UCP for low-symmetric phases are considered. The temperature dynamics of the thermal expansion tensor elements is studied. The technique of goniometer equatorial circumference fragments calibration is implemented by studying the homogeneity of a synthesized $Bi_4Ge_3O_{12}$ single crystal weighing 52 kg. According to the UCP refinement results in the high-angle diffraction region (~ 120°) for 9 its fragments with a relative error of 4×10^{-5}, it was shown that the initial sample is single-phase and homogeneous. The study also showed that $Bi_4Ge_3O_{12}$ can be considered as an effective diffraction standard due to the high intensity of reflexes and low FWHM values, as well as the availability of a developed technique for obtaining crystals of high quality with reproducible characteristics [8]. Its UCP, averaged over all the studied crystals, was 10.5180(5) Å.

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PP-45

Application of molecular modelling and SAXS data to reveal structural features of DNA-aptamers

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Aptamers, which are short sequences of nucleotides (DNA or RNA), have become an object of special interest in various fields of science and medicine due to their ability to bind highly specifically to a variety of targets, including proteins, small molecules and even whole cells. Their unique properties make aptamers promising tools for use in diagnostic systems, therapeutic applications, biosensors and other biotechnological developments.

As the applications of aptamers expand, there is a growing need to better understand their spatial structure, as it is their three-dimensional configuration that determines their ability to recognise and bind to target molecules. Traditional methods, such as X-ray crystallography and nuclear magnetic resonance (NMR), do not always provide detailed information about the structure of aptamers, especially when they function under near-physiological conditions.

Small-angle X-ray scattering (SAXS) offers a unique opportunity to complement traditional techniques and provide valuable information on the shape and flexibility of aptamers in solution. This technique allows the collection of data on the structural characteristics of biomacromolecules without the need for crystallisation, which is particularly relevant for the study of dynamic and structurally flexible objects such as aptamers.

The use of SAXS data in combination with molecular modelling allows the atomic structure of aptamers to be reproduced with a high degree of accuracy. Molecular modelling, including molecular dynamics, quantum chemistry and docking methods, allows not only the static structure to be simulated, but also the dynamic aspects of aptamer behaviour when interacting with target molecules to be investigated. This provides insight into the mechanisms of binding and adaptation, which in turn facilitates more informed design of new aptamers with improved properties.

Aptamers have been investigated for the treatment and prevention of a number of serious diseases, including brain and lung cancers, as well as viral diseases such as SARS-CoV-2.

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PP-46

Temperature camera TK-1200 intended for high energy synchrotron X-ray diffraction

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Conducting temperature studies of the crystalline structure of materials has always been of interest to scientists, technologists, manufacturers, and developers. At the moment, dozens of devices have been created that allow us to determine what exactly is happening inside materials at the atomic level, and the result becomes even more valuable and multifaceted when it is supplemented with data on temperature effects on the structure and properties of the sample. In combination with a synchrotron source, a temperature camera opens up the possibility of literally observing the changes occurring inside the material with varying temperature in real time with very high accuracy [1].

For this purpose, we have developed the TK-1200 temperature camera (Fig. 1) for temperature X-ray studies. This device is a vacuum furnace with a cooling circuit and windows for the X-ray beam, which in turn are protected by graphite and PM-A film. In addition, the TK-1200 design allows for controlled rotation of the sample around its own axis by 360° in the plane of the sample, thereby studying the diffraction of internal stresses. Detailed characteristics are presented in Table 1.

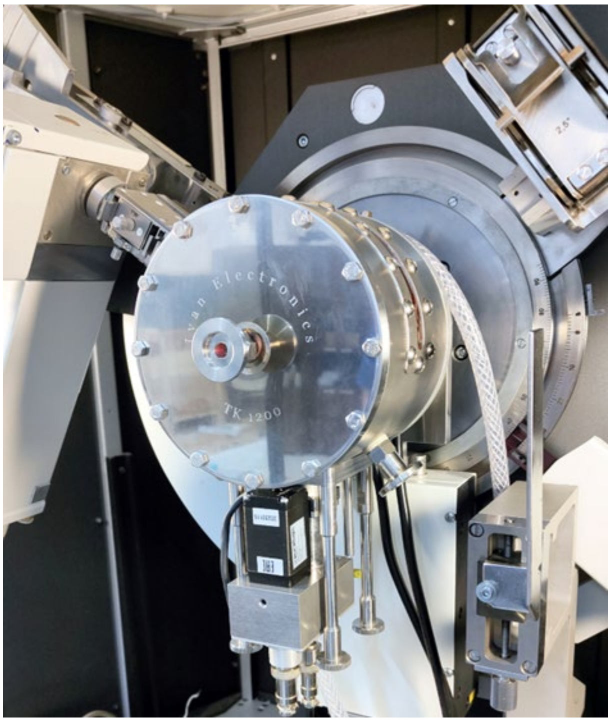

Fig. 1. Photo of the TK-1200 temperature camera on a vertical goniometer

PP-46

Table 1. Characteristics of the TK-1200 temperature camera for diffraction studies.

Characteristics	Value
Operating temperature range	From (+25°C) to + 1200°C
Temperature determination accuracy	±2°C
Temperature maintenance accuracy	±0.5°C
Diffraction angle, 2Θ	From -1° to 152°
Window radiolucency (Cu Kα)	~63.5%
Vacuum equipment, pressure	<10^{-6} mbar
Sample rotation	Oscillation from 0° to 360° with speed from 0°/s to 180°/s

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PP-47

Mössbauer absorption spectroscopy using synchrotron radiation

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Mössbauer absorption spectroscopy using synchrotron radiation is an effective method for studying the properties of various elements, which allows studying local electronic and chemical states in materials. The use of synchrotron radiation significantly increases the efficiency of measurements.

The combination of Mössbauer spectroscopy in its energy region with the high brightness of synchrotron radiation allows, unlike radioactive sources, to provide a beam of several microns in size. Therefore, studies of small samples that take weeks with a radionuclide gamma resonance source can be completed in minutes using a synchrotron, and year-long scientific research can be completed in a few days.

The MS-2020 Mössbauer spectrometer, developed and manufactured at the Southern Federal University, Research Institute of Physics, with a movable resonant detector and a synchronous Doppler modulation system, significantly increases the energy resolution in transmission Mössbauer spectra and, due to the movement of the resonant detector, opens up opportunities for recording synchrotron radiation at a new level.

The spectrometer includes a synchronously moving resonant scintillation detector with a radionuclide source (optionally replaceable for synchrotron radiation) based on two Doppler modulators, two control electronic units of the spectrometer and special software that allows setting the required value of motion desynchronization in the modulation modes used, such as constant acceleration, constant speed, and the "interval" mode. The sample under study is positioned motionless between the moving resonant detector and the source. Optionally, it is possible to place the sample in a cryostat or heater, which will allow studying magnetic and non-magnetic phase transitions, etc.

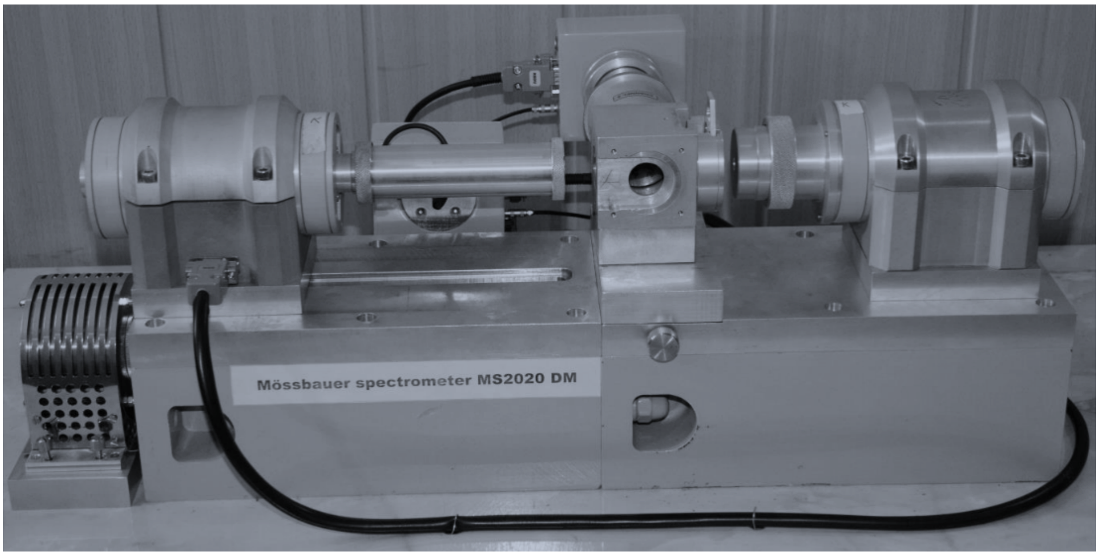

Fig. 1. Mössbauer spectrometer MS-2020 with double modulation, basic model

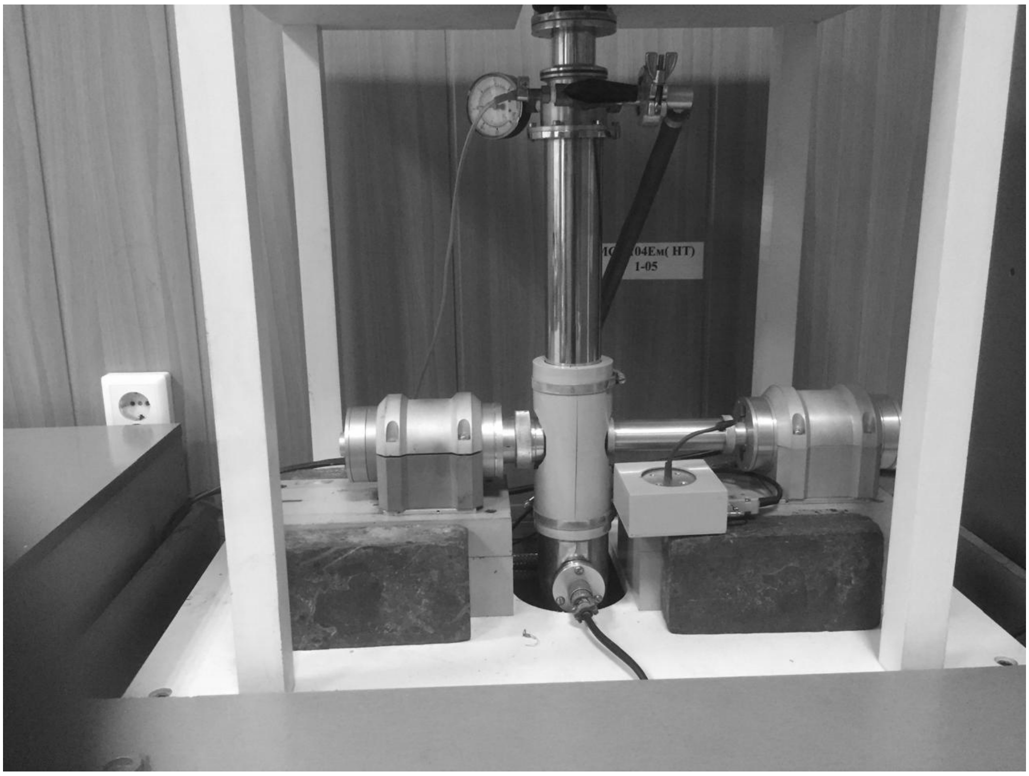

Fig. 2. Mössbauer spectrometer MS-2020 with double modulation and helium refrigerator

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PP-48

Experimental investigation of band structure of nano-inclusions in $Fe_xNi_yTiSe_2$

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Thermodynamically, intercalate compounds based on quasi-two-dimensional transition metal dichalcogenides are solid solutions with limited stability on the composition-temperature plane. When the stability limit of the homogeneous solid solution is exceeded, its decomposition with the formation of new phases is observed [1]. The strong anisotropy of the diffusion coefficient practically prevents diffusion in the direction perpendicular to the base plane, so that the decomposition products are encapsulated in the interlayer space of the host lattice [2]. The confined nature of the interlayer space allows it to be considered as an ideal site for the synthesis of inclusions, as there is no contamination by unwanted atoms (e.g. oxygen). At the same time, the small size of the interlayer space automatically makes the inclusions nanoscale [1,3]. The small inclusion size allows us to expect an epitaxial influence of the host lattice on the crystal and electronic structure of the inclusions. However, our previous studies on the decomposition of homogeneous intercalates with the formation of nanoinclusions did not reveal any new crystalline forms of the decomposition products. At the same time, it was found that the inclusions have a flat shape and are coherently oriented with respect to the host lattice in such a way as to ensure maximum coincidence of the interatomic distances of the base plane of the host lattice and one of the lattice planes of the inclusion. This suggests a strong interaction between the inclusion and the host lattice. In particular, electron transfer between them can be expected, so that it is possible to obtain both the host compound and the inclusion compound in a state with a lattice not perturbed by doping, but with a modified carrier concentration. In effect, we are talking about a bulk epitaxial composite.

A comparative study of the electronic structure of the matrix lattice, which acts as a substrate, and a needle-shaped inclusion ('whisker') formed in it during growth has been carried out. It was found that despite the different composition of the matrix lattice and the inclusion, their electronic and zonal structures are almost identical. This is due to the redistribution of nickel atoms at temperatures close to room temperature. Thus, the difference in Fermi energies between the inclusion materials and the main crystal can act as an effective stimulus for the redistribution of constituents even at temperatures as low as room temperature.

PP-48

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PP-49

Exploring crystallization and melting of Polyether Ether Ketone (PEEK) with Fast Scanning Calorimetry (FSC)

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In recent years, significant advancements have been made in developing thermoplastic matrices for composite materials, with Polyether Ether Ketone (PEEK) standing out as a key polymer in this area. PEEK is known for its exceptional toughness, strength, and rigidity, as well as its remarkable resistance to chemicals and radiation. These combined properties, along with its long shelf life, suggest that PEEK may offer advantages over thermoset materials due to its ease of fabrication, reparability, and reprocessing capabilities [2].

The technique called Fast Scanning Calorimetry (FSC) on a chip allows for rapid thermal measurements at heating and cooling rates of up to 10^5 Ks^{-1}. This approach facilitates the characterization of a sample's thermal behaviour without causing uncontrolled changes during the heating process by kinetically avoiding the chemical transformation of monomers. Moreover, FSC is capable of identifying closely occurring phase transitions. Previous research has shown the effectiveness of this relatively new method in analysing individual particles of energetic materials during rapid heating [1].

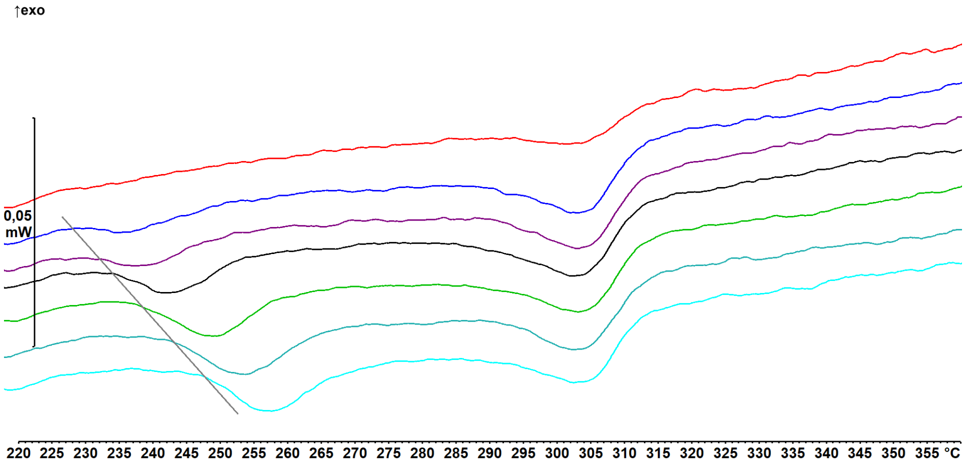

Fig. 1. FSC-curves depending on the crystallization time at 210°C (1-2-3-10-80-640-3600s from up to down).

The multiple-melting behaviour seen in semi-rigid chain polymers like PEEK has been a subject of discussion for over fifty years, yet a conclusive explanation for this phenomenon remains elusive. This double-melting behaviour is evident in PEEK samples that have been isothermally crystallized or cooled from the melt, as observed through classical DSC and FSC methods. The higher-temperature melting peak typically begins between 280-310°C and is largely unaffected by the sample's thermal history, whereas the onset of the lower-temperature melting peak is significantly influenced by the crystallization conditions (see Fig. 1). A straightforward explanation for PEEK's double-melting behaviour might be the presence of two distinct polymorphs. However, no polymorphs of PEEK have been detected via X-ray diffraction (see Fig. 2). But, techniques like FSC combined with synchrotron radiation can provide insights into these processes and their nature. [3].

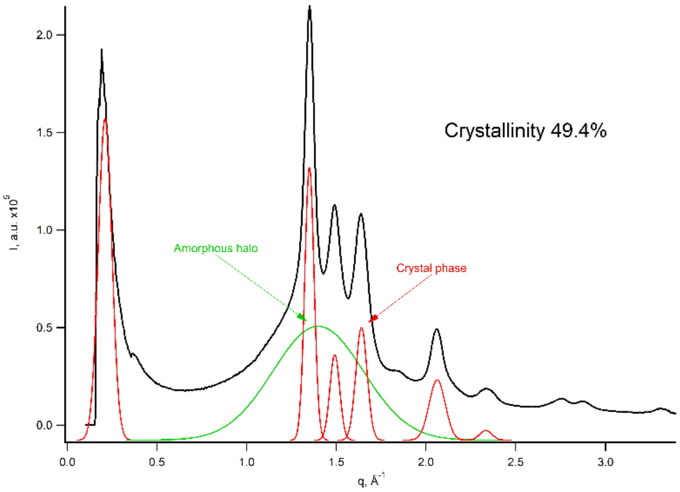

Fig. 2. Small-angle X-ray scattering (SAXS) image of PEEK.

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PP-50

Brush polymers based on acrylates and the study of their self-assembly processes

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Materials based on brush polymers are of great interest for study. Brush elastomers, obtained by grafting long side chains onto a linear backbone, exhibit a wide range of mechanical properties, such as the Young's modulus characteristic of gels with linear chains, and significant reversible deformations. The focus of this research is on brush block copolymers based on n-butyl acrylate (PBA) and tert-butyl acrylate (PtBA), with degrees of polymerization for the side and main chains of 50 and 15, respectively. The synthesis of PtBA homopolymers with degrees of polymerization of 21, 50, 100, and 150 was carried out, as well as the synthesis of potassium methyl methacrylate (KOMA), which is necessary for converting the macroinitiator into a macromonomer, and the macromonomer $PtBA_{50}$-MA, as well as a brush graft polymer based on them. Modern methods of physicochemical analysis were used to characterize the obtained compounds: gel permeation chromatography, NMR, and IR spectroscopy. Further studies of the physical properties of these polymers using synchrotron methods are planned.

PP-51

Electronic and crystalline structure of Bi/InAs(111)A-(2x2) interface

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After the discovery of graphene and its unique properties, special attention is paid to obtaining graphene-like structures made of heavy elements atoms, such as bismuthene, antimonene and other. Increasing the strength of spin-orbit coupling is expected to increase a band gap in the Dirac spectrum of such materials, which is essential for controlling the conductivity of the system and realizing potential spintronic devices. Furthermore, the theoretical studies predict that bismuth bilayer and bismuth thin films are 2D topological insulators. However, the crystalline and electronic structure of thin films and 2D materials is influenced by the substrate material it is interfaced with or grown on. It is known, that 2D electron gas (2DEG) is formed on n-InAs(111)A surface. The Rashba spin splitting of the InAs surface 2DEG can be tuned via external electric field, making it perspective platform for realizing Datta-Das type spin transistor. The influence of Bi atoms addition on the InAs 2DEG states spin-orbit coupling strength is of great interest for future spintronics applications.

The aim of this work is to study the conditions of bismuth adsorption and epitaxial growth on InAs(111)A surface, and their mutual influence on the crystalline and electronic structure of the interface formed.

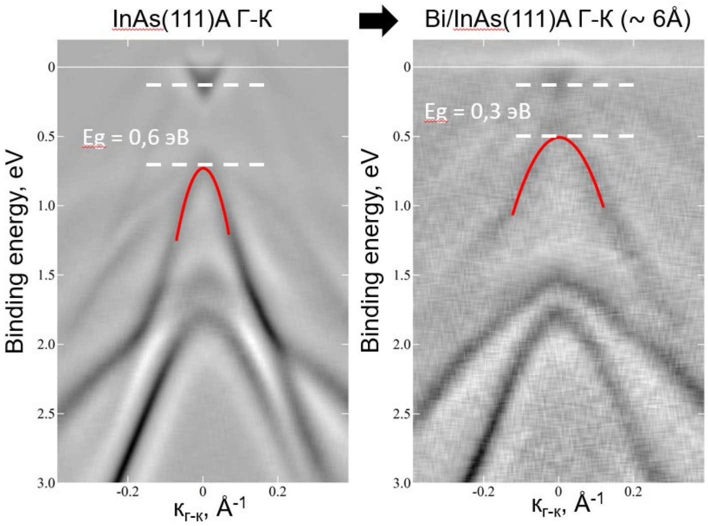

Fig. 1. Changes of InAs(111)A-(2x2) surface band structure after deposition of 6 Å thick Bi layer at room temperature revealed by ARPES.

Bismuth was deposited from the Knudsen cell onto the surface of InAs(111)A-(2x2) substrate at room temperature in UHV ($<5\cdot10^{-10}$ mbar) conditions. The electronic structure of the grown Bi/InAs(111)A films surface was probed in-situ using angle resolved photoelectron spectroscopy (ARPES). The crystalline structure and the chemical state of atoms on the surface were studied using X-ray photoelectron spectroscopy (XPS), X-ray photoelectron diffraction (XPD) and low energy electron diffraction (LEED). Usually, these methods are realized on synchrotrons due to the high intensity and the small spot size of the synchrotron radiation. But in the present work XPS, XPD and ARPES measurements were conducted with a laboratory equipment SPECS GmbH ProvenX-ARPES system in Rzhanov Institute of Semiconductor Physics.

It was found, that at the first stage of growth (Bi layer thickness < 8 Å) complex $InAs/InAs_{1-x}Bi_x$ /Bi(111) transition layer is formed. The surface electronic structure at this stage is close to the band structure of InAs(111)A surface. But decrease of band gap width and increase of holes effective mass occur compared to InAs (Fig. 1). When thickness of bismuth film is increased, the electronic structure of the film changes and looks similar to the electronic structure of the surface of the Bi(111) crystal. However, the LEED pattern indicates the formation of a phase with a rectangular unit cell, which may be explained by the formation of domains with the Bi(110) structure or the two-dimensional α-Bi phase.

PP-52

Mouse fibroblast cells – porous silicon nanoparticles hybrid structures by synchrotron studies

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Bio-nano-hybrid materials can be obtained by combining inorganic nanostructures with biological objects. Porous silicon nanoparticles (PSi NPs) have a number of specific, unusual biological properties. These properties make it possible to combine PSi NPs with organic mammalian cells. Such bio-nano-hybrid structures have a huge research interest in the scientific community. High–precision diagnostics of such materials and structures is an important part of their effective application, including in theranostics and biomedicine. Changes in the physicochemical state of the surface, which largely determines the properties of PSi NPs when integrated with the biological structure, are extremely relevant for study. The synchrotron XANES and XPS allows obtaining information about the physicochemical state, composition, structure, atomic and electronic structure of not only nanoscale objects, but also biohybrid nanostructures, which are characterized by an extremely developed surface.

An aqueous suspension of porous silicon nanoparticles was used as a material integrated into cells. The suspension was obtained by grinding porous silicon films (electrochemical etched Si) in the ball mill. The organic part of the biohybrid structure for this work were 3T3 NIH cell cultures (mouse fibroblasts) grown on the gold surface for three days. As a result of the integration of PSi NPs into 3T3 NIH cells, a hybrid sample was obtained, the incubation time of silicon nanoparticles was 72 hours. XANES spectra were registered for $L_{2,3}$ – edge of silicon atoms or K – edge of oxygen atoms. NanoPES beamline of the Kurchatov synchrotron radiation center (NRC "Kurchatov Institute") was used.

The results of atomic and electronic structure and composition synchrotron studies of the biohybrid structure "mouse fibroblasts - silicon nanoparticles" by XANES and XPS spectroscopy together with scanning electron microscopy data allow us to conclude that nanoparticles combined with biological objects are subject to changes in the composition, structure and physico-chemical state of the active surfaces. In its turn this should allow precision variation of the nanoparticles impact to mammalian cells by controlled integration.

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PP-53

Epitaxial Sn-Si ultrathin nanolayers structure by synchrotron study

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Heat generation can be considered a limiting factor in the creation and production of silicon-based electronic devices. A significant reduction in thermal conductivity can be achieved through the formation of periodic multilayer structures Sn/Si. Thus, disturbances in the movement of phonons are more noticeable due to the higher mass ratio Sn/Si. At the same time, studies of the atomic and electronic structure, the physico-chemical state of such structures are extremely important from the point of view of their formation technology and subsequent application. The aim of this work was to study the atomic and electronic structure of silicon-tin-based epitaxial nanostructures.

The studied samples were obtained by molecular beam epitaxy on a Si(100) substrate with a 50 nm thin epitaxial Si buffer layer. About 5 monolayers of tin were deposited on Si buffer layer (on pre-cleaned and dried silicon substrates) in an ultrahigh vacuum chamber. For possible modification of the composition and structure, the obtained samples were annealed *in-situ* in an ultrahigh vacuum of 10^{-9} Torr at 800 °C for 10 min. Additionally Si-Sn solid solution thin epitaxial layers limited in thickness by 5 nm and sufficiently beyond the known and achievable solution limits were grown on the surface of Si buffer and studied as well.

Atomic and electronic structure studies were carried out by XANES (X-ray absorption near edge structure) spectroscopy and XPS (X-ray photoelectron spectroscopy). Synchrotron radiation of the BESSY II (Helmholtz Center Berlin, Berlin, Germany) and Kurchatov (National research Center "Kurchatov Institute", Moscow, Russia) storage rings was used.

By comparing the spectra of the reference phases of metallic tin, SnO_2 and SnO with experimental ones, the features of the electronic spectrum and the composition of epitaxial nanolayers of tin and Si-Sn solid solution films before and after annealing were evaluated. Further, using XANES spectra calculated from the first principles (linearized augmented plane wave method within the framework of the Density Functional Theory) by the Wien2k software package for materials such as Sn, SnO, as well as orthorhombic and tetragonal SnO2, a semi-quantitative assessment of the composition of the epitaxial nanolayer before and after annealing by Linear Combination LCF (Linear Combination Fitting) was performed. The results obtained showed the presence of all three oxides in the initial epitaxial tin film. Also re-distribution of the free electronic states near the conduction band bottom was studied and revealed the additional states formation in the band gap in case of solid Si-Sn solution. It is shown that the growth of the tin nanolayer on the surface of the silicon buffer layer does not lead to a noticeable interatomic interaction at their interface. As a result, there are no noticeable distortions of the electron-energy spectrum for epitaxial Sn film if compared to the Si-Sn solid solution one. The formed epitaxial Sn nanolayer has a complex composition of various tin oxides. However, high-temperature *in-situ* ultrahigh vacuum annealing leads to a restructuring of the phase composition of the surface layers of such a structure, which is accompanied by the migration of oxygen atoms from atop tin atoms deep into the structure to silicon. This leads to complete oxidation of the surface of the buffer layer of single crystalline silicon and the restoration of 5 monolayers of the original tin oxide to a metallic state. In case of Si-Sn epitaxial film the electronic structure redistribution is “cured” as the annealing result.

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PP-54

Determining the atomic structure of biological molecules using SAXS

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The current study illustrates the outcomes of constructing the three-dimensional atomic structure of aptamer molecules in authentic solution conditions through the use of small-angle X-ray scattering (SAXS) techniques, in comparison with X-ray diffraction (XRD) data. The theoretical modelling based on SAXS data yielded satisfactory results, indicating that this approach represents a promising avenue for obtaining the atomic structure of small molecules, which, in turn, can inform the design of new aptamers. The initial stage is the modeling of the secondary structures based on the nucleotide sequences. Subsequently, the actual full-atomic modeling of the three-dimensional structure is performed based on the secondary structure. This provides an initial insight into the atomic structure of the aptamer and its principal structural components in three-dimensional space. Subsequently, molecular dynamics calculations are conducted on the aptamer, employing a realistic physiological environment. This provides information on the conformational changes of the aptamer in solution, with explicit consideration of the solvent, temperature, and pressure. Subsequently, the atomic structure derived in the preceding step is subjected to refinement through the implementation of quantum chemical calculations utilising the FMO method. Pairwise interactions are then analysed with a view to elucidating the physical phenomena occurring between the aptamer and the protein. Following this, the theoretical scattering curves are subjected to a comparative analysis with the experimental SAXS scattering curves. This step represents a critical phase in the selection process, whereby only those conformations of the aptamer exhibiting the least deviation from the experimental data are retained.

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PP-55

Investigation of the impact of surface morphology of electrospun polymer matrices on cell adhesion and proliferation

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The development of modern technologies in the field of tissue engineering is determined by the use of interdisciplinary approaches based on functional biopolymers [1]. One of the ways to obtain tissue-engineered constructs is the electrospinning method. These polymeric constructs consist of a multitude of nanofibers and represent a framework mimicking the extracellular matrix. In such a framework, porosity, pore diameter and fiber diameter are the defining parameters [2]. The main problems that present themselves are the uneven distribution of cells, cell attachment and lack of cell migration in the nanofiber matrix.

The aim of this study was to evaluate cell adhesion and distribution in polymer matrices having different surface morphologies obtained by electrospinning. Polylactide (PLA) with a molecular weight of 110 kDa was used as a biodegradable polymer and dichloromethane (DCM), hexafluoroisopropanol (HFIP), and tetrahydrofuran (THF) were used as solvents. Polymer matrices were prepared using MECC NF-500 electrospinning setup, with a polymer concentration of 100 mg/ml. The diameter and porosity of the fibers were determined using a Carl Zeiss CrossBeam 550 scanning electron microscope. Mechanical characteristics: dynamic mechanical analyser TA Instrument RSA-G2. The cell line of glioma C6 (rat).

Porous fibers were obtained using DCM-PLA, DCM/THF-PLA, and smooth fibers were obtained using HFIP-PLA. The best mechanical properties was shown by polymer matrices with a smooth fiber surface, while fibers with a porous surface had lower properties because pores are considered as defects on the fiber. Based on the data obtained, we concluded that cell adhesion and proliferation increase as the diameter and number of pores on the surface of the polymer fiber increase. Micrographs obtained by SEM and DAPI staining confirmed the attachment of cells to the polymer matrix. Fibers having a smooth surface morphology showed the lowest results for cell adhesion and proliferation. DCM/THF-PLA has shown better cell attachment and can be considered as an optimized material for further research in this direction.

PP-55

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PP-56

New class of biomimetic materials: Structural studies with synchrotron radiation

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Self-assembled networks of bottlebrush copolymers are promising materials for biomedical applications due to a unique combination of ultra-softness and strain-adaptive stiffening, characteristic to soft biological tissues [1]. Transitioning from ABA linear-brush-linear triblock copolymers to A-g-B bottlebrush graft copolymer architectures (fig.1 a, b) allows significantly increasing the mechanical strength of thermoplastic elastomers. The corresponding increases of the aggregation number and extension of bottlebrush strands lead to a significant increase of the strain-stiffening parameter up to 0.7, approaching values characteristic of the brain and skin tissues. Network reconfiguration without its disassembly is an efficient approach to adjusting the mechanical performance of tissue-mimetic materials to meet needs of diverse biomedical applications. The structural reorganization of the self-assembled networks was monitored by the real-time synchrotron small-angle X-ray scattering (fig.1, c). This showed that selective solvent annealing resulted in a substantial, irreversible reconfiguration of the spherical domains of grafted side chains in the bottlebrush matrix, leading to an increase in domain size and the distance between them. In contrast, the evolution of the structure in a selective solvent for the bottlebrush matrix was found to be reversible.

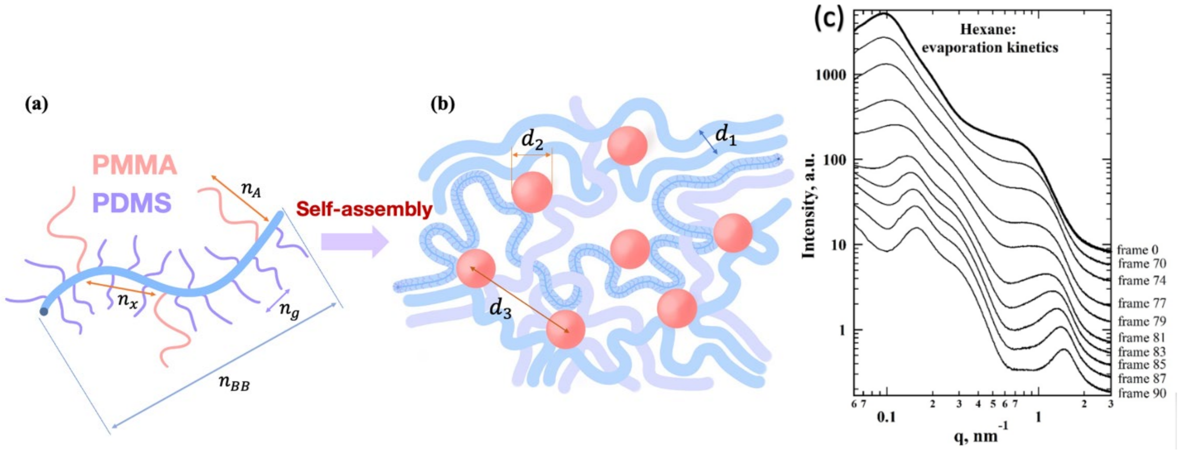

Fig. 1. A) (a) Controlled radical polymerization of assorted macromonomers yields A-g-B brush-like graft copolymers with controlled grafting density of the PDMS brush (B) Microphase separation of the PMMA and PDMS block results in the formation of a physical networks linked by PMMA domains dispersed in the PDMS bottlebrush matrix. (c) SAXS profiles corresponding to bottlebrush system

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PP-57

Segmentation of photoelectron spectra using convolutional neural network

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X-ray photoelectron spectroscopy (XPS) is an indispensable technique for the quantitative determination of sample composition and electronic state of elements applied in modern catalysis and material science. Currently used methods of analysis of XPS spectra rely on experience and knowledge of spectroscopists and usually are conducted manually. Improvements of excitatory source, including application of the synchrotron radiation, advance the quality of experimental data and allow one to obtain larger datasets. However large datasets are quite difficult to process manually. Despite becoming XPS instruments more automated to fit both the inelastic scattering and the line shapes of the peak regions is a challenge, that generally involves task of peak detection. Classical algorithms of spectra analysis may cause a significant error in peak detection and fitting, and therefore cannot be used in automatization task. Recently, machine learning based algorithms have been successfully applied to data analysis in wide range of physical methods [1, 2].

The search of XPS peak maximum and peak region can be considered as a segmentation problem. Then PeakOnly model approach [2] was modified to detect not only regions but also peak maxima.

Synthetic data was used for machine learning model training. Each synthetic spectrum included noise, peaks and inelastic scattering background. Peaks was simulated from pseudo-Voigt functions with random parameters. To label the training data, the peak area was defined at three sigma from the peak maximum. Inelastic scattering background was simulated from normal cumulative distribution function. To precisely detect overlapping peaks and satellites, the dataset was expanded with small satellite-like peaks also generated from pseudo-Voigt functions. Final dataset contains 4000 artificial spectra for training and 400 for testing.

Full training process took 10 minutes on CPU (6 cores, 12 threads, 3.0 GHz) with 8 Gb RAM. Since the architecture was optimized for fast inference, the spectrum process took less than a second on a home laptop. The final model achieved 15% error in the intersection over union metric for the peak area and peak maxima detection in the test data.

For the first time convolutional neural network for segmentation of any XPS spectra was developed. Developed CNN model is ready to use for the framework for the fast XPS data analysis.

The design of model architecture, training and testing was performed using PyTorch in Python.

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Local atomic structure of FeO coatings obtained by thermal annealing magnetron deposited iron films

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Thin films of metal oxides have been widely described in the literature due to their photochemical stability. Thin film oxide coatings based on transition metals of groups IV, V are widely used in the production of microelectronic and optical components, protective and biocompatible coatings [1].

Iron (III) oxide thin films have potential advantages [2] for photoelectrochemical hydrogen production. It is stable in aqueous solutions with pH above 3, has a suitable band gap of 2-2.2 eV, which provides 40% absorption of sunlight. Magnetite (Fe_3O_4) has been used for thousands of years as one of the important magnetic materials. In particular, modified Fe_3O_4 thin films have excellent electrical and magnetic properties compared to pure films [3]. But the conditions for obtaining various iron oxide phases must be controlled using various methods of structural and chemical analysis, and thus the main goal of this study is to study the local atomic structure depending on the oxidation conditions of magnetron-deposited iron films.

Visualization of the surface morphology of magnetron-deposited iron films oxidized in air at different temperatures was carried out using a scanning electron microscope (SEM), and X-ray structural analysis (XRS) was used to identify the obtained iron oxide phases. Distribution of various iron oxide forms across the film thickness was carried out by X-ray photoelectron spectroscopy (XPS). The local atomic structure was studied by EXAFS spectroscopy.

XANES/EXAFS (Fe-K) spectra of the studied samples were recorded at the EXAFS spectroscopy station (SSTRC, Novosibirsk). The state and local atomic structure in the samples were studied by XAFS (XANES/EXAFS) spectroscopy based on the fluorescence yield with selective background cutoff. The lengths of interatomic bonds and the corresponding coordination numbers were determined. The data obtained by different methods are in good agreement with each other. Possible variants of structural models are considered in detail.

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PP-58

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PP-59

In situ investigation of the formation of porous Co-Pd alloys in the process of reductive thermolysis

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Porous cobalt–based alloys obtained by thermolysis of multicomponent precursors are promising metal materials for the production of heterogeneous catalysts for various applications [1-5]. The advantage of the proposed synthesis method is the possibility of obtaining alloy systems at temperatures significantly lower than the melting point of the alloy components – this avoids sintering and provides a relatively high specific surface area of the alloy, which is one of the most important requirements for heterogeneous catalysts.

However, the synthesis at relatively low temperatures can lead to an uneven distribution of metal components in the alloy, due to a decrease in the rate of their mutual diffusion. Based on the results of previous studies on porous nickel-based alloys [6], precursors for the synthesis of Co–Pd alloys – $[Co(NH_3)_6]Cl_3$ и $[Pd(NH_3)_4]Cl_2{\cdot}H_2O$, were selected. The alloys obtained from these precursors by reductive thermolysis at 800 °C showed high activity in the model reaction of catalytic decomposition of hydrocarbon substrates, accompanied by the growth of carbon nanofibers (CNF). Nevertheless, during the experiment on the study of intermediate products of thermolysis of precursors of the $Co_{0.9}Pd_{0.1}$ alloy by XRD and TEM methods, it was shown that the components of the alloy are not uniformly distributed.

To determine the causes of the heterogeneous distribution of the alloy components, a study of the process of reductive thermolysis of precursors and alloy formation was conducted using the *in situ* XRD method based on the Siberian Synchrotron Radiation Center of BINP SB RAS. The experiment was carried out in an Anton Paar XRK-900 high-temperature chamber at station No. 6 of the VEPP-3 storage ring using an OD-3M position-sensitive detector [7]. Based on the data obtained, assumptions are made to explain the above-mentioned uneven distribution of elements in the obtained alloys. Due to the experiments carried out, data on the phase composition of the alloy were obtained under conditions similar to those of the catalytic reaction of CNF growth. The results of the experiment helped to select alternative precursors for the synthesis of porous Co-Pd alloys.

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PP-60

Do crystallization media influence to macromolecule-ligand complex structure? Molecular dynamics study

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A knowledge of macromolecule 3D-structure is a cornerstone to structural biology. An X-ray scattering is a basic technic to resolve atomic co-ordinates over decades. While an analysis of diffraction picture is considered in general as solved problem, a main issue in biomolecular X-ray scattering researches is to grow a monocrystal. Crystallization conditions should be set in a very fine manner because of high complexity of macromolecular systems. The problem became even more difficult when macromolecule-ligand complexes are considered. Use of precipitators or cosolvents is a common technic. It is clear however that such additives can influence to macromolecular structure or intermolecular interactions between macromolecule and ligand. Unfortunately, there are no experimental ways to check whether crystallization condition affect 3D structure and whether macromolecule structure in crystal is the same as in solution or physiological media.

Computer simulation techniques are able however to answer this question. Having atomic coordinates from crystallographic experiments one can further change media and investigate structural changes. Molecular dynamics (MD) simulation is the most suitable method for this purpose. The aim of this work is to check an influence of solute nature and concentration on macromolecule structure and protein-ligand interaction energy. We choose SARS-CoV-2 Main protease as an example of macromolecule due to high interest in search of its inhibitors recent years. A test ligand was one of successful anti-covid drug candidates suggested recently [1]. A protease structure was obtained from PDB database (7NG6), docking in Vina software was used to generate complex coordinates. 100ns MD simulation in different media were carried out further. We choose 1,4-dioxane and DMSO as two popular cosolvents. MD models of ligand complex in pure water, ~5% and ~10% solutions of dioxane in water and same concentrations of DMSO in water were obtained. Common structural parameters of protein, such as radius of gyration, RMSD and secondary structure parameters were studied as well as H-bonds and interaction energy between ligand and protease. We found that parameters in different media indeed are not the same. Discrepancies are not high however and we can infer that solution composition has no critical influence to obtained structure.

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PP-61

Development Status of the 1-2 Beamline “Structural Diagnostics” at the SRF SKIF

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The progress of the project development and the time schedule of the new synchrotron beamline 1-2“Structural Diagnostics” at the SRF SKIF dedicated to high-resolution powder diffraction and macromolecular crystallography are to be presented. The beamline uses a cryocooled in-vacuum undulator (15.6 mm period, 1.25 T peak field) for photon generation together with refractive (CRL) optics and a cryocooled double-crystal monochromator (Si 111, 6-27keV) to deliver a high brightness focused monochromatic beam to the sample. The report will outline the development status of each component of the station, as well as its key parameters. In addition, a description of the techniques and equipment that will be available to users in the high resolution powder diffraction and macromolecular crystallography sections will be presented.

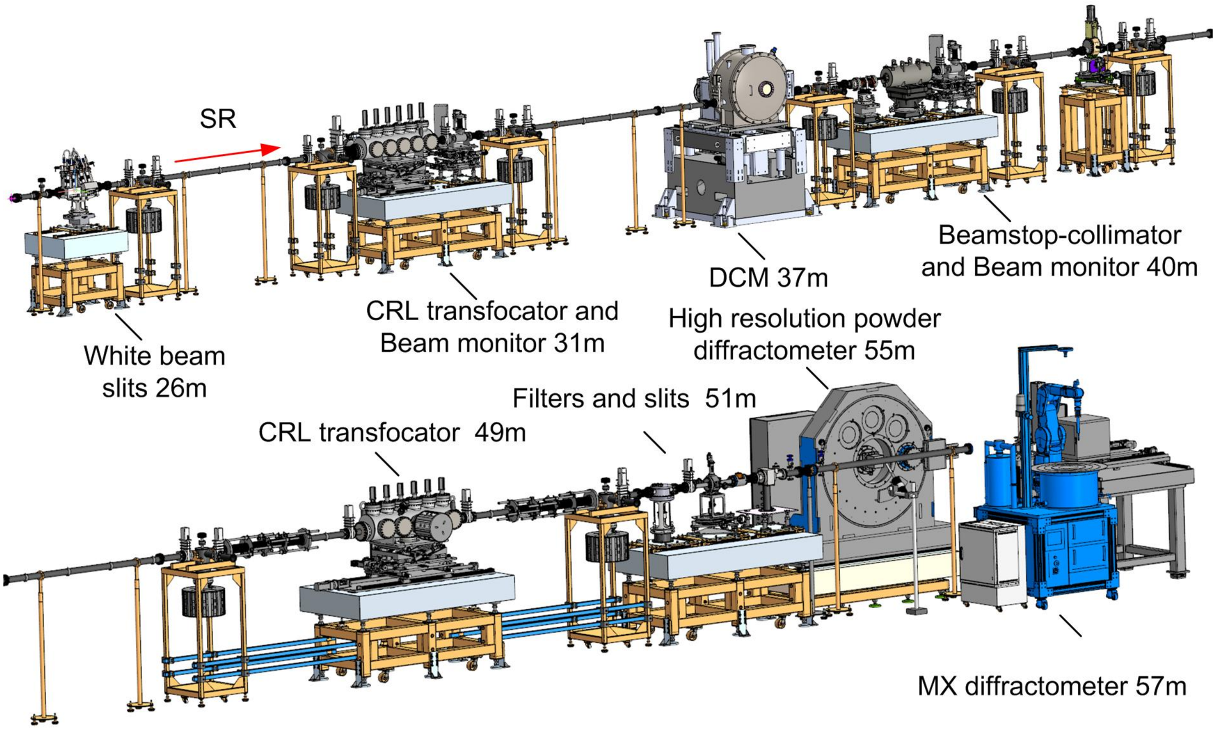

Fig. 1. 1-2 Beamline layout

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PP-62

Program for automated X-ray phase analysis of SR diffraction patterns in *in situ* experiments

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The study of *in situ* diffraction experiments on a synchrotron is associated with the processing of large volumes of synchrotron radiation (SR) data. In 2023, we developed and in 2024 upgraded an experimental software and methodological support (SMS) system for automated X-ray phase analysis of sets of diffraction patterns, known as "diffraction movies", which are obtained during materials science experiments on SR sources. The SMS allows *in situ* analysis of the phase composition, lattice parameters and microstructure of materials that arise when the temperature, reaction medium, etc. change during the experiment.

The current version of the automated XRD analysis system includes the following components:

a) a module for 3D and 2D visualization of a set of SR diffraction patterns for selection of informative ones for further phase analysis;

b) a module for phase identification, quantitative X-ray phase analysis using the multi-reflective reference intensity ratio (RIR) method and the sizes of coherent scattering regions using the Scherrer method, based on the IPS RFA program [1]. IPS RFA provides rapid analysis of informative diffraction patterns by the author's method of selective cluster phase identification;

c) a module of generation of templates for automatic Rietveld analysis based on phase identification data;

d) a module for automatic Rietveld analysis of quantitative phase composition, lattice and microstructure parameters for the flow of SR diffraction patterns;

e) a control module that provides storage of experimental data and execution of the above functions with this data.

The automated XRD analysis system allows implementing the following technique of on-flow analysis of SR diffraction patterns in real time.

1. Setting up the system based on the results of analysis of several preparatory experiments in different modes for further automatic analysis of the main experiment data.
2. Performing automatic analysis of the flow of SR diffraction patterns (1-2 minutes per 1 diffractogram) to provide the possibility of regulating the modes of the main experiment in real time (for the case of phase composition transformations commensurate in time).
3. Rapid visual graphical control of the results of on-flow analysis on the screen; in case of registration of significant unidentified reflections of new phases, go to point 1.

The capabilities of the system are discussed based on the results of the *in situ* analysis of SR experiments on the study of high-temperature phase transitions and multilayer coatings deposition using the vacuum electron-ion-plasma stand (VEIPS) developed at the Research Center for Beam-Plasma Engineering and Synchrotron Research of the IHCE SB RAS.

After the next version of the system is finalized, it may be in demand at diffraction stations of synchrotron radiation centers and their users.

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PP-63

Study of the effect of aliphatic monocarboxylic acids on carbamazepine polymorphism

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Polymorphism is a phenomenon in which the same solid can exist in the form of different crystal structures. The physico-chemical properties of different polymorphs of the same substance can differ radically from each other [1]. In this regard, it is important to study the properties of polymorphs and find new polymorphic forms, as well as improve existing methods for obtaining these forms. This is especially relevant in the case of pharmaceutical compounds, where polymorphism control is essential due to the possibility of undesirable forms forming during the manufacturing and storage processes. Sometimes, on the other hand, a metastable polymorph can be the desired product due to its superior characteristics.

The object of this study is carbamazepine, a psychotropic drug that is used in the treatment of many diseases of the nervous system. Currently, there are five polymorphic forms of carbamazepine known. The most stable form, in terms of its thermodynamic stability, is carbamazepine III. Various polymorphic modifications of carbamazepine may differ in the rate of dissolution, peak concentration of the drug and its bioavailability [2].

In this paper, the effect of additives of saturated monocarboxylic acids on polymorphic transitions of thermodynamically stable III form of carbamazepine into metastable II form is investigated. A significant increase in the rate of transition from carbamazepine form III to modification II was shown when using a catalytic amount (5 mole %) of carboxylic acids with a long carbon backbone of C16 and C18 in a mechanochemical experiment (palmitic and stearic acids, respectively). The change in melting temperatures of binary mixtures of carbamazepine-carboxylic acid in relation to the melting temperature of individual saturated carboxylic acids was estimated using the DSC method. Experiments on the dissolution of carbamazepine's stable form in fatty acid melts have shown the growth of needle-like crystals of the metastable form II. This suggests that it is possible to obtain this polymorph of carbamazepine without the need for mechanical action. A possible mechanism for the polymorphic conversion of carbamazepine (III) into carbamazepine (II) during processing with fatty acids and heating has been proposed.

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PP-64

A novel approach for synthesis of Pt/g-C_3N_4 photocatalysts for solar energy conversion

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The development of alternative energy sources has attracted considerable attention due to the growing demand for energy resources and the detrimental impact of greenhouse gases on the climate. Hydrogen is regarded as one of the most promising energy sources due to a number of factors, including its high combustion heat, its widespread occurrence in nature, and the absence of toxic products during combustion. Nevertheless, the prevailing techniques for the generation of hydrogen rely on the utilization of fossil fuels. A further environmental concern is the annual increase in CO_2 emissions, which have a detrimental impact on the atmosphere. The use of solar energy to produce synthetic fuels, including hydrogen and methane, has the potential to significantly contribute to the effort of addressing environmental issues. "Green" H_2 and CH_4 could be obtained from available substrate (H_2O, CO_2) under the light irradiation in the presence of semiconductor-based photocatalysts.

A promising new photocatalyst is graphitic carbon nitride (g-C_3N_4) due to its energy structure, stability, and lack of toxicity [1]. The unmodified g-C_3N_4 catalyst exhibits low activity in photocatalytic reactions, primarily due to the high recombination rate of photogenerated electrons and holes. A widely used approach for modifying g-C3N4 is the deposition of metal particles or their compounds on the surface of the semiconductor. The spatial separation of charges occurring at the semiconductor-metal interface or between two different semiconductors results in a significant increase in the lifetime of photogenerated electrons and holes. Of the various cocatalysts that are employed in photocatalytic hydrogen production, platinum and its compounds are the most commonly utilized.

The aim of this work was to study the influence of the method of synthesis of the Pt/g-C_3N_4 photocatalyst on the activity in the of H_2 evolution from an aqueous solution of glucose and in the CO_2 reduction reaction (CO_2 RR) under the visible light irradiation (400-440 nm). The synthesis of g-C_3N_4 was carried out by calcining melamine at 600 °C for 2 hours. An obtained sample of g-C_3N_4 underwent hydrothermal treatment at different temperatures (120 – 180 °C). After washing with distillate water and drying, platinum (1 wt.%) was deposited to the surface of g-C_3N_4 from a H_2PtCl_6 solution by reduction with a $NaBH_4$ solution. The activity of photocatalysts was studied in the reaction of H_2 evolution in a static reactor using a 0.1 M aqueous solution of glucose as a substrate under the LED irradiation (440 nm). The CO_2 RR was carried out in a static reactor in atmosphere of CO_2 (99.995%), ultrapure water (2 mL) was used as H^+ source. The results of the study of the activity of photocatalysts are presented in

Table 1. Hydrothermal treatment of g-C_3N_4 with H_2O_2 solution results in significant increase in photocatalyst activity.

Table 1. Photocatalyst activity in H_2 evolution from glucose aqueous solution (440 nm) and in CO_2 RR (400 nm).

T of hydrothermal treatment, °C	H_2 evolution	CO_2 RR		
	W (H_2), µmol h^{-1} g$_{cat}^{-1}$	W (CH_4), µmol h^{-1} g$_{cat}^{-1}$	W (CO), µmol h^{-1} g$_{cat}^{-1}$	W (H_2), µmol h^{-1} g$_{cat}^{-1}$
RT	0	0.4	0.3	2.0
120	189	0.2	0.1	8.2
140	310	1.7	0.3	5.6
160	266	2.8	0.2	7.2
180	0	1.1	0.2	5.9
140*	109	0.6	0.2	2.7

* hydrothermal treatment in distilled water

A set of research methods, including X-ray absorption spectroscopy, X-ray photoelectron spectroscopy, transmission electron microscopy, and X-ray phase analysis, were used to characterize the photocatalysts, thereby establishing that the partial destruction and oxidation of the g-C_3N_4 surface occur during hydrothermal treatment in a peroxide solution (Fig. 1).

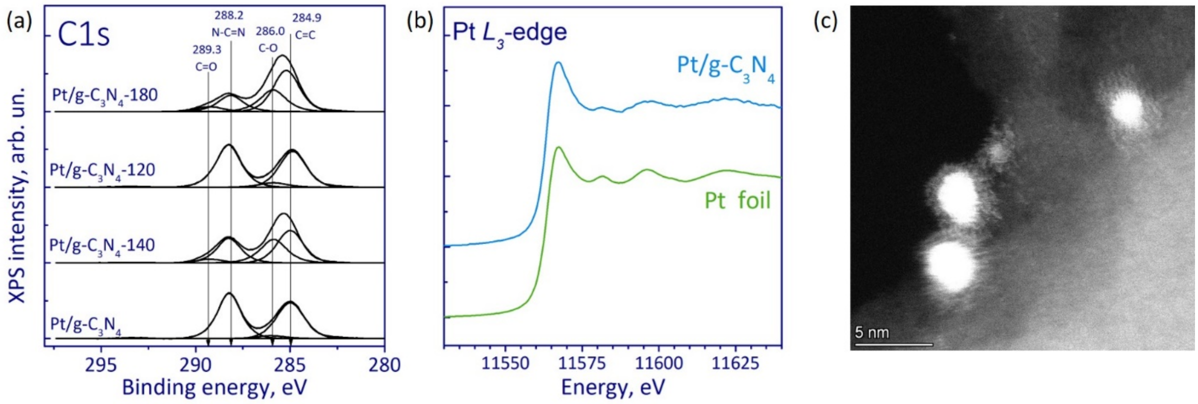

Fig. 1. (a) C1s core-level spectra of Pt/g-C_3N_4 photocatalysts, (b) XANES Pt L_3-edge spectra of Pt/g-C_3N_4 photocatalyst and reference (Pt foil), (c) HAADF STEM image of Pt/g-C_3N_4-140.

During the oxidation process, oxygen-containing functional groups are formed on the surface of g-C_3N_4, which facilitates the adsorption of reagents, including glucose, water, and carbon dioxide. The platinum particles are in a metallic state for all synthesized photocatalysts. Furthermore, a strong interaction between Pt and g-C_3N_4 was observed, which stabilizes the Pt nanoparticles. The work presents a simple synthetic route to increase the activity of g-C_3N_4-based photocatalyst.

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PP-65

Study of the structure of ceramic coating of the Y-Al-O system

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In this work, the structural and phase transformations occurring because of annealing of the Y-Al-O coating system were studied using synchrotron radiation. The coating was applied on an NNV 6.6 – I1 installation using the vacuum-arc method from two single-component Y and Al cathodes. After deposition of the coatings, the samples were annealed in a vacuum furnace with the following mode: heating at a rate of 10 °C/min to a temperature of 550 °C, then holding at this temperature for 30 minutes, then heating to a temperature of 800 °C and holding for 1 hour at a temperature of 800 °C. To study the qualitative phase composition and phase stability of the coating in real time during heating of the sample in vacuum to 1400°C, a synchrotron radiation source was used - the VEPP-3 electron storage ring of the Institute of Nuclear Physics, Siberian Branch of the Russian Academy of Sciences (INP SB RAS), a high-temperature X-ray camera Anton Paar HTK-2000, a position-sensitive single-coordinate detector OD-3M-350, software - the Fityk v.1.3.1 program for processing the measurement results. Studies of the structural and phase state during vacuum annealing showed that in the initial state the coating is an amorphous structure - a smooth increase in the diffracted intensity with a maximum in the region of angles 2θ ~ 29 ... 31 ° is observed in the X-ray diffraction pattern. Individual reflexes are recorded, which belong to yttrium and aluminum. During heating of the sample to a temperature of 1400 °C, a change in the phase composition of the coating is observed in a series of X-ray diffraction patterns, which begins at a temperature of ~ 1200 °C. The amorphous component disappears, and predominantly the phases of mixed oxide YAlO3 and yttrium oxide Y2O3 appear. Reflexes related to phases containing titanium or carbon are still not observed. No further changes in the phase composition of the coating occur during the holding of the sample at a temperature of 1400 °C. The results of the study of the change in the phase composition during vacuum heating of the YAlO coating showed that for complete crystallization of the coating and the decomposition of secondary phases, the required annealing temperature is 1200º C. In this case, the phase composition of the coating will be represented predominantly by the required phase YAlO3 with a small content of the phase Y2O3.

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Content

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Abstracts

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