

# Synchrotron and Free electron laser Radiation: generation and application (SFR-2018)

June 25 - 28, 2018

Budker INP

## Book of Abstracts

Соорганизаторы: ИЯФ СО РАН и НГУ



**N\*** Novosibirsk  
State  
University  
\*THE REAL SCIENCE

Новосибирск  
2018

УДК 535.33/.34; 538.97-405  
ББК 22.344; 22.386

**Book of abstracts** of International Conference "Synchrotron and Free Electron Laser Radiation: Generation and Application".

**Книга абстрактов** Международной конференции по генерации и использованию синхротронного и терагерцового излучения.

**ISBN 978-5-904968-06-9**

Настоящий сборник абстрактов является рабочим документом Международной конференции по генерации и использованию синхротронного и терагерцового излучения SFR-2018 (ИЯФ СО РАН, г. Новосибирск, 25 - 28 июня 2018).

Книга предназначена для научных сотрудников и инженеров, занимающихся исследованиями на синхротронном, терагерцовом излучении, разработкой элементов ускорителей заряженных частиц, а также может быть полезной студентам и аспирантам соответствующих специальностей.

ISBN 978-5-904968-06-9

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## New SR beam line at VEPP-2000 complex on a service of FCC and LHC

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Electron beam induced synchrotron radiation and kept near equilibrium orbit by strong electrical field of proton bunches changes focusing forces, which act on the proton beam and reduce the lifetime of protons. On this moment, a-C material coated on an inner chamber wall is the most perspective material for minimization of SEE. In the framework of the collaboration between BINP and CERN for FCC and upgraded LHC, it is necessary to investigate the photon-stimulated desorption and secondary electron emission for Cu vacuum chamber with amorphous carbon coating at temperature range from 60 K to 300 K.

1

## Terahertz localized surface plasmon modes on subwavelength metal disks

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Using of structures on which multipole localized surface plasmon resonances can be excited is promising for the developing of terahertz (THz) biosensors. In this paper, the surface plasmon resonances on corrugated metal disks in conjunction with a C-shaped resonator in the THz range using numerical simulations have been studied. The parameters of the structures for which THz multipole resonances are excited were found.

2

## SSRF beam line phase II project

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In SSRF beam line phase II project there are 16 beam lines will be built. Besides, the storage ring will be improved. Two of the cells will be modified by using superbends to create two short straight sections. Two long straight sections will modified to double mini beta\_y function lattice by adding a triplet in the middle for each. Also a superconducting 3rd harmonic cavity will be added to lengthen the bunch length, several types of insertion devices are developed to provide radiations under users' requirement.

3

## Chemical structure and molecular arrangement in electrodeposited polypyrrole layers doped with dodecyl sulfate anions: NEXAFS and XPS analysis

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Electrodeposited films of polypyrrole are widely used in many technological applications, including anticorrosive protection of metal and steels [1]. It is known that the protective effect of coatings can be significantly improved by doping the polymer with alkyl sulfates or alkyl sulfonates, for example, dodecyl sulfate anions [2]. In this work, polypyrrole films were grown by electropolymerization of pyrrole from aqueous solutions in the presence of dodecyl sulfate additives. For the first time, the chemical structure and spatial arrangement of the molecules in the as-grown polymer films were studied by means of XPS and angular-dependent NEXAFS spectroscopy. The spectra were measured at the Russian-German beamline at BESSY II (HZB, Berlin).

It has been found that the films are characterized by one dodecyl sulfate anion for each nitrogen atom. One-fourth of all the nitrogen atoms are in radical cations, with the rest atoms being in the protonated amino groups. This structural feature leads to almost complete disappearance of  $\pi^*$  resonances in the nitrogen K edge NEXAFS spectra. The films grow through 2D growth mechanism and comprise of thin plates preferentially oriented in-plane substrate surface. Linear dichroism of the NEXAFS spectra was observed for these films. The changes observed in the spectra indicate the presence of 2D-polypyrrole sublayers with pyrrole rings lying in-plane the substrate and sub-layers of surfactant anions with their tails oriented upright the substrate. High anticorrosive properties of the doped polypyrrole films may arise from the revealed structural and molecular arrangement features.

This work was supported by Russian Foundation for Basic Research (No.16-43-180228), bilateral Program “Russian-German Laboratory at BESSY II” and partially by FASO of Russia within the state assignment No. -17-117022250038-7.

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4

## Linear dichroism of X-ray absorption spectra and molecular arrangement in electrodeposited polyaniline and polypyrrole films

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Conductive polymers are used in practice as materials for electrochemical devices (batteries, fuel cells, supercapacitors), anticorrosive protective coatings and electromagnetic radiation protective layers. The properties of such materials depend on not only chemical structure of the polymer, but also on the spatial arrangement of the chains being charge conductors. But the spatial arrangement of the chains in functional films of conductive polymers and its effect on the physicochemical properties of polymers is far from being comprehensively studied and understood. Diffraction methods turn out to be inapplicable to analysis of such materials because of their relatively weak crystallinity. In this work, molecular arrangement in electrodeposited polyaniline and polypyrrole films was studied with use of linear dichroism of x-ray absorption spectra (NEXAFS) measured at both nitrogen and carbon K absorption edges. The spectra were measured using synchrotron

radiation at the Russian-German beamline at the BESSY II (HZB, Berlin). The pronounced dichroism of the x-ray absorption spectra was observed for polypyrrole films. When going from the normal to the grazing angle of the exciting radiation incidence, the  $\pi^*$  resonances grow, with  $\sigma^*$  resonance intensity simultaneously decreasing. This indicates preferential arrangement of most pyrrole rings in-plane the substrate. Dichroism is stronger for initial stages of deposition characterized by 2D growth of films, as compared with later deposition stages, at which the films have "cauliflower"-like morphology. In the case of polyaniline, the grown films have different morphology depending on the electrodeposition conditions and seen as either smooth homogeneous films (in stirring electrolyte) or rough non-homogeneous films containing polymer particles deposited on the surface (quiescent electrolyte). The pronounced dichroism was observed only in the second case. The observed dichroism was stronger for the films containing more polymer particles deposited on the film surface. For such samples, the spectra at both absorption edges exhibited weaker  $\pi^*$  resonances, indicating predominantly up-right arrangement of the benzene rings in the particles. Changes observed in other resonances allow suggestion about in-plane orientation of the polymer chains themselves in the particles relative the substrate surface. This work was supported by Russian Foundation for Basic Research (No.16-43-180228), bilateral Program "Russian-German Laboratory at BESSY II" and partially by FASO of Russia within the state assignment No. -17-117022250038-7.

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## Formation of catalytically active nanoparticles on surface of various supports.

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Fine metal and oxide particles supported by various oxides are an important class of catalytic systems. The surface of oxide support can strongly affect the degree of dispersion, structure and catalytic properties of supported nanoparticles. Structural characterization is known to play key role in understanding of catalyst structure-activity relationships. The most interest is in the structure of catalytically active nanoparticles. However, traditional routine XRD methods are usually ineffective for this study. The contribution to XRD pattern originating from small fraction of active component is insignificant in most cases. Moreover, diffraction patterns of highly dispersed materials are characterized by strong background and broadened peaks. Radial distribution function (RDF) of electronic density or atomic pair distribution function (PDF) method is effective for studying the local structure (short range atomic arrangement) of nanoparticles. This method is based on the Fourier relationship between intensity of coherent X-ray scattering and RDF of electronic density [1-3]. Using this technique, one can directly define interatomic distances and coordination numbers of atomic arrangement. The PDF method is used to determine: 1) phase analysis of nanomaterials; 2) changes in the structure associated with the particle size; 3) particle sizes <2 nm; 4) structural defects; 5) the structural aspect of the fixing of nanoparticles in the matrix. To determine features of the local structure of supported nanoparticles as against well crystallized analogues a comparison of the experimental RDF and the model one constructed on the basis of known structural data is used [4]. In this work we report a comparison of the possibilities of methods PDF and EXAFS and some examples of the RDF analysis application: 1) to determine the structure of highly dispersed active component in supported catalysts 2) to elucidate structural aspects of interaction between the support and active component. The Au(Pt)/ $\gamma$ -Al<sub>2</sub>O<sub>3</sub>/C/ SiO<sub>2</sub> catalysts with different metal content were considered. RDF data strongly suggested an epitaxial interaction between supported metal Au and Pt particles and the surface of support (Al<sub>2</sub>O<sub>3</sub>). Such interaction may be a reason for high thermal stability of supported metallic nanoparticles. The catalyst Pt/SiO<sub>2</sub> was studied by the X-ray Diffraction Radial Electron Density technique and EXAFS spectroscopy. It was found that the sample, kept in air and additionally untreated, contains the phases of metallic platinum Pt<sub>0</sub> and oxide platinum PtO (~1:2). The EXAFS data were analysed, assuming three platinum particle models. Model 1 contains the shortest Pt-Pt distance, which is the same for the particle bulk and its surface. Model 2 has two different short distances for the particle bulk and its surface.

Model 3 additionally has two different Debye-Waller factors for the particle bulk and its surface. It was shown that the second model is more correct for the oxidised sample and the Pt-Pt distance between surface atoms is shortened by  $\sim 0.14\text{\AA}$ . For reduced samples, the obtained structural data favour third model. The Cu/ZrO<sub>2</sub> catalysts with tetragonal (t) and monoclinic (m) zirconia were considered. RDF analysis revealed that copper (II) oxide chain clusters network were the main copper species, while particles with CuO bulk structure were not formed significantly. The obtained data were in agreement with the incorporation of some copper ions into zirconia lattice. RDF data also suggested interaction of active component with zirconia surface in Cu/m-ZrO<sub>2</sub> catalysts, which efficiently stabilized small CuO particles. The model of epitaxial growth of CuO particles on certain planes of m-ZrO<sub>2</sub> was proposed.

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## SRXRF study on the ecological state of technotronic surroundings in the Novosibirsk region

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The method of X-ray fluorescence analysis involving synchrotron radiation (SR XRF) has been first used at the station of element analysis of the SSTRC of the Institute of Nuclear Physics (VEPP-3 store ring) to analyze the content and composition of elements in soil and plant samples growing in places with the different level of pollution in the Novosibirsk region. The plants *Hemerocallis hybrida* (sort Speak to me) growing near the industrial enterprises and highways of Novosibirsk, urban-type locality Koltsovo, and the town of Berdsk were the objects of a given study. The territory of the Central Siberian Botanical Gardens was taken as a control site. A comparative element analysis was performed both for the leaves and rhizomes of *H. hybrida*, collected at the end of vegetation at the control and seven experimental sites, and for the samples of soil on which the studied plants grew. The SR XRF method provided reliable data on the composition and content of no less than 20 elements of ground and underground plant organs and of the soil from the sampling sites. The peculiarities of the composition and content were revealed in the elements of the vegetation organs of *Hemerocallis hybrida*, growing under a technotronic impact depending on the level of pollution. It has been established that the total content of microelements in the leaves and rhizome of the plants from the most polluted regions around industrial enterprises and along highways increases 3.5-6.2 times as compared with control. It is shown that the values of the ratios of the key elements Fe/Mn, Ca/Fe, Zn/Cu, and Sr/Ca change substantially under technotronic stress. The level of the accumulation of micro- and macroelements was determined to depend on the peculiarities of plant organs and on the polluting strength of the region, in which they grew. It has been established that Pb, Ni, Zn, Fe, Ti, and Cr are the main elements-pollutants accumulated in excess concentrations in the leaves and roots of the plants growing in the zones of strong traffic. Using *Hemerocallis hybrida* we have estimated the ecological state of the territories with the different level of industrial and transport pollution in the Novosibirsk region. It is demonstrated that *Hemerocallis hybrida* of the Speak ty me sort can be used as a bioindicator of industrial and transport pollution.

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## Stability of hydrous silicates and carbonates as a part of H<sub>2</sub>O and CO<sub>2</sub> cycle in deep Earth: the high-pressure diffraction studies

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The global cycle of major volatiles such as H<sub>2</sub>O and CO<sub>2</sub> remains among the key topics of modern petrology. In particular, active interest is taken in water recycling associated with subduction of oceanic plates into the mantle. Mineral serpentine, Mg<sub>3</sub>Si<sub>2</sub>O<sub>5</sub>(OH)<sub>4</sub>, is shown to be the main water reservoir in oceanic lithosphere [1], and therefore its dehydration produces a large impact onto seismic activity and magma generation in subduction zones. Since the dehydration temperature can be effectively decreased in the presence of alkali chlorides [2], we aim to estimate this effect for serpentine dehydration. Here we characterize the decomposition of serpentine in the reference salt-free system, based on in-situ HP-HT diffraction (SSTRC, Novosibirsk) and Raman spectroscopic studies in diamond anvil cell. The global carbon cycle includes, besides outer shells of the Earth, large-scale reservoirs of the Earth's mantle and core. Earlier the transport of oxidized carbon into the mantle was considered solely within the system CaCO<sub>3</sub>-MgCO<sub>3</sub>-FeCO<sub>3</sub>. Recent findings of Na-Ca carbonates in deep-seated rocks and experiments suggest the participation of these compounds in the formation of deep sodium carbonatite melts and makes them important objects of modern geochemistry. We report on the compressibility of some of the natural (mineral shortite) and synthetic (nyerite) Na-Ca carbonates up to 100-120 kbar as the first step of determination of their PVT equations of state. This work is supported by the state assignment project (0330-2016-0004), Russian Foundation for Basic Research (grant No 18-05-00312) and the Ministry of Education and Science of Russian Federation (project RFMEFI62117X0012). [1] Rüpke et al. (2004) *Earth and Planetary Science Letters*, 223(1), 17-34. [2] Aranovich and Newton (1997) *Contributions to Mineralogy and Petrology*, 125(2), 200-212.

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## Superconducting 22-pole 7 Tesla wiggler for DELTA synchrotron radiation source

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The Dortmund Electron Accelerator (DELTA, Germany) operated as 1.5 GeV synchrotron radiation source requires a superconducting wiggler as an insertion device for three x-ray beamlines with photon energies up to more than 30 keV. The beamlines are separated each by 15 mrad from the central one and each beamline uses a horizontal aperture of 5 mrad. To meet these requirements the insertion device must have a period of 127 mm and a magnetic field of 7 Tesla for a vertical aperture of beam vacuum chamber of 10 mm and the length from flange to flange of 2.2 m. The superconducting 22-pole wiggler with the field of 7 Tesla and with the period of

127 mm operated with zero boil-off mode are described in this article. The conception and main approaches for the design of the magnetic and cryogenic system as well as the main parameters and the test results of new 7 Tesla superconducting wiggler for DELTA synchrotron light source are presented.

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## The upgrade of the superconducting wiggler magnet installed at the ELETTRA storage ring.

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The Superconducting multipole Wiggler (SCW) originally manufactured by BINP (Budker institute of nuclear physics at Novosibirsk) was installed at the ELETTRA storage ring (Triest, Italy) in 2002. In 2013 upgrade of wiggler cryostat was made by BINP to reduce liquid helium consumption. From June 2013 to January 2017 the wiggler functioned without problems of helium consumption (although it quenched 12 times). In January 15, 2017 while there were preparations for setting the accelerator at 2.4 GeV for the users the SCW was damaged by quench. The upgrade of wiggler magnet was proposed by BINP. The magnet was reconstructed in 2017. The new coils have been made with reduced quantity of winds. This coil design is more close to optimum from point of view current-field relationship for superconducting wire and also decreases stored energy. In January 2018 the reconstructed magnet has been assembled into cryostat and installed at the ELETTRA storage ring.

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## Spontaneous coherent cyclotron THz super-radiation from a short dense electron bunch

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Laser-driven photo-injectors allow formation of compact and accessible sources of dense electron bunches with a moderate energy of 3-6 MeV, picosecond pulse durations, and charges of up to 1 nC and even greater. These bunches can be used for realization of relatively simple and compact terahertz sources operating in the regime of spontaneous coherent radiation. This type of radiation is realized, when the effective phase size of the electron bunch with respect to the wave is small enough, so that the wave packets emitted by each of the electrons add up basically in phase. THz sources based on the spontaneous emission have a number of advantages as compared to the more traditional free-electron laser (FEL) based on the undulator emission induced due to bunching of a long electron beam by the radiated wave. First of all, an evident advantage is a relatively high efficiency of the energy extraction from electrons, which can be achieved in a simple microwave system based on the “ready-for-radiation” bunch. Actually, such an oscillator does not require either a wave feedback system or an input wave signal to provide the high-efficiency stimulated character of the radiation process. A high-efficiency together with a narrow frequency band of the radiated rf signal is provided in a relatively short and simple system (namely, just amplifier-like waveguide system). One more important advantage is that the phase of the radiated rf signal is fixed by the electron bunch phase. A key problem in realization of THz source based



on the spontaneous coherent emission from a short electron bunch is a strong Coulomb repulsion in dense bunches, which leads to the increase of the axial bunch size. If the “operating” radiation mechanism of the THz source is based on the longitudinal electron bunching (either ubitron or cherenkov masers), then axial expansion of the bunch leads automatically to an increase in the bunch phase size with respect to the radiated wave and to the stopping of the spontaneous emission process. Naturally, in the cyclotron maser, the Coulomb repulsion also provides an increase in the axial size of the bunch. However, we show that in cyclotron masers this problem can be solved by the use of the group resonance regime, where the group velocity of the radiated wave is close to the axial electron velocity. In this case, the effect of compensation of the Coulomb repulsion in the phase space takes place. This means that the increase in the axial size of the bunch caused by the Coulomb repulsion does not lead to an increase in the phase size of the bunch, so that the cyclotron phases of particles with respect to the radiated wave stay almost constant. This effect is useful also because the group resonance regime is very attractive from the viewpoint of organizing the wave emission process. This is due to the fact, that in this situation an effective super-radiation regime is realized. In this regime, the radiated wave doesn’t “run away” from the electron bunch, which leads to accumulation of the radiation in the region close to the bunch and, as a result, to formation of a powerful short wave pulse propagating together with the bunch.

The work is supported by Russian Foundation for Basic Research Project 18-32-00351

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## Resonant interatomic Auger transitions

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The interatomic Auger transitions in the presence of core electron levels with close energies are studied theoretically and experimentally. We show that the Coulomb transitions of holes between such levels lead to a resonance (in terms of the energy difference) enhancement of the Auger spectra.

!http://yadi.sk/i/YWf\_QF-p3UaX2w

!https://yadi.sk/i/UgVO7wyP3UaW8k

Figure 1. Diagram of the interatomic Auger process Cu<sub>2</sub>pCu<sub>3</sub>pIn<sub>4</sub>d (a) and Cu<sub>2</sub>pIn<sub>4</sub>pIn<sub>4</sub>d (b) on atoms with interacting internal levels.

Figure 2. The interatomic Auger spectrum Cu<sub>2</sub>p<sub>3</sub>pIn<sub>4</sub>d and the 2pVV copper spectrum in the CuInSe<sub>2</sub>.

We use the CuInSe<sub>2</sub> compound to illustrate our approach. Figure 1 shows a diagram for the amplitude of interatomic Auger transitions Cu<sub>2</sub>pCu<sub>3</sub>pIn<sub>4</sub>d and Cu<sub>2</sub>pIn<sub>4</sub>pIn<sub>4</sub>d. The matrix element of the interatomic Auger transition is usually very small, however, we show that the Coulomb interaction of the levels of neighboring atoms leads to a multiplication of the Auger spectrum intensity (without interaction) by the enhancement factor  $F = \frac{(W/\Gamma)^4}{(E_2 - E_1)/\Gamma^2 + 1}$ . Here  $W$  is the matrix element of the Coulomb hole transfer between the states of atoms 1 and 2 (Cu<sub>3</sub>p and In<sub>4</sub>p) with energies  $E_1$  and  $E_2$  and half-width at half maximum of  $\Gamma$ . It is important that the factor  $F$  resonantly increases with decreasing energy level difference. The experimental spectrum in Fig. 2, obtained on the Russian-German synchrotron line BESSY-II at photon energies of 1200 and 1210 eV, shows the intense line of the interatomic Auger transition. Binding energies Cu<sub>3</sub>p / 2 eV (75.1 eV) and In<sub>4</sub>p (73.5 eV) differ by only 1.6 eV, so the half-width 1.5 eV Coulomb transfer holes between levels results in a significant enhancement of the intensity. It can be seen that the Cu<sub>2</sub>p<sub>3</sub> / 23pIn<sub>4</sub>d spectrum contains three peaks of 830, 838 and 846 eV arising when the orbital angular momenta  $l = 1$  and 2 of the copper and nickel holes are added. A spin-orbit replica of 858, 866 eV also shows the transition originating from Cu<sub>2</sub>p state with a total angular momentum  $j = 1/2$ .

Thus, the resonance enhancement of the cross section at close energy levels on neighboring atoms makes it possible to observe interatomic Auger transitions in the x-ray range. This work was supported by the RNF (project No. 17-12-01500).

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## Element composition of the mountain Altai plants

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A study has been first made of the element composition of both the plants of six species from three families, growing in the Mountain Altai and the soil samples from their habitat by method of X-ray fluorescence analysis using synchrotron radiation (SRXRF) at the station of element analysis of SCSTR of the Institute of Nuclear Physics SB RAS (VEPP-3 store ring). The material studied was represented by the plants of six species of three families, collected in July of 2017 in the South-East and Central Altai. *Potentilla fruticosa* and *Sibiraea altaiensis* of the Rosaceae family, *Myricaria longifolia* of the Tamaricaceae family. The plants *Caragana* of the Fabaceae family are represented by three species - *Caragana bungei*, *Caragana pygmaea* and *Caragana pygmaea* subsp. *altaica*. We have analyzed the leaves and stems of bushes and soil samples of four habitats of the plants studied. Reliable data were obtained on the content of 20 elements in plant and soil samples. A comparative analysis of soil samples from the different habitats of the Mountain Altai indicate that the content of most of the elements varies steadily within a narrow range ( $\nu \leq 2,5$ ), calculated as  $\nu = C_{max}/C_{min}$ . The greatest variability was recorded only for Br, As ( $\nu > 5$ ) and Mo ( $2,5 \leq \nu \leq 5$ ). The highest total content of K and Ca is revealed in the leaves and stems of the representatives of *Caragana* and in the leaves of *S. altaiensis*. The high total content of microelements was found in *C. pygmaea* and *C. pygmaea* subsp. *altaica*. Considerable differences were recorded in the accumulation of elements in the aerial organs of the bushes studied. In the leaves, the content of Mn varies within a very narrow range ( $\nu \leq 1,5$ ); that of Pb varies within a narrow range ( $\nu=2,3$ ); the content of Cr, As, Ca, Zn, Cu and K varies, on the average, in the mean range ( $2,5 \leq \nu \leq 5$ ), the content of the rest elements varies within a large range. In stems, the concentration of Cu varies within a short range ( $\nu=1,8$ ), Mn, Zn, Pb and K – within the average one, the others – within a wide range. The high content of Ca, Fe, Sr, Mn, Zn, Ti, Zr, Mo and Co is determined in the leaves and stems of the plants *Caragana*, mainly *C. pygmaea* subsp. *altaica*; the high content of Y, Rb, Br, Cu, Cr and Ni is found in the leaves of *M. longifolia*, K – the leaves of *S. altaiensis*. Thus, it has been established that the highest accumulation of macro- and microelements is typical of the representatives of *Caragana* of the Fabaceae family, the low content is typical of the plants *Potentilla* and *Sibiraea* of the Rosaceae family. The content of most of the chemical elements in aerial organs of the representatives of various taxons varies at high level. The data obtained on the content of 20 elements are reliable and may be included in databases.

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## Status of the FELBE THz/IR FEL Facility: Overview of the Machine Performance and User Activities

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The FELBE User Facility at the ELBE Center for High Power Radiation Sources offers two FELs dedicated to optical studies of materials over a wide range of the THz and IR spectrum (1.2 – 60 THz / 5 – 250  $\mu\text{m}$ ). Driven by a CW SRF linac, the FELBE FELs deliver beam at 13 MHz to a suite of eight User Labs. The high rep. rate CW operation provides high average power as well as outstanding data statistics, resulting in excellent S/N for even very challenging measurements. For example, the SNOM Group at the Technische Universität Dresden has pioneered methods using the tunable FEL beam with scattering scanning near-field infrared microscopy (s-SNIM) to achieve spectrally resolved nanoscale images with sub-wavelength resolution[1, 2]. While nanoscale imaging is gaining interest, the ultrashort pulses have long been the most important feature for most FELBE Users performing time-resolved measurements of transient processes in 2-dimensional materials[3], nanostructures[4], and correlated systems[5]. High field studies are also possible utilizing either an 8 T DC magnet or the 70 T pulsed field magnet at the adjacent Dresden High Field Magnet Lab[6, 7], and nonlinear processes can be driven by the  $\mu\text{J}$  level pulse energy of both FELs.

Work is constantly underway to improve the machine, beam delivery, and the User Labs; and in 2017, a major upgrade was completed with the installation and commissioning of a new U37 undulator. This resulted in enhanced performance over the tuning range of the new FEL (7.5 – 60 THz / 5 – 40  $\mu\text{m}$ ), and User operation of the new U37 FEL began during the second half of 2017. A description of the performance and operational aspects of both FELs will be presented, with an emphasis on the commissioning measurements of the new U37 FEL. An overview of the FELBE User program, instrumentation, and infrastructure will be given along with highlights of several key User results.

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Up to now free-electron lasers are much powerful generators in THz-band able to operate at multi-megawatt power level. Such devices are driven by relativistic electron beams (REBs) of  $\sim 5$  - 10 MeV oscillating in magnetostatic undulators and, thus, are rather bulky and costly. At the same time, generation of powerful THz radiation can be realized when using moderately-relativistic (0.5 - 1.5 MeV) high-current (1 - 3 kA) electron beams together with intense mm wave to pump transverse oscillations of the electrons (so-called RF-undulator), which could possess much shorter period (wavelength) in comparison with “traditional” magnetostatic undulators (with additional twice higher Doppler up-conversion in counter scattering scheme). In FEL of such type (FEL-scatterron) it is attractive to exploit an inter-cavity scattering regimes when pumping wave is generated by the same or parallel electron beam and, thus, no additional powerful microwave sources is needed.

Original project of multi-MW long-pulse two-stage FEL-oscillator based on two parallel sheet REBs is under development at the high-current accelerator ELMI in collaboration between BINP RAS (Novosibirsk) and IAP RAS (N.Novgorod). The basis for this project is 75 GHz planar FEM with two-dimensional distributed feedback, which was elaborated currently with a record-level output power ( $\sim 50$  MW) and narrow-band spectrum. In the two-stage scheme, this 75 GHz FEM module is used as a driver (i.e. the first stage of the oscillator). This 75 GHz radiation is transported by special waveguide (set of the Bragg deflectors) to the second channel and used as a pumping wave. At the second stage this wave undergoes stimulated scattering at the supplementary REB to produce THz radiation.

According to simulation, proposed scheme allows THz radiation pulses of  $\sim 10$  - 20 kW power to be obtained in the so-called SASE-regime. Installation in the FEL-scatterron section of advanced Bragg resonator to provide feedback loop for THz radiation, would result in increase of the output power of the THz channel up to a multi-megawatt level. As a result, energy content of up to 1 - 10 J in the THz-band pulses of hundreds nanoseconds to microsecond pulse duration can be achieved.

Key components of the electrodynamic system for two-stage FEM were manufactured and good coincidence of their “cold” tests with results of simulations was demonstrated. Experimental studies of this FEM scheme is in progress currently.

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## Free Electron Laser Based on a Multi-Stage System of RF Wigglers

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One of the key problems in realization of short-wavelength FELs is a small efficiency determined the so-called FEL parameter, which is typically  $\sim 0.001$  for existing X-ray FELs. Evidently, a similar condition limits the spread in initial energy of particles forming the operating electron bunch.

A well-known way for a significant increase in the FEL efficiency is the use of tapered undulator and realization regime of trapping and adiabatic deceleration of electrons [1-3]. In this approach, the undulator parameters (period and/or undulator field) are profiles so that the resonant energy [the electron energy corresponding to the exact resonance] decreases slowly with the increase of the axial coordinate. A fraction of electrons is trapped by a potential well created by the

field of the combination wave, end energies of the trapped particles are decreased together with the resonant energy. In principle, it is possible to exceed the FEL parameter limitation on the basis of this approach. However, it is difficult to provide trapping of a significant fraction of the electron beam in the case of a significant energy spread, as only electrons which are close enough to the resonance with the wave can be trapped.

In this work, we develop an idea of the “non-resonant” trapping regime [4,5], which, in principle, can provide an effective trapping of an electron beam with a great energy spread. In this case, at the beginning of the interaction the resonant energy is not close to the averaged initial electron energy but significantly higher. Different energy fractions of the beam are trapped in different points of the electron-wave interaction region, where their initial energy becomes close to the resonant energy. However, such non-resonant trapping is effective, if the wave is amplified fast enough during the trapping process (when the FEL parameter is big enough). We propose to avoid this difficulty by means of the use of a multi-stage (or “multi-pulse”) character of the trapping process, when the non-resonant trapping it provided several times in several consecutive undulator sections. In each act of the trapping (in each section) only a relatively small fraction of the electron beam is trapped by the amplified wave and pass their energy to the wave. However, repetition of this process from section to section involves in the electron-wave interaction almost all particles of the beam. As a result, an great electron efficiency (significantly exceeding the FEL parameter) can be achieved for an electron beam with a big (in the scale of the FEL parameter) spread in initial energy.

An important feature of the “multi-stage” trapping regime is the following: when the electron beam enters into a new undulator section, the trapping process starts from the very beginning. This means that any phase correlation between undulator sections is not needed. In the case, when every operating section represents an rf undulator, this means that every undulator section can be driven by independent (non-synchronized) rf-power sources.

As an example of the rf undulator, we consider the so-called “flying” undulator [6]. This is an rf pulse co-propagating with the electron beam in a helical corrugated waveguide. A benefit of the Doppler up-shift of Compton scattering is not lost due to the mode having strong -1st spatial harmonic transverse fields at axis of the corrugated waveguide. A 30 GHz, 10 ns, 1 GW relativistic backward wave oscillator can power a 10 m long rf undulator with effective undulator strength  $K = 0.3$  and the effective undulator period [U+F0BB] 5 mm. Multi-stage trapping is appealing for XFELs driven by high-current bunches having large energy spread. The laser-plasma accelerator presently provides electron beams with a typical current of a few kA, a bunch length of a few fs, energy in the few hundred MeV to several GeV range, and energy spread of 1%-10% [7]. In simulations, we consider an example of XFEL on a base of the described principles with a 250 MeV, 100 pC electron bunch of 20 [U+F06D] m length and 5 [U+F06D] m radius aimed to produce radiation at [U+F06C] [U+F0BB] 11 nm wavelength. The energy spread is 1%-3%. Simulations predicts a possibility to achieve the saturated efficiency of several percent, which it is several times more than the FEL parameter of this system.

The work is supported by IAP RAS Project 0035-2014-0012, and Russian Foundation for Basic Research Project 18-02-00765.

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## THz undulator radiation of stabilized dense electron bunches

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Laser-driven photo-injectors are capable to form very compact and dense electron bunches with a particles energy of 3-6 MeV, picosecond and subpicosecond duration, and charge of the order of 1 nC. When moving in the periodic field of the undulator with a period of a few centimeters, such bunches can generate coherent radiation in the terahertz range. The power and duration of such a generation is limited by spreading of the electron bunch under the action of the Coulomb repulsion, which under normal conditions does not allow for the effective implementation of such a scheme. However, it is shown in [1] that providing of a special configuration of the magnetic field, when a so called regime of negative electron mass is realized in the system, can significantly slow down the Coulomb repulsion.

The negative-mass regime of the electron motion is realized, when the electron moves in a combination of periodic undulator field and relatively strong homogeneous longitudinal magnetic field, and the cyclotron frequency corresponding to the longitudinal field is slightly higher than the undulator bounce-frequency of the particle. Then increase of the electron energy will lead to a reduction of its cyclotron frequency, since the latter is inversely proportional to the relativistic mass-factor of the particle. The electron approaches the undulator-cyclotron resonance, which is accompanied by a resonant increase in its transverse velocity. When it is close enough to the resonance, such a transverse velocity pumping occurs due to decrease of the longitudinal electron velocity. Thus, increasing the energy of the particle causes it to slow down in the longitudinal direction, which can be regarded as a consequence of its effective mass being negative. With regard to the dynamics of the electron bunch, it means that the Coulomb force leads not to the repulsion but to the attraction of electrons.

According to simulations, in the negative-mass regime the radiation power is greatly increased and its frequency band is significantly narrowed. For example, one can propose a rather prospective design in which the electron bunch with the particles energy of 5.5 MeV (mass factor of 12) scatters the undulator field having a period of 2.5 cm into the wave with a frequency of about 2 THz. In this case, the resonant cyclotron frequency corresponds to the longitudinal field of about 5 T, and effective negative mass regime is realized at the longitudinal field from the range near 8 T and the undulator field amplitude of about 0.2 T. Simulations predict formation of a  $\sim 10$  MW /  $\sim 20$  ps pulse with a very high electron efficiency (up to 20%), and with a possibility for the frequencies ranging from 1 to 3 THz.

Effective coherent undulator radiation is possible only when the longitudinal size of the bunch does not exceed the radiation wavelength. If the duration of the initial bunch exceeds this wave period, it should be pre-compressed. The simulation shows that the electron attraction in the Coulomb field of the bunch in the negative mass regime can be used to provide the self-compression of the bunch [2].

An evident disadvantage of the negative-mass undulator is the requirement of a strong axial magnetic field. An alternative method for the bunch compression can be the undulator super-radiation of a long-wavelength wave in additional (auxiliary) long-period undulator [3]. If the bunch is short enough, then the bunch front is placed close to the maximum of the decelerating phase of the radiated wave, whereas the tail is placed close to the “zero” wave field. This effect can be used for creation of a “bicolor” THz source based on the spontaneous emission from a short bunch, so that the super-radiation of the auxiliary long-wavelength wave is used to compress the bunch down to a size shorter than the wavelength of the operation short-wavelength wave.

The work is supported by IAP RAS Project 0035-2014-0012, and Russian Foundation for Basic Research Projects 16-02-00794 and 18-32-00351, and Israeli Ministry of Science, Technology and Space.

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## X-ray diffraction study of the structure of single crystals of Ni3Al alloy using synchrotron radiation after intense plastic deformation

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X-ray analysis has been applied for the study of the ultrafine-grained structure as a result of intense deformation in crystals Ni3Al alloy ordered by the L12 type using synchrotron radiation. In the initial state the material under study is in the single crystal ordered state and is oriented along the compression axis [211]. It should be noted that along with the ordered phase there is a small fraction of the disordered crystal oriented along the [100] axis. Compression of samples in Bridgman anvils and subsequent twisting at different angles has led to a change in their structural state. After compression between anvils and at reaching an average quasi-hydrostatic pressure of 8.0 GPa, the material changes in single-crystal structure to fragmented state. In addition to large crystalline fragments, submicrocrystalline disoriented regions appear. This work was supported by the Russian Foundation for Basic Research (project No. 16-03-00182-a). X-ray synchrotron measurements were carried out at the experimental station Diffractometry in the "Hard" X-ray Range at the Center of Collaborative Access of the Siberian Synchrotron and Terahertz Radiation Center, Budker Institute of Nuclear Physics, Siberian Branch of the Russian Academy of Sciences, Novosibirsk.

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## Apparatus-software tomography complex: the use of the regularization procedure in algebraic approach to image reconstruction

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The use of algebraic approaches with regularization terms in tomography can improve the quality of images with a low signal-to-noise ratio. Also they work well if the object under investigation contains highly absorbing inclusions or the measurement conditions are unstable during the measurement time. They win in competition of reconstruction techniques with a limited number of projections. Then why the main approaches of reconstruction in industrial and medical tomography set-ups are not algebraic ones with regularization? The talk provides an informative look at the problem. We formulate a list of tasks that need to be solved before the approach becomes widely used: high speed operation (working with data, optimality of mathematical methods and algorithms, and optimality of numerical realizations), choice of the method to solve the optimization problem depending on the optical properties of the object under probing and the choice of a regularization procedure depending on the morphological properties of the object under study. To solve the last problem in our efforts we use some list of the features

of the tomographic images. The paper presents an overview of mathematical tools used by different groups in the world to regularize the solution in the tomography problem. Results of the reconstruction for both model examples and real measurements are used to illustrate the main ideas. The measurements were carried out on the apparatus-software tomographic complex TOMAS developed and continuous operating at the Institute of Crystallography FSRC “Crystallography and Photonics” RAS. Also, approaches to solving the optimization problem (in the presence of highly absorbing inclusions) are analyzed. The results obtained by the quadratic programming method and the results obtained using the penalty method are compared. The average value and the variance value calculated for ROI, together with the width of the boundary profile form a list of the compared parameters.

This work was supported by the Federal Agency of Scientific Organizations (Agreement No 007-/3363/26) in data collection. This work was supported by Russian Foundation for Basic Research (project #17-29-03492) in the algorithmic part. The authors wish to express their appreciation to N. Vlasova for technical support.

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## Order-disorder phase transition and amorphous phase formation in superlocalized shear bands of Ni<sub>3</sub>Ge single crystals

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The loss of stability of the homogenous plastic deformation of single crystal specimens of the Ni<sub>3</sub>Ge alloy was investigated under uniaxial constant-rate compressive loading. A phenomenon of high temperature strain superlocalization was observed. Plastic deformation in this case occurred in a band a few tens of microns wide, reaching the values of hundreds of percent. Using synchrotron radiation it was shown that the phenomenon of strain superlocalization is associated with the formation of polycrystalline substructure in the strain localization band. Order-disorder phase transition and amorphous phase formation in superlocalized shear bands were revealed. This work was supported by the Russian Foundation for Basic Research (project No. 16-03-00182-a). X-ray synchrotron measurements were carried out at the experimental station Diffractometry in the “Hard” X-ray Range at the Center of Collaborative Access of the Siberian Synchrotron and Terahertz Radiation Center, Budker Institute of Nuclear Physics, Siberian Branch of the Russian Academy of Sciences, Novosibirsk.

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## Development of laboratory X-ray microtomography setups and tomography reconstruction algorithms

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Nowadays X-ray microtomography is convenient and widely used tool for scientists in laboratory and synchrotron sources. In this work, we show our progress in development of laboratory X-ray microtomography setups. Using X-ray optical elements (bubble lenses and asymmetrically cut crystals) allows us to reach spatial resolution up to 1  $\mu\text{m}$  at field of view about 1 mm. Without optical elements, spatial resolution is 10  $\mu\text{m}$  at field of view about 20 mm. The special role in tomography measurement is reconstruction algorithms. We use both traditional FBP algorithm and algebraic algorithms with regularizations. This allows obtaining good quality tomography reconstruction from X-ray images (projections) with low signal-to-noise ratio (SNR). In the paper we present latest developed tomographic setup that allows to take measurements in fully automatic mode with remote access of tomography data.

This work was partially supported by the Federal Agency of Scientific Organizations (Agreement No 007-/3363/26) in part of tomography setup construction and by the Russian Foundation for Basic Research (project #17-29-03492) in images corrections and tomography reconstruction algorithms.

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## X-ray diffraction imaging of three-dimensional location of linear crystalline defects

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Nowadays, the development of modern microelectronics is due to the permanent improvement of non-destructive X-ray diffraction methods for studying microelectronics devices and semiconductor crystalline materials, which are used for. Intrinsic crystal structure defects often cause the deterioration parameters and characteristics of microelectronics. Thereby, identification of the imperfections of the spatial crystal structures is one of the most important study issues, particularly, from viewpoint of the development of non-destructive X-ray methods of diagnostics and control of the real structure of crystalline objects.

In the present work, based on the X-ray topo-tomography method elaborated [1], experimental and theoretical studies are described in application to linear defects in silicon single crystals. The corresponding 2D topographic dislocation images are simulated and analyzed by use of the numerical solutions of the Takagi-Taupin equations that describe the two-wave X-ray diffraction by distorted crystals. The 3D-image quantitative reconstruction of the dislocation half-loop near the crystal surface is done with estimating the dislocation sizes.

This work was supported by the Federal Agency of Scientific Organizations (Agreement No 007-/3363/26) in part of diffraction tomography experiments and by the Russian Foundation for Basic Research (project #17-29-03492) in part of reconstruction algorithms.

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## Application of X-ray microtomography to the investigation of gallbladder stones

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Cholelithiasis is characterized by the appearance and growth of stones in the gallbladder. Nowadays gallbladder disease comes to the first place among pathology of a digestive tract. It tends to grow steadily and is to be relevant in gastroenterology. Despite the fact that there are many attempts to use computed tomography techniques for the practical diagnosis of gallbladder stones, the problem of diagnosis of cholesterol cholelithiasis from its other forms remains unresolved. In this work we have investigated the set of gallbladder stones. The experimental setup for microtomography with conventional X-ray source was developed in FSRC "Crystallography and Photonics" RAS, Moscow. The measurements were performed at the energy of X-radiation 17.5 keV. Our approach to analysis of experimental data as well as the reconstruction algorithm for X-ray tomography measurements was described in [1]. The X-ray microtomograms of gallbladder stones in the amount of 23 pieces were measured and 3D-distribution of the linear absorption coefficients was calculated by algebraic retrieval algorithm. A good agreement between values of linear absorption coefficients obtained from X-ray tomography experiments and theoretical calculations for cholesterol type gallbladder stones is demonstrated. The possibility of using the X-ray tomography method to diagnostic of gallbladder stones at the range of probe radiation from 50 to 100 keV is discussed.

This work was supported by the Federal Agency of Scientific Organizations (Agreement No 007-/3363/26) in part of tomography experiments.

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## The first observation of the free induction signals of OH radicals in the terahertz region

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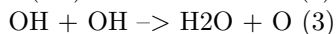
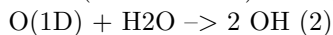
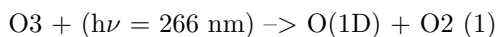
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Hydroxyl radical OH is the main oxidizing agent in combustion processes and in atmospheric chemistry. The rotational spectrum of OH is comparatively simple - there are less than 10 absorption lines in the terahertz region, corresponding to transitions between the lower rotational states of the radical. The FID signal was observed at the OH absorption line at 83.8 cm<sup>-1</sup>, that corresponds to the transition from the lowest rotational state of OH. Hydroxyl radicals were generated in the following sequence of chemical reactions



The concentration of OH radicals was  $\sim 10^{15} \text{ cm}^{-3}$ , lifetime  $\sim 300 \dots 700 \text{ ms}$ . The pulse of UV laser (266 nm) was synchronized with the FEL pulses. Shape of the FEL pulse after the optical cell was recorded by an ultrafast Schottky diode detector and 30GHz oscilloscope. Absorption line of the OH radical is a [U+F04C]- doublet with a splitting of 4.3 GHz therefore the FID signal contains characteristic beats with a period of 0.23 ns. During the lifetime of the OH radical, many pulses of FEL pass through the cell, each of which initiates a FID signal. We could register a sequence of 10 pulses simultaneously, obtaining separate frames of the birth and death of the radical. The influence of the magnetic field on the FID signal was studied by numerical simulation. A longitudinal magnetic field leads to a rotation of the polarization plane of the signal. The angle of rotation depends on the time after the FEL pulse. The effect of a nonmonotonic rotation of the polarization was predicted - after a certain time after the pulse, the direction of rotation changes.

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## Synchrotron radiation spectroscopy study of Mechanochemical Modification of Iron Particles by Polymers and Surfactants

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High-energy ball milling of metals in organic media makes it possible to modify the chemical composition of both the surface and the bulk of particles, including modification by chemically inert substances. As-modified particles may be potentially used for various composite materials, for example, microwave absorbing magnetodielectric composites. In this work, synchrotron radiation study of iron particles mechanochemically modified under their ball milling with various polymers (polystyrene, polydiene, paraffin) and surfactants (stearic acid and perfluorononanoic acid) have been carried out. XANES and EXAFS spectra measured at the iron K edge in the transmission mode were used to analyze the transformation of the local atomic environment of iron atoms in the bulk of particles (formation of a nanostructure, carbide formation) [1, 2]. The iron K edge XANES and EXAFS spectra, as well as carbon K edge, fluorine K edge, and Fe L<sub>2,3</sub> edge NEXAFS spectra measured in the total electron yield mode allowed us to analyze in detail the chemical transformation taking place in a thin surface layer of particles, including the processes of destruction, adsorption of organic molecules and formation of chemical crosslinking between different molecules in the surface layer [1-5]. For plate-like particles prepared in the presence of surfactants, polarization dependences of NEXAFS spectra were obtained, analysis of which made us to reveal the features of spatial arrangement of the molecules in the surface layer of the particles and its transformation during processing [1-4].

This study was supported by the Federal Agency for Scientific Organizations (No. AAAA-A17-117022250038-7), Scientific Program of UB RAS (No. 18-10-2-21) and bilateral Program "Russian-German Laboratory at BESSY II", HZB Berlin.

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## X-ray reflectometry, microtomography and small-angle scattering in the study of not completely ordered systems.

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Not fully ordered system (not crystals) are widespread in the surrounding nature, as well as in a number of new technologies. It seems natural to want to know what is inside, how different natural formations are arranged, or how accurately we created what we wanted. X-ray study can help us in this. As is known, X-ray structural analysis allows to reveal the internal structure of crystals up to the location of individual atoms. However, in the absence of complete orderliness, other phenomena related to the use of x – rays can be used, such as reflection, absorption and scattering. X – ray reflectometry is a method of investigation of layered systems with smooth section boundaries compared to the used radiation length. It is important to note that the analysis of the radiation scattering allows to quantify the roughness of the surface under study in a sufficiently wide range of spatial frequencies. It is shown that for a number of substrate materials as a result of their processing it is possible to achieve roughness at the level of one Angstrom. Such requirements are imposed today on substrates both for optical applications (for example, laser gyroscopes) and for microelectronics. Moreover, it is shown that this method allows to reveal and characterize the regular monoatomic steps created at some types of processing on the surfaces of single crystals. We show the possibilities of x-ray reflectometry for characterization of multilayer systems used in microelectronics, as well as for the analysis of layers on the surface of liquids, which sometimes can be considered as models of biological membranes. In all these cases, we are talking about layers with a thickness from fractions to several tens of nanometers. However, you often have to examine objects where there is no any order. Then one can apply X-ray microtomography. The report presents the principles of x-ray microtomography, including physical ideas used in the design of microtomographs and three-dimensional reconstruction of two-dimensional experimental data. A number of examples show the effectiveness of the use of x-ray microtomography in biological and medical research, as well as in industrial applications. We present possibilities of the method of topotomography to identify spatial locations of defects in crystals. It is shown that today in laboratory conditions it is possible to achieve micron resolution. For a number of applications, such resolution is not sufficient. This happens when we want to investigate the structure of protein molecules and their complexes, viral particles, the structure of polymers, etc.. The report presents some examples of such studies.

This work was supported by the Federal Agency of Scientific Organizations (Agreement No 007-/3363/26).

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## Influence of structural defects, functional groups and dopant impurities on the electronic structure of multiwalled carbon nanotubes

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The complex of X-ray photoelectron analysis methods (XPS and NEXAFS) were used to investigate the structure, composition, number and type of functional groups, the density of electron states near the Fermi level of multi-walled carbon nanotubes (MWCNTs), which were nitrogen doped and modified with ionic and thermal treatments. On the basis of experimental data on the density

of electronic states near the Fermi level, a band structure model for MWCNTs containing various types of defects was made. It is shown that doping of MWCNTs with nitrogen (N-MWCNTs) leads to an increase in the density of occupied states near the Fermi level, which indicates an increase in the number of free n-type charge carriers in the N-MWCNTs surface layer. It was found that annealing of N-MWCNTs in vacuum leads to a more significant overlap of the bands, which is due to annealing of structural defects and an increase in the amount of electrically active nitrogen. It was determined that irradiation of N-MWCNTs by a continuous beam of argon ions with an energy of 5 keV and a dose of  $\sim 10^{16}$  ion / cm<sup>2</sup> leads to the formation of a band gap ( $\sim 1.2$  eV) due to the destruction of the  $\pi$ -subsystem of sp<sup>2</sup>-hybridized carbon and strong oxidation of the surface of carbon nanotubes. An increase in the fluence of the ions to  $5 \times 10^{16}$  ion / cm<sup>2</sup> leads only to an increase in the number of oxygen-containing groups on the surface of carbon nanotubes without changing the width of the bandgap. The obtained results showed that the application of nitrogen doping, ion beam and thermal treatment allows directional influence on the electronic properties and chemical activity of the MWCNTs surface.

The work is supported by the RFBR (Russian Foundation for Basic Research, Russia), research project no. 18-32-00233 mol\_a.

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## Prospects of an X-ray FEL Oscillator driven by a SCRF linac or on a by-pass of a MBA Storage Ring

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An X-ray FEL Oscillator (XFEL), a low-gain device in which an X-ray pulse trapped in a X-ray cavity formed by Bragg crystals receives repeated FEL interaction will broaden the scientific vista for photon science of hard X-rays in areas requiring fully coherent and ultra-fine spectral bandwidth (BW). A 7-8 GeV super-conducting linear accelerator producing a constant stream of electron bunches is an ideal driver for an XFEL. With an optimized injector, it can produce fully coherent X-ray pulses with 3 meV BW and time averaged brightness 10<sup>28</sup> photons/s/(mm-mr)<sup>2</sup>/0.1% BW at 14.4 keV, which is higher by three orders of magnitude expected from LCLS-II-HE. By using a transverse gradient undulator (TGU), an XFEL is also feasible in a bypass of a large storage ring with multi-bend achromats, an "ultimate storage ring (USR) such as PETRA-4. Due to the beam cooling requirement, the ring-based XFEL will need to be operated in a pulsed mode with  $\sim 1\%$  duty factor. On the other hand, the X-ray BW could be an order of magnitude smaller than that based on a linac.

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## Headway on a Compact THz FEL at KAERI

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At the end of the 90s the first compact THz FEL at KAERI put into operation. Since a large number of experiments on this FEL were held. Also its specifications were improved during this time. A number of user stations were designed for this FEL. It operates now, but its lifetime is almost terminated and needs frequent repairs. In this connection, a development of a new machine seems reasonable. Its basic scheme was chosen similar to the old one. It consists of a microtron, an electron beamline, and a FEL structure.

A microtron is an RF resonance electron accelerator with a constant frequency and leading magnetic field, and a variable harmonics number. RF power is supplied by a magnetron in our project. A solid state switches modulator for it is based on storage capacities, and a high-voltage transformer. This project seems to be the cheapest alternative of a comparably high quality electron beam of the energy of several MeV.

The electron beam comes further through a beamline to the undulator. This beamline is intended to obtain the optimal beam parameters in the FEL structure at all expected entrance ones and the whole range of the undulator strengths. We selected a hybrid electromagnetic scheme for the undulator. It contains magnetically soft poles and both permanent magnets and conductive busbars. The magnetic field is controlled by current in the busbars. The main advantage of this design over the conventional hybrid one (without coils) is the absence of moving parts.

The peak electron beam current in our case is comparably small and the FEL emission wavelength is long, so it is a problem to get lasing in an open resonator. So a waveguide scheme for our FEL was selected to reduce the mode size. The main problem in this case is wave absorption in a waveguide. A lenticular shape and dielectric coating are used to solve it.

Injection of an electron beam into and extraction from a waveguide, and emission outcoupling in this case are interrelated and sophisticated problems. We consider combining a blind mirror of the FEL waveguide cavity and a beam dump, so the extraction is not necessary. A mesh mirror is used for injection and outcoupling. Another tilted mirror with a hole separates an electron beam and THz emission.

The expected basic parameters of the FEL are the following:

- Wavelength tuning range 0.4 . . . 0.6 mm;
- Spectral width 1%;
- Peak power 5 kW;
- Macropulse average power 600 W;
- Macropulse duration 5  $\mu$ s;
- Repetition rate up to 100 Hz.

Currently, the scheme and the composition of equipment for the FEL were determined. Each part has been simulated and optimized. Thus, both the conceptual and technical designs are ready. The microtron has been commissioned, and is being optimized now. The undulator has been manufactured and is being commissioned. All the magnets for the beamline has been manufactured and tested. The following parts are to be mechanically designed and manufactured: the optical resonator, a vacuum chamber for the beamline, and supports. After that the whole machine can be assembled and commissioned.

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## Structure of composites based on multi-walled carbon nanotubes and metal oxides formed by magnetron sputtering.

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Using the XPS and NEXAFS methods, as well as SEM and TEM, the structure of composites based on multilayer carbon nanotubes (MWNT) and tin oxides (SnOx / MWNT) and titanium (TiOx / MWNT) obtained using magnetron sputtering was studied. The obtained data showed that the formation of composites by this method provides high adhesion of metal oxides to the MWNT surface due to the formation of chemical bonds (Sn-O-C, Ti-O-C) at the interfaces of the composites. The formation of heteroatomic chemical bonds is provided by the formation of structural defects and functional oxygen-containing groups in MWNT walls when metal oxides precipitate on their surfaces. It is shown that metal oxides in composites are represented by a mixture of oxides of variable composition with oxygen deficiency and defective crystal structure. The presence of dangling bonds and structural defects provides a high chemical activity of metal oxides and their fixation on the functional groups present on the outer walls of the MWNT. The results of the analysis showed that the use of thermal treatment of composites in air in the temperature range of 500-700 ° C leads to the crystallization of metal oxide films on the MWNT surface. The authors thanks Roslikov V.E. and Kan V.E. for synthesis of the composites.

The work was supported by a grant from the Russian Foundation for Basic Research project N<sup>o</sup> 18-32-00233 mol\_a

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## Two types of coherency, fine mode structure, and obtaining of the ultramonochromatic tunable terahertz radiation on the NovoFEL

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Laser nature of a continuous pulse-periodical radiation of the Novosibirsk terahertz free-electron laser appears in a good coherency of its pulses and very narrow synchronized longitudinal modes. Filtration of one of the modes by a system of three resonance Fabry-Perot interferometers allows to create laser source with monochromaticity which is sufficient for typical high-resolution THz spectroscopy ( $\delta f/f \leq 1E-7$ ,  $\delta f \leq 0.2$  MHz). Features of the source compared to other alternative devices are a wide tuning range (1.5-3 THz) and much more high output power (up to 100 mW).

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## Terahertz high-harmonic gyrotrons with irregular microwave systems

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 The conventional low-harmonic gyrotrons operating in the terahertz frequency range require very strong magnetic fields. The fields can be decreased by using high-cyclotron harmonic operation of the gyrotron performed in the configuration of the so-called large-orbit gyrotron (LOG) with an axis-encircling electron beam. This configuration improves both the electron-wave coupling and the mode selectivity significantly. In the experiment [1], a 80 keV/0.7 A LOG was realized. Stable single-mode third-harmonic operation was observed at frequencies up to 1 THz with an output efficiency of ~1%. Recent experiments on this installation are aimed to improving this system by using irregular cavities with decreased diffraction Q-factors [2]. The next our step in LOG development is aimed to creation of a 30 keV/0.7A CW gyrotron. The experimental setup is based on the use of a 5 T cryomagnet and a cusp gun forming axis-encircling electron beam with a pitch-factor of 1.5. The main scope is to provide gyrotron operation at the second-third-fourth cyclotron harmonics at the frequencies 0.26 THz, 0.39 THz, and 0.52 THz, respectively, with the output power level of hundreds of Watts for DNP/NMR applications. An increase in operating magnetic field up to 6.3 T together with an increase in the voltage up to 45 keV should allow achieving frequencies up to 0.65 THz at the fourth cyclotron harmonic.  
 In order to decrease Ohmic losses in the 80 keV/0.7 A LOG, we tested a sectioned system with a klystron-like electron-wave interaction [3]. In the sectioned-cavity experiment, the operating current (0.3-0.5 A) was slightly lower as compared to earlier experiments. Selective excitation at the third harmonic was achieved at a magnetic field close to 10.2 T. The output rf signal at a frequency of 0.74 THz corresponded to the transverse mode TE<sub>3,5</sub>. The detected output rf power was 100–250 W. According to simulations [4], the share of the ohmic losses in this experiment

was relatively low (20%–25% of the rf wave power emitted from the electron beam) as compared to the first experiment [1], where the losses were as high as 85%.

The 30 keV/0.7 A gyrotron based on a 5 T cryomagnet and an axis-encircling electron beam with a pitch-factor of 1.5 was tested in first pulsed experiments. The same regular cavity was used to provide excitation of the mode TE 2,5 at the second cyclotron harmonic (0.267 THz) and of the mode TE 3,7 at the third harmonic (0.394 THz) at slightly different magnetic fields. In order to increase the efficiency of the third-harmonic gyrotron, as well as to provide operation of the fourth-harmonic gyrotron at a frequency of 0.52 THz with the power level of  $\sim 100$  W, special cavities with a decreased diffraction Q-factors are required. It was proposed [5,6] to use a cavity consisting of several regular sections, which are separated by short non-regularities providing the [U+F070]-shift of the wave phase between the sections. Such a configuration ensures the “gyrotron-like regime” of the electron-wave interaction for a far-from-cutoff mode possessing a relatively low diffraction Q-factor. This approach is used to design operating cavities for pulsed gyrotron (80 keV, 1.0-1.3 THz) operating at third and fourth cyclotron harmonics, as well as for the CW gyrotron (30 keV, 0.38-0.65 THz) operating at the second and third harmonics. Several methods for improving selectivity of excitation of high harmonics based on the use of cavities with short irregularities are proposed and tested in experiments.

The work is supported by the Russian Science Foundation, project # 17-19-01605.

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## Resonant photoemission spectroscopy of materials for photovoltaics

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The electronic structure of CuIn<sub>1-x</sub>Ga<sub>x</sub>Se<sub>2</sub> single crystals has been determined by the method of resonant photoemission, and the main regularities of its transformation have been established with a change in the concentration x from 0 to 1. The dependence of the shape of the spectra of valence bands on photon energy has been studied. It is shown that the integrated intensities of photoemission are determined by atomic photoionization cross sections. The processes of direct and two-step generation of photoelectrons accompanying photoemission and the participation of internal states in electron spectra from valence bands were studied. In the case of threshold excitation of the Cu 2p level, two-hole final states in photoemission were obtained. The strong interaction of the holes leads to a multiplet splitting of these states. Using the energy dependence of the atomic photoionization cross sections, the partial densities of the states of the components are determined.

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## Dynamical observation of X-ray Laue diffraction on single-crystal tungsten during pulsed heat load



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The normal operation of fusion reactor based on tokamak involves periodical transient heat loads to divertor plates. The currently the divertor plates of ITER is supposed to be covered by tungsten. The tungsten tends to cracking as a result of the pulsed heating. The reasons of the crack formation are deformation and mechanical stresses caused by the raise of temperature of thin surface layer. The diagnostic of the dynamic of the deformations and stresses in tungsten under pulsed heat load is under developments at the VEPP-4M beamline 8 (scattering station "Plasma"). The observation of the material deformation is based on the change in the X-ray diffraction scattering angle as a result of the non-uniform material extension. Previous experiments demonstrated measurements of residual stress and measurements of the depth-average deformation dynamics. Currently the scattering station "Plasma" was upgraded. Namely, a the energy of the Nd:YAG laser pulsed was increased from 1J to 50J. The pulse duration remains  $\sim 140\mu\text{s}$ . The number of frames of the one-dimensional gas X-ray detector DIMEX was increased from 30 to 100. And the intensity of the synchrotron radiation with energy 69keV (K-edge of tungsten) increased with a factor about 50 due to the new wiggler. Also a fast pyrometer was developed to measure the temporal dependence of the temperature of the heated surface. The combination of the improvements resulted in the measurements of the diffraction peak shape dynamics during the heating of the  $500\mu\text{m}$  thick single crystal tungsten. The change in scattering angle was measured to be about  $2^\circ$ . The three stages of the diffraction peak evolution can be clearly distinguished here: the heating of the surface, the equalizing of the heat distribution transversely to surface and cooling to room temperature. During the first stage approximately from  $0\mu\text{s}$  and to  $140\mu\text{s}$  (the heating of the surface) the surface temperature increases. The scattering angle of X-ray grows coupled with the temperature. Consequently the large angle side of the diffraction peak moves to the large angles. The equalizing of the temperature distribution transversely to the surface occurs at the second stage (approximately from  $140\mu\text{s}$  and to  $240\mu\text{s}$ . The equalizing of the temperature means that the cooler layers warm up and hotter layers cool down. Consequently the change in scattering angle is positive at the lesser angles and is negative at the larger angles. As a result the diffraction peak becomes narrower. The long term evolution during the third stage (cooling to the room temperature) was not measured. However, the final state of the diffraction peak shape was measured after several seconds. Note that the initial and final stats are static. So the diffractograms were measured significantly more accurately. According to the relation between temperature and scattering angle the latter decreases after cooling. The difference between initial and final positions of diffraction peaks means that the plastic deformation was happened.

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## Numerical simulation of diffraction of synchrotron radiation in a single-crystal tungsten.

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The influence of pulsed heat load on the tungsten wall of the divertor in a fusion reactor is expected to cause cracks, which will lead to the appearance of plastic deformations. In order to understand how these deformations are formed, and how to avoid it, one has to measure their dynamics during a pulsed heat load. Currently, the diagnostic of the dynamic of such deformations based on the method of fast diffractometry is under development at the BINP SB RAS. The method of fast diffractometry consists in measuring the diffraction pattern of a synchrotron radiation beam (SR) scattered on a sample of a single-crystal, and then in reconstruction of the deformation of the sample according to the form of a diffractograms. The work was carried out in the Laue back reflection method; a mosaic single-crystal tungsten was used as a sample. The model takes into account the absorption of SR, mosaicism of sample, and the kinematic theory of diffraction was used. Firstly, an analytical calculation of SR beam propagation in the sample was carried out, as a result of it an integral expression for the intensity distribution of the scattered radiation was obtained. This expression determined the geometry of scattered SR. The next step was to take into account the mosaicism of a single-crystal tungsten by introducing a model distribution function which describes the orientation of crystal planes in a single-crystal. After that, the convolution of these two expressions was carried out by using numerical methods. Pulsed heat load leads to compression and stretching of the surface that as a result is expressed in a rotation of the “reflecting” crystalline plane. The same effect can be achieved by sample rotation. That’s why final expression is determined by the angle of the sample position relatively to the SR beam and the distribution function parameters. Wolfram Mathematica program was created to simulate the scattering of the SR beam in a mosaic single-crystal tungsten to calculate the instrument functions of the problem of the deformation reconstruction in a sample according to the form of diffractograms. Numerical calculations and comparison with experimental data were carried out, and as a result, revealed a discrepancy in the shape of the interference peak. This discrepancy was reduced by using a more appropriate distribution function.

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## Feedback suppression of fast vertical oscillations of the VEPP-3 SR beam

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Experiments have been performed to suppress vertical oscillations of the synchrotron radiation (SR) beam of the VEPP-3 storage ring by introducing a fast feedback. These oscillations are caused both by current pulsations in the magnetic elements and by mechanical vibrations, and can not be suppressed by the existing beam stabilization system.

In experiments, the beam position sensor which was a previously developed differential detector included pin-photodiodes, an electronic section (providing filtering and double correlated sampling of signals), and an electromechanical system (automatically adjusting sensitive element to the center of SR beam).

The signal corresponding to the beam displacement from regulation position was transmitted (after processing and amplification) on a fast corrector – a pair of saddle coils imposed directly to the metal vacuum chamber of storage ring, which formed a negative feedback. A ratio calculator (for independence of feedback strength from the beam intensity) and PID-regulator (for correction of frequency characteristic) could be included in the signal processing channel.

Both the upper operating frequency and the maximal stable feedback were determined by the speed of the magnetic corrector (by delay in the penetration of the field through the metal wall of the chamber). Nevertheless, when the system was turned on the amplitude of oscillations with the frequency of power network (50 Hz) was reduced by 30 dB, and the amplitude of the powerful disturbance at frequency of 690 Hz was reduced by 25 dB.

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## Spin-Resolved Photoemission for the investigation of Rashba spin-splitting and topological surface states

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The development of high-resolution angular resolved photoelectron spectroscopy (ARPES) and recent discovery of new quantum matter such as topological materials and Rashba systems with their peculiar spin-texture of the surface states stimulate to develop high-performance Spin-resolved ARPES (SARPES). Recent high-resolution radiation synchrotron sources and the newly developed high performance spin polarimeters open a new era of surface science using SARPES measurement. This allows solving one of the important problems of solid-state physics: the description of the electron freedom degree in a solid through the connection of the energy  $E^*$  with momentum  $k^*$  in a crystal and the band structure of quasiparticles. An important role belongs to the electron spin which determines such properties as ferromagnetism, spin-polarized surface and interface states, as well as unusual properties in topological insulators, in which a rigid link between the spin projection and the direction of the momentum is discovered.

In the first part the problems of detecting the electron spin polarization using modern spin detectors with spatial resolution in the photoemission method with an angular resolution will be considered. The second part will be devoted to the study of spin-polarized Dirac states in three-dimensional topological insulators, including crystal topological insulators, in graphene-like bismuth monolayer, as well as the condition for the formation of a combined Dirac and Rashba electronic structure of the spin-polarized surface states.

This work is partially supported by the Ministry of Education and Science of the Russian Federation and by Siberian Branch of the Russian Academy of Sciences (Project II.8.70).

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## Application of XANES and XPS in using synchrotron radiation in soft X-ray region for the study of the electronic structure of graphite and few-layered graphene fluorides

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The compounds of fluorinated few-layered graphene are of great research interest as promising materials in nanoelectronics, optoelectronics, sensorics, and catalysis. The study of the electronic structure makes it possible to reveal the relationship between the structural features and the physicochemical properties of the compounds. X-ray absorption spectroscopy (XAS) and X-ray photoelectron spectroscopy (XPS) using synchrotron radiation in the soft X-ray range are among the most effective for studying the electronic structure of such nanomaterials. The work presents the results of combined synchrotron based XANES and XPS investigation of fluorinated graphene layers of different composition. Experimental CK, FK, NK XANES and XPS were obtained on the Russian-German beamline RGLB at synchrotron source BESSY II, and for dielectric samples the XPS results were supplemented by measurements on a laboratory spectrometer. The parameters of the electronic structure are studied when the composition of the fluorinated graphene layer varies from C<sub>2</sub>F to CF. Relationship between the local atomic and electronic structures are revealed depending on the size effect and composition. It is shown that with a decrease in the concentration of fluorine atoms, the energy gap decreases. Examples are given of the influence of the size effect on the composition and electronic structure of aminofluoride of graphite and few-layered graphene upon interaction of graphene fluorides with ammonia. It turns out that as a result of the interaction, fluorine atoms are removed from the surface (the reduction reaction) and the amine group is attached to the carbon layer. It can be noted that

the concentration of fluorine in the aminofluorides increases with decreasing number of layers. It is presented the examples of the application of XANES and XPS in studying the mechanism of surface layer reduction in graphite fluorides of various compositions under the action of electron irradiation, changes of composition in depth, and changes in the electronic structure when the fluorine atoms are removed. The study of the interaction between the graphite fluoride matrix and various guest molecules in intercalation compounds of graphite fluoride such as bromine, ferrocene are presented. The results of this research are important for studying the mechanism of reduction of graphite fluorides and developing methods for the synthesis of graphene based compounds.

This work is financially supported by RFBR research project N 16-03-00048

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## Opportunities of dielectric laser accelerator on high-brightness radiation and industry application

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A dielectric laser accelerator (DLA), operating at the optical frequencies with a pulse rate close to 1 GHz, is expected to generate high-energy nano-electron bunches with a moderate average current. The nano-electron bunch permits highly efficient electron superadiance in the EUV and x-ray spectrum. In this talk, I will compare the peak and average brilliance of envisioned DLA-driven radiation sources against the 3rd- and 4th-generation light sources. Since a DLA operates with a much less average beam power, superior performance of a DLA-driven radiation source stands out when the brilliance under comparison is normalized to beam power. I will also present the opportunities of a DLA beam and radiation source in the applications of radiation therapy and EUV lithography with specifications from the industry.

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## In situ approach to investigations of CuFeAl-composite catalysts in catalytic oxidation of CO: X-ray adsorbstion spectroscopy

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**Keywords:** In situ XAS, operando XRD, CuFeAl-composite catalysts, CO oxidation, catalytic combustion.

Catalytic oxidation of gasification products of solid fuels is allows utilizing low-grade fuels such as lignite, peat, and firewood as well as various industrial wastes. At the same time catalytic combustion produces a significantly lower amount of harmful emissions then "traditional combustion" of fuels. CuFeAl-composite catalysts demonstrate high activity and stability in catalytic oxidation of gasification products of solid fuels. Moreover, the catalysts are inexpensive and ecologically clean. In this contribution we present our first results of in situ investigations of the catalyst state in reaction conditions. Since carbon monoxide is the main product of gasification of solid fuels we performed investigation CuO, Fe<sub>2</sub>O<sub>3</sub>, and CuFeAl-composite catalysts in CO and CO+O<sub>2</sub> mixture in a wide temperature range. We applied three methods: XANES, EXAFS, and additionally

operando XRD. XANES is very useful for identification of different chemical states of copper and iron and allows us to study the chemistry of the catalysts under reaction conditions. X-ray diffraction techniques allow us to study the phase composition, but, unfortunately, the technique cannot identify nanoparticles and amorphous phases. This shortcoming can be eliminated by EXAFS which may clarify the structure of local environment of copper and iron atoms even when their concentration is extremely low. In situ XAS experiments were performed at the Structural Materials Science station at Kurchatov Center for Synchrotron Radiation. The spectrometer is equipped with high temperature chamber that allows collecting XAS spectra within temperature range from RT to 600°C in the gas mixture at atmospheric pressure [1]. Operando XRD/MS experiments were carried out at the “High Precision Diffractometry II” station at “Siberian Synchrotron and Terahertz Radiation Center” and at lab Bruker D8 Advance diffractometer (Boreskov Institute of Catalysis). The both diffractometers are equipped with XRK 900 reaction chambers (Anton Paar GmbH) that allow observing the diffraction patterns within temperature range from RT to 900°C in the reactant mixture at atmospheric pressure [2]. We found that fresh CuFeAl-composite catalysts consist of CuO, Fe<sub>2</sub>O<sub>3</sub>, and Al<sub>2</sub>O<sub>3</sub>. In a CO flow, the reduction of copper from Cu<sup>2+</sup> to Cu<sup>1+</sup> and Cu<sup>0</sup> started at temperature about 200°C; at 600°C copper is mainly in the metallic state. At the same time the reduction of iron started at temperature about 400°C and at 600°C about 20% of iron is in the metallic state. Operando XRD study allows us to determine the phase transition of iron-containing phase during the reduction in a CO flow. The reduction process occurs in the next manner: Fe<sub>2</sub>O<sub>3</sub> → Fe<sub>3</sub>O<sub>4</sub> → FeO and Fe<sup>0</sup>. In CO:O<sub>2</sub> = 2:1 mixture, the reduction of copper from Cu<sup>2+</sup> to Cu<sup>1+</sup> started at temperature about 400°C and at 600°C about 50% of copper is in the Cu<sup>1+</sup> state, whereas iron is slightly reduced to Fe<sup>2+</sup> state at 600°C. The following increase the partial pressure of O<sub>2</sub> leads to shift initial reduction temperature to high temperature range. Thus, the use of complimentary methods (XANES, EXAFS, and XRD) allows us to determine the chemical state of copper and iron, phase composition in the catalyst during the oxidation of CO. The data presented can facilitate to clarify the mechanism of oxidation of CO.

Acknowledgement. This work was supported by the Russian Science Foundation, grant 17-73-20157.

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## Element composition of species of the genus *Dasiphora* (Rosaceae) of the Russian Far East and East Siberia

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This study focuses on determination of element composition of plants belonging to different species of the genus *Dasiphora* (the Rosaceae family) occurring in East Siberia and the Russian Far East and on level of species with high content of macro- and microelements. The plants of five species (*D. fruticosa*, *D. parvifolia*, *D. mandshurica*, *D. gorovoi*, *D. davurica*, and the variety *D. davurica* var. *flava*) were collected in 2013-2014 in the Russian Far East (Primorsky Krai) and in East Siberia (Buryatia) along with the samples of soil at the sites of the plant growth. Element composition was analyzed by the method of X-ray fluorescence analysis using synchrotron

radiation (SRXRF) in the element analysis station of the Shared-Use Center SSTRC Budker Institute of Nuclear Physics, SB RAS (VEPP-3 storage ring). The content of 21 elements (K, Ca, Ti, V, Cr, Mn, Fe, Co, Ni, Cu, Zn, As, Se, Br, Rb, Sr, Y, Zr, Nb, Mo, Pb) were determined in aboveground organs (leaves and stems). The maximum total content of macroelements (K, Ca) was found in the aboveground organs of two taxa – *D. davurica* and *D. davurica* var. *flava* (232-331 mg/g), and the minimum in the leaves of *D. mandshurica* (14 mg/g) and in the stems of *D. fruticosa* (15 mg/g). The highest total content of microelements, from 1937 to 2435 mg/kg, was found in the aboveground organs of *D. gorovoi*, *D. mandshurica*, and *D. fruticosa*, and a minimum in the leaves and stem of *D. davurica* var. *flava*, 215 and 233 mg/kg respectively. We found that each species had a certain concentration of elements. The highest content of Ti, V, Co, Ni and Nb was found in the stems of *D. fruticosa*, Zr in leaves of *D. parvifolia*. The aboveground organs of *D. mandshurica*, accumulate a high level of Br, and have an increased content of Fe, Mn, Cu, As, Sr and Pb in the stems. The leaves and stems of *D. gorovoi* plants accumulate, predominantly Mn, Cr, Rb, Y and have particularly high content of Zn, the concentration of which 5.5 times exceeded the average concentrations of this element in plants (Kabata-Pendias, Pendias, 1989). The highest content of Mo was found in the stems of *D. davurica* var. *flava*. The leaves and stems of *D. davurica* and the stems of *D. fruticosa* include the traces of Se that contrast the other species, where Se is below the detection limit or completely absent. The high content of Ti, V, Mn, Co, Ni, Cu, Zn, As, Rb, Y, Zr in plants correlates with content of these elements in the soils of the sampling sites. We found that the high content of a number of individual elements examined, depending on the organ of a plant, characterizes three representatives of the genus *Dasiphora* – *D. fruticosa*, *D. mandshurica* and *D. gorovoi*. Additionally, these species demonstrate the excessive levels of the potentially toxic chemical elements: Ni and Pb. Kabata-Pendias A., Pendias H. Trace Elements in Soils and Plants; Mir: Moscow, Russia, 1989. 439 p.

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## Measurement of the dynamics of residual stresses in copper during heating

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In a fusion reactor plasma impacts the divertor as periodic heat pulses. These heat pulses cause residual plastic deformations and mechanical stresses in the divertor which leads to the divertor's material being destroyed. Residual deformations and stresses can be relieved due to high temperature of the divertor which is caused by the constant flow of plasma. The relaxation of deformations and stresses may bring the material back to its initial state during the time interval between two subsequent heat pulses, so that after the second plasma pulse stresses would not exceed ultimate tensile strength and the material would not be destroyed. The temporal dependence of residual stresses is needed to be examined in order to test the possibility. The relaxation of stresses in copper samples were measured using X-ray diffractometry in development of the technique. In order to create residual stresses one sample was irradiated by an electron beam on the BETA facility while another one was left in its initial state for the comparison. Experimental station "Anomalous scattering" on the beam line 2 of VEPP-3 was used to obtain diffractograms that were used to obtain scattering angle – tilt angle dependencies. By expressing the variation of crystal lattice interplanar distance and using it in Bragg's law, a formula of scattering angle – tilt angle dependency was derived. The dependencies were substituted in the formula and used for calculation of deformation tensor components, which in their turn were used for calculation of stress tensor components. The difference between the diffraction maximum angles of the irradiated and the non-irradiated samples was a significant  $\sim 0.1^\circ$ . For the dynamic measurement of the residual stresses samples were examined at constant tilt angle and changing temperature at scattering station "Diffraction movie". The samples were heated for the relaxation of stresses. Time dependencies of temperature and diffraction maximums were measured. By

combining those time dependencies the diffraction maximum angle – temperature dependencies were acquired. The relaxation of the samples in the condition of changing temperature was studied by comparing of the irradiated sample's graph to the non-irradiated sample's graph with the latter having no relaxation effect.

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## X-ray beam-shaping refractive optics and its applications

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The most advanced X-ray sources, such as third-generation synchrotrons and free electron lasers (XFEL), are capable to generate high brightness coherent radiation, especially in the hard X-ray region. The availability of such beams facilitates to the development of a new generation of X-ray optics, which goes far beyond simple focusing optical elements. This optics possesses new optical functions, which allow forming the intensity of the wave front with almost complete freedom. Such beam-shaping elements use the most outstanding properties of synchrotron radiation such as brightness, monochromaticity and coherence. As for the possible employment of X-ray beam-shaping devices based on refractive optics, is not limited to beam conditioning applications only. It can be extended to the field of interferometry, coherent diffraction, phase-contrast microscopy, ultrafast and nonlinear optics studies.

For example, one of the most striking demonstrations of the beam-shaping optics is a special class of refractive optical elements having axial symmetry that are capable to convert a point-like source to a narrow axial straight line segment. These optical elements are called axicones. Recently, we demonstrated an X-ray parabolic refractive axicon lens as a novel type of X-ray beam-shaping element [1]. Under coherent X-ray illumination, the parabolic axicon generates Bessel-like beam propagated along the optical axis and ring-shaped beam in the far field. Such optical transformations can be used in areas requiring special illumination, as well as extended focused beams, for instance, in diffraction and imaging techniques, in metrological, source diagnosis and beamline alignment applications. Moreover, such beam-shaping capabilities can significantly simplify some existing experimental layouts or lead to completely new optical schemes for X-ray techniques based on synchrotron and XFEL sources.

The work was supported by the Ministry of Education and Science of the Russian Federation grant contract № 14.Y26.31.0002.

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## Quick-XAFS as a tool for study of chemical processes

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Investigations of chemical processes in Chemistry have gain new opportunities thanks to Synchrotron Radiation Facilities allowing researchers to control and manage synthesis of inorganic

compounds and new materials as well understand chemical reactions in details. One of the techniques for preparing nanoparticles (mono-, bi-, n-metallic) is a thermal decomposition of inorganic compounds such as molecular, complex compounds in the presence of a support material and without it under different atmospheres.

The work presents results of in-situ Quick-XAFS and XRD study of thermal decomposition of the double complex salt  $[\text{Pd}(\text{NH}_3)_4][\text{OsCl}_6]$  in an inert atmosphere. The study purposes two objects. The first one is to find out all intermediate products, some of them has sometimes a short lifetime and could not be synthesized or isolated as the product from the thermal chemical process. Meanwhile the intermediate has its ability to effect on a final product. The second object is to observe a formation of bimetallic nanoparticles or nanoalloys (the final product) in real time. The compounds containing two metals were shown to be used for preparing bimetallic alloys with composition different from that a binary phase diagram predicts for a bulk alloys.

Quick-XAFS and XRD experiments were performed at Rock beamline of Soleil Synchrotron (Paris, France) and ID11 beamline of ESRF (Grenoble, France) respectively. All the thermal decomposition intermediates were found. One of the results of the study is a demonstration of distinguish in the thermal decomposition of  $(\text{NH}_4)_2\text{OsCl}_6$  as the intermediate product in the complex compound compared to a pure ammonium hexachloroosmate(IV) under the same condition. The formation of the PdOs bimetallic nanoalloy includes the following steps. At first, the Pd metallic particles are appeared. Then the bimetallic particles are formed. Finally, a partial segregation of the PdOs nanoalloy happens. Quick-XAFS and common XAFS experiments with  $[\text{Pd}(\text{NH}_3)_4][\text{OsCl}_6]$  are compared demonstrating noticeable limitation of the common XAFS. In particular, it concerns to beginning process of formation of the small metallic particles.

This work is financially supported by RFBR research project N 17-03-00693 A/

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## XAFS, XPS, and Mossbauer spectroscopy studies of valleriite, nanocomposite mineral and a prototype for new low-dimensional chalcogenide materials

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Valleriite is a mineral composed of alternating layers of brucite-like phase  $\gamma\text{Mg}(\text{OH})_2 \bullet z\text{Al}(\text{OH})_3$  and sulfide close to chalcopyrite  $\text{CuFeS}_2$ . Generally, valleriite is not very abundant, but “coppery” ores of Noril’sk ore provenance contain up to 20% of this mineral, industrial values of copper, nickel and are enriched in platinum group metals. Flotation of the coppery ores is poor, and even small admixtures of valleriite to of other ores suppress the recovery of values. Valleriite is also of interest as a material with very special character in terms of the structure of “noncommensurate” sulfide and hydroxide layers, and their electronic and magnetic properties, including probable persistent electric charge of the layers. Here, we report Cu K-, Fe K-edge XANES and EXAFS measured at BM23 beamline, ESRF, in combination with XPS,  $^{57}\text{Fe}$  Mössbauer spectroscopy and other techniques in order to get insights, first of all, into specific characteristics of the sulfide layers of two valleriite samples of Noril’sk ores. The sample 1 was associated with pyrrhotites  $\text{Fe}_{1-x}\text{S}$ , and the sample 2 associated with chalcopyrite  $\text{CuFeS}_2$  was a product of its incomplete interaction with serpentine minerals. It was found, in particular, that the sulfide layers in both valleriites show the photoelectron and Cu K-edge XANES spectra slightly distinct from those of chalcopyrite. While the Cu+-S bonding in the first coordination sphere was similar, the differences in the Cu-Fe and Cu-Cu coordination were found for the two valleriites and chalcopyrite, interpreted mainly in terms of various concentrations and the state of iron. The Mössbauer spectra suggest that, in



contrast to chalcopyrite with ordered positions of Cu<sup>+</sup> and Fe<sup>3+</sup> ions and the antiferromagnetic character, the sample 2 contains about 25% of Fe<sup>2+</sup>-S species responsible for the negative charge of the sulfide layers; the sample 1 appears to have more than 90% of Fe<sup>3+</sup>. The sulfide layers exhibit rather high conductivity and are in electron equilibrium with hydroxide entities, at least under the XPS experiment conditions. The negative charge of the sulfide “phase” affects the chemical properties of the mineral, in particular, promoting the reduction and deposition of platinum in elemental form.

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## Compact THz radiation source for DNP-enhanced NMR

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Nuclear magnetic resonance (NMR), which is along with X-ray crystallography one of the basic methods for determination of the protein molecular structure, greatly suffers from its low sensitivity. Therefore, the development of the instrumentation and technique for the NMR sensitivity enhancement methods such as dynamic nuclear polarization (DNP) is a topical problem. For current high-field NMR spectrometers, DNP technique exploits and requires radiation in the terahertz frequency range, namely at the frequencies of 260-650 GHz with power level of 1-100 W. This power level is far beyond capacity of solid-state devices or conventional slow-wave vacuum electron tubes and could be provided nowadays only by expensive and complicated devices such as gyrotrons and free-electron lasers, which hinders the widespread of the DNP technique. We develop an alternative and cheaper approach, which is a combining of an NMR spectrometer and a very compact low-voltage gyrotron (“gyrotrino”) in a single cryomagnet [1- 3]. This eliminates the need for an additional superconducting magnet, results in a shorter terahertz transmission line, and can make DNP systems more available for research laboratories.

The integration of the gyrotron with NMR-spectrometer in a single cryomagnet causes a number of specific features, which result from two requirements to be met for integrated version of THz oscillator, first, the matching of oscillator and DNP frequencies, and second, a very restricted space in the spectrometer cryomagnet bore. We show that the frequency matching condition can be fulfilled in the case of the gyrotron with a very low operating voltage of 1.5-2 kV. In turn, such low voltage complicates the design of the electron-optical system since it results in a very small anode-cathode distance, a low electrical field at the emitter, and strong influence of the initial velocity spread caused by emitter surface roughness. Despite these difficulties, a two-electrode electron gun was designed, which can form a laminar electron beam with the required parameters. To mitigate high-sensitivity of the electron gun to the thermal displacements of the electrodes, the mechanism of anode-cathode distance adjustment is also designed. Due to lack of free room in the spectroscopy bore, the gyrotron collector cannot be placed in a stray magnetic field, so the electron beam deposition occurs in a uniform magnetic field, which results in a power density of 10 kW/cm at the inner collector wall. Additionally, the mode converter of the designed compact gyrotron is very unconventional since we have organized the power output from the cathode end of the gyrotron cavity and placed all the converter mirrors at the one side of the electron beam. The feasibility of the gyrotron generation under very low operating voltage was examined experimentally using an existing CW gyrotron initially designed for operation at the second cyclotron harmonic with a relatively high voltage. To form a low-voltage helical electron beam with a sufficiently large pitch-factor, a positive voltage was applied to the first anode of the

gyrotron three-electrode magnetron-injection gun with a negative voltage at the cathode. CW gyrotron operation at voltages down to 1.5 kV has been demonstrated at a frequency of 252 GHz. This work was supported by the Russian Science Foundation under project No. 16-12-10445.

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## Morphological changes in the mammals skeletal muscle caused by powerful terahertz laser irradiation

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Morphological changes in M.Wistar skeletal musculature, caused by focused radiation of the first stage of the Novosibirsk FEL, are considered. Optical and electron microscopy investigations of the prepared samples were performed. The aim of the study was determination of the specific changes to the tissue in comparison with CO<sub>2</sub> laser irradiation. Detailed morphological description and possible causes of damage are considered.

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## Morphological changes of the human healthy blood erythrocytes caused by terahertz laser radiation

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The purpose of this study was to assess the effect of terahertz radiation on the erythrocytes of healthy volunteers. Exposure of the prepared samples was carried out by non-focused radiation of the Novosibirsk FEL at wavelengths of 130-200  $\mu\text{m}$  with an average power density of 20 W / cm<sup>2</sup>. Samples were studied histologically by means of optical and atomic force microscope in tapping mode in native form. The work was carried out in order to determine the specific damage to the cells membranes and possible formation of exosomes under the influence of powerful terahertz radiation. Pathological changes in the membranes are described with the exposure time increasing, up to complete destruction. Possible reasons the changes are considered.

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## Formation of nanosized graphite hydrosol under the influence of terahertz laser radiation

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The present report describes a new phenomenon - short-term exposure of water with focused terahertz radiation of free electron laser leads to the formation of nanoparticle suspension (hydrosol). Composition of the particles formed corresponds to the material of containers used in the experiment. The present report is focused on the graphite hydrosol formation. High purity (99.5%) graphite electrode was used in the experiments. Initial material and the particles formed were investigated by atomic-force microscopy. Hydrosols are technologically convenient form, suitable for scientific and technological applications which require uniform deposition and high catalytic activity, they are useful in medicine, nonlinear optics, as well as the alternate method of sample preparation for chemical and elemental analysis.

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## Two-dimensional polymer refractive micro-lenses for X-ray microscopy.

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High-performance of laser-like third generation X-ray synchrotron radiation sources provide incredible brightness and coherence, which triggered the development of compound refractive lenses (CRL). Since the first demonstration in 1996 [1] they have been realized in a wide spectrum of designs - in one and two dimensions with spherical, parabolic or kinoform profile. CRLs are fabricated from materials with high refractive index such as Al, Be, C, Ni or Si, they are able to focus high-energy radiation down to micro- and nanoscales, thus fulfilling almost every task arising in modern synchrotron beamlines.

However, the best achievable resolution of metal-based lenses is in the order of 100 nm [2], and it is mainly influenced by an internal polycrystalline micro-structure, which introduces parasitic scattering and distortions. Another limit is the diffraction-limited resolution that is determined by the numerical aperture (NA), which needs to be maximal. It is clear that the smaller radius of the parabola allows to achieve a higher resolution, approaching the diffraction limit. As for beryllium lenses, the existing manufacturing technology does not allow to produce lenses with a radius smaller than 50  $\mu\text{m}$ .

In connection with all the limitations discussed above, silicon microfabrication technology was applied to reduce the radius of lenses. Silicon one-dimensional nano-lenses have the smallest radii of several microns and are currently capable to execute focusing down to 50 nm [3]. However, silicon planar lenses have a major drawback - their one-dimensional profile makes it impossible to perform two-dimensional imaging of nano-objects. Even with perfectly aligned cross-geometry lenses, the presence of aberrations is simply inevitable.

Therefore, for fabricating small-radius lenses without parasitic X-ray scattering, we decided to use an alternative approach for lens manufacturing from amorphous polymer materials. It turns out that at the moment the most promising is additive manufacturing or 3-D printing. Two-photon absorption lithography (2PP) possesses an unprecedented geometrical freedom, allowing to print very complex designs, including overhanging and self-intersecting structures, inaccessible by conventional methods. This is a simple, reliable and relatively cheap method of forming structures with sub-100 nm feature size from a wide range of processed materials.

Applying the two-photon absorption lithography we manufactured X-ray micro-lenses from the ORMOCOMP polymer. Radius of curvature of a single parabolic surface was in the order of  $5 \mu\text{m}$ , physical aperture was  $24 \mu\text{m}$ , and the distance between the parabola apexes was  $5 \mu\text{m}$ . The focusing properties of lenses were investigated at the Micro-optics test bench in X-ray optics laboratory of the Emmanuel Kant Baltic Federal University using the Metal Jet (Excillum<sup>TM</sup>) microfocus tube with a liquid-gallium jet as an anode [4]. The detected intensity as a function of wire position obtained by knife-edge scan was differentiated and fitted by a Lorentzian function with the FWHM of  $5 \mu\text{m}$ . The resulting beam profile is depicted by a red line. Taking into account the experimental error caused by the knife stage accuracy ( $\pm 1 \mu\text{m}$ ), the size of the focused radiation is  $5 \pm 1 \mu\text{m}$ . The imaging properties of the lenses were studied at the ESRF ID13 beamline. X-ray microscopy with polymer CRL was realized at 12.7 keV. The X-ray image of the test object Siemens star was obtained at a distance of 1 m from the lens, where the resolution of 200 nm is clearly visible.

To investigate the stability of CRL to X-ray radiation, test structures (cubes) were irradiated for different periods of time, and then characterized by scanning electron microscopy, Raman spectroscopy, and energy dispersive spectroscopy. The results show that X-ray irradiation leads to oxidation of polymer material with simultaneous shrinkage possibly caused by high energy radiation induced cross-linking.

Based on the obtained experimental results, it follows that the radius of curvature can be reduced to 2-3 microns with the current configuration of our 2PP setup and the existing set of polymer materials. Applying more sophisticated 2PP techniques, such as stimulated emission-depletion lithography or diffusion-assisted direct laser writing, it is possible to reduce this value down to a submicron scale.

\*\*Acknowledgement.\*\*

The manufacturing of polymer lenses was supported by grant contract No 14.W03.31.0008, design and X-ray tests were performed by support of grant contract N° 14.Y26.31.0002.

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## Thin scintillator CsI(Tl) films for precise tomography. Development and applications.

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0.5-20  $\mu\text{m}$  thin CsI:Tl scintillation screens with high spatial resolution were prepared by the thermal deposition method. It was proposed that the spatial resolution of the prepared conversion screens can be significantly improved by an additional substrate heating as well as by deposition of a carbon layer. Also thin CsI:Tl films deposited on mylar substrates can be used for non-destructive diagnostics of the spatial profiles of low energy beams of charged particles. We present the characteristics of the scintillators in dependence on the type and temperature of the substrates.

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## Method preprocessing data acquired in tomography experiment

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For correct numerical interpretation of tomographic experiment i.e. estimation of objects attenuation coefficients it is important to obtain reconstruction of high quality which depends directly on methods of processing registered data during experiment. Data processing flow begins with its preparation for application of reconstruction algorithm. Necessary part of data processing contains subtraction of black field, normalization considering empty data and taking logarithm. This part is not enough for obtaining reconstruction of high quality when working with real data since it is not ideal. Real data includes noise and distortions due to changes of the set-up geometrical parameters during the experiment. We have analyzed five possible types of data corruptions during experiment and suggested corrections for them. The first one addresses effects from beam intensity instability during the experiment. The second one corrects shifts regarding beam decentralization. The third one is used to find rotation axis of object. The fourth one considers that radiation is polychromatic. The fifth one is about ring artefacts due to defective pixels in the detector. We have also developed a method of blind automatic adjustment of parameters for every suggested method. All these methods were tested with both real and synthetic data. For synthetic data creation we have simulated all mentioned corruptions. Both synthetic and real experiments show that suggested methods improve reconstruction quality. In real experiments level of agreement between automatic parameters adjustment and experts is about 90%.

This work was partially supported by the Federal Agency of Scientific Organizations (Agreement N<sup>o</sup> 007-/3363/26) in part of tomography setup construction and by the Russian Foundation for Basic Research (project N<sup>o</sup> 17-29-03492) in images corrections and tomography reconstruction algorithms.

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## Novosibirsk Free Electron Laser Facility

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The Novosibirsk FEL facility has three FELs, installed on the first, second and fourth orbits of the ERL. The first FEL covers the wavelength range of 90 – 240  $\mu\text{m}$  at an average radiation power of up to 0.5 kW with a pulse repetition rate of 5.6 or 11.2 MHz and a peak power of up to 1 MW. The second FEL operates in the range of 40 - 80  $\mu\text{m}$  at an average radiation power of up to 0.5 kW with a pulse repetition rate of 7.5 MHz and a peak power of about 1 MW. These two FELs are the world's most powerful (in terms of average power) sources of coherent narrow-band (less than 1%) radiation in their wavelength ranges. The third FEL was commissioned in 2015 to cover the wavelength range of 5 – 20  $\mu\text{m}$ . The Novosibirsk ERL is the first and the only multiturn ERL in the world. Its peculiar features include the normal-conductive 180 MHz accelerating system, the DC electron gun with the grid thermionic cathode, three operation modes of the magnetic system, and a rather compact (6 $\times$ 40 m<sup>2</sup>) design. The facility has been operating for users of terahertz radiation since 2004.

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## Nonlinear THz spectroscopy of graphene and GaAs quantum wells using a free-electron laser

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The mid-infrared and THz free-electron laser facility FELBE in Dresden, Germany, provides intense, nearly transform-limited picosecond pulses, which can also be combined with synchronous pico- or femtosecond pulses from near-infrared tabletop lasers, thus providing unique research opportunities to advance our knowledge on the interaction of intense mid-infrared and THz [U+FB01]elds with materials and devices. This talk reviews some recent experiments using FEL-based intense narrow-band terahertz fields, in particular pump-induced optical anisotropy and nonlinear four-wave mixing in graphene, and dressing of excitons, exciton-polaritons, and intersubband transitions in GaAs quantum wells.

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## A study of pre-nucleation intermediary structures arising in the reaction of H<sub>2</sub>PtCl<sub>6</sub> with sodium sulfide

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Recently, a series of experimental findings which are difficult to understand within the frame of classical nucleation theory (CNT) have been established. In particular, a formation of extraordinary large-scale (in comparison with the size of classical nuclei) pre-nucleation species has been observed in a range of processes such as crystallization of calcium carbonate, calcium phosphate, silica gel formation, and protein crystallization. According to in situ tapping-mode atomic force microscopy (AFM), the liquid droplet-like species from 10-50 to 200 nm and more in lateral size were arisen during the reaction, which is in good agreement with time-resolved dynamic light scattering (DLS) and small-angle X-ray scattering (SAXS) data. In this contribution, we try to examine the pre-nucleation process taking place in a similar reaction between aqueous hexachloroplatinic acid (3•10<sup>-4</sup> M) and sodium sulfide with molar ratio of 3-to-1 at room temperature. In comparison to analogous gold species, the platinum chlorocomplexes has been suggested to be more inert akin to ligand exchange. Moreover, in contrast to relatively well examined gold sulfide, platinum sulfides, both PtS and PtS<sub>2</sub>, whether in bulk or nanoscale form, are poorly studied, and the data related to their structure and electronic properties are controversial. X-ray absorption spectra were acquired in fluorescence mode on BM23 beamline of the ESRF (Grenoble, France). Energy discrimination was accomplished with double-slit crystal-analyzer using Si(111) reflections. Energy scale was calibrated using Pt foil as a standard. LII,III-edge Pt spectra were measured with energy-dispersive silicon Vortex detector situated at 90° toward incident X-ray beam, with Lα<sub>1</sub>-line was being used. Experimental data were fitted, fixing the Debye-Waller factors, and varying interatomic Pt-Cl and Pt-S distances, using the Demeter ver. 0.9.25 software suite. As fit results suggest, the replacement of Cl atoms by sulfur in the Pt first coordination sphere takes place during the 1st hour of the reaction, although some Cl ligands persist within the sphere even in 7.5 hours after the start of reaction. The nearest ligand environment of Pt is akin to Pt-S distances and coordination numbers can be assigned to PtS<sub>2</sub>-like one, with the second and more distant spheres are rather strongly disordered. Dynamic light scattering (DLS) data obtained with Malvern Nanosizer ZS instrument shows rapid growth of scatterers up to 150-200 nm for ~60 minutes after the start of reaction. Then, after relatively slow stage of scatterer enlargement, hydrodynamic diameter (D<sub>h</sub>) of scatterers reached its maximum. And finally, a slow decrease in D<sub>h</sub> was observed. Interestingly, no precipitate forms, which draws explanation of the phenomena observed within the frame of classical nucleation theory inconsistent. In conclusion, we propose a model, which includes a rapid formation of PtxSy

species within liquid clusters growing up to 150-200 nm and forming ‘dense droplets’, within which nucleation (and nucleation associated with it) takes place giving rise to solid particles, 12-15 nm in size.

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## X-ray multilens interferometers based on Si refractive lenses

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The evolution of X-ray sources such as third generation synchrotrons and free electron lasers has led to a development and utilization of coherence-related techniques. The laser-like properties of such sources allow one to realize the paraxial schemes of interferometry techniques like Young double slit interferometer or a grating interferometer. The idea of these setups is based on creating of a set of secondary sources by narrow slits. This approach has some disadvantages associated with relatively large size of secondary sources, intensity losses and low resolution of an interference pattern as well as limitations in the use of interferometers in a hard X-ray region (>30 keV) where they become weakly absorbing.

A new approach to creating of small secondary sources is to utilize interferometers based on refractive optics [1]. Recently, we demonstrated bilens and multilens interferometers which under coherent illumination generate array of mutually coherent beams focused at some distance [2-3]. The size of the focal spots is restricted to the diffraction limit and can be less than tens of nanometers. These interferometers can be used in the wide X-ray energy range while maintaining high energy efficiency. The field of applications of the interferometers based on refractive optics is not limited only by a beam diagnostic and can be extended into a beam conditioning and beam-shaping areas. Moreover, such systems of lenses open up new opportunities for development of phase contrast imaging techniques.

The work was supported by the Ministry of Education and Science of the Russian Federation grant contract № 14.Y26.31.0002.

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## High-efficient broadband THz absorber

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Perfect THz absorption has attracted increasing research interest in recent years due to the potential applications in photo-detectors [1]. Here we present the high efficient broadband THz

absorber, which is a low-resistance silicon plate with a 3-level structure formed on one side. Since low-resistance silicon has a high absorption in the THz range, it was decided to use it as a THz absorber. To increase the amount of absorption, it is necessary to reduce losses associated with Fresnel reflection. Antireflection film coating of silicon surface [2] and antireflection silicon structuring [3] have been considered. To provide broadband absorption, a 3-level antireflective structure has been realized by use of reactive ion etching (Bosch process) used for fabrication of silicon terahertz diffractive optical elements in [2,4]. Two samples of an absorber from p-type  $\rho=0.54 \text{ Ohm}\cdot\text{cm}$  silicon wafers with a diameter of 50 mm and thickness of 500  $\mu\text{m}$  were fabricated. To characterize the absorbing properties of the samples, transmittance/reflection measurement was performed by use of Menlo Systems Tera K8 THz spectrometer. The measured absorption was more than 95% in the range of 0.5-2.0 THz. It can be concluded that fabricated structures can be used as effective broadband absorbers of THz radiation and promising for sensitive elements in various THz radiation detectors. Similar structures could be used for reducing of reflection losses at the air-silicon interface of transmissive silicon optical elements of the THz range.

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## Terahertz optical components for control of high-power FEL beams

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The commissioning of coherent high-power sources of THz radiation [1] generated a need for optical elements to control this radiation. Optical elements for terahertz waves are somewhat different from the classical optical elements. In the case of high-power radiation, which damages conventional plastic lenses like polypropylene or TPX ones, the use of silicon diffractive optical elements (DOEs) solves the problem [2-6]. Binary silicon diffractive optical elements (diffractive lenses and beam splitters) for manipulation of the terahertz Novosibirsk Free Electron Laser (NovoFEL) radiation have been considered before in [2]. Applications like imaging, material ablation, generation of continuous optical discharge, and even more exotic uses of the terahertz range, e.g. the field ionization of individual atoms, require focusing of THz radiation [3,4], often with an enhanced focal depth [4]. Non-diffractive Bessel beams with angular orbital momentum (vortex beams) with different topological charges were formed using silicon binary phase axicons with spiral zone structures [5]. Such beams have great potential for use in data transmission and remote sensing. Lithographic etching of silicon substrates [2 - 5], which is practiced in fabrication



of binary elements, has disadvantages in the case of multilevel elements, when an expensive and complicated procedure of alignment of photomask is required. Binary (two-level) elements, in turn, have limited energy efficiency. A general possibility of laser-ablation microfabrication of silicon high-efficiency high-power diffractive optics for the terahertz range was shown in [6]. The present work studies high-power optics for the terahertz range. Transmission and reflective optical elements for the terahertz range are considered, as well as antireflection structuring and antireflective coatings of silicon surface [2]. The computational and experimental results are presented. REFERENCES

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## Pump-Probe setup at the NovoFEL facility for measurements of carrier relaxation processes in semiconductors

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The detection mechanism of photoconductive detectors is the generation of free charge carriers upon absorption of photons whose energy is larger than the ionization energy. The relaxation of these carriers down to their ground state ultimately limits the speed of response as well as the sensitivity. A thorough understanding of the relaxation mechanism can help to improve the performance of detector devices. In case of extrinsic germanium detectors the ionization energy is in the THz range. In this paper we investigate the relaxation of excited carriers in n-Ge:As pumped into the lowest excited state 2p0 in a single color pump probe setup at the Novosibirsk free electron laser.

## XRF SR application for studying of moonmilk from Botovskaya cave (Eastern Siberia)

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**Annotation.** The abstract presents the data on chemical composition of moonmilk samples from Botovskaya cave, the largest cave in Russia. Concerning with the host rocks, moonmilk is characterized by the increased Ca and Sr and reduced Ti, Mn and Fe contents. The investigation of this type of cave sediments with the use of XRF SR method was done for the first time.

**Introduction** Cave investigations are of a great interest for modern scientific society. The sediments of the caves are the source of geochemical, geological and paleo environmental information demanding the application of modern research technique. X-ray fluorescence analysis with the use of synchrotron radiation (XRF SR) differs from the other methods by its all-purpose usage. XRF SR method is multi-elemental, has sensitivity sufficient for the determination of rare elements, guarantees the accuracy of measurements and can be used for objects of different origin and composition. Moreover, XRF SR is a non-destructive analysis, allowing to work with a small amount of material, which is an important advantage while examining the samples from such hard-to-access objects as caves. In this abstract, we present the results of studies of two probe of moonmilk sampled from the Botovskaya cave located at Eastern Siberia.

**Objects and Methods** Botovskaya Cave is the longest in Russia and located on the Verkhnelensky karst plateau within the Central Siberian plateau. The host rocks are represented by algal limestones of the Ust-Kutskaya suite of the Lower Ordovician, which form a layer with observed thickness up to 8 m, grasped between the marine sandstones of the same formation [Filippov, 1994]. The cave is one-storey, labyrinth type, has several entrances, located in the upper part of the left side of the river Boty. The length of the cave passages is 66743 m (according to the data of the speleoclub "Arabica", February 2011). In the cave are widely represented the collapse, water mechanical, organogenic and chemogenic secondary formations, ice is observed in some cave areas. Chemogenic sediments are represented by incrustations, stalactites, stalagmites, subaquatic rims of puddles, incrustations of their bottom, cave pearls, helitites and moonmilk. Lunar milk is a special type of chemogenic formations. It is a soft clay-like substance that becomes fluid when touched. The ability of lunar milk to liquefy was called microtixotropy. In the caves, moonmilk occurs in the form of films, streaks, lumps on the walls and on the floor. The composition of lunar milk, depending on the rocks in which the underground cavity is embedded, it can be calcite, gypsum, alumino-silicate and phosphate. The chemical composition of lunar milk was determined by the XRF SR method using the Measurements technique for determining the elemental composition of samples of magmatic, metamorphic, sedimentary-metamorphic and sedimentary rocks by X-ray fluorescence analysis using synchrotron radiation as an x-ray source [Daryin A.V, 2013]. Previously it was approved that the accuracy of the results obtained with the applied measurement technique ensures the reliability of geochemical data [Markova et al., 2012]. The analysis was carried out at SRC Siberian Center for Synchrotron and Terahertz Radiation" (Institute of Nuclear Physics, SB RAS, Novosibirsk) The chemical composition of the enclosing rocks and clay sediments was determined by the method of silicate analysis (analyst Samoilenko M.M) at the CRS Earth's Crust Institute SB RAS in Irkutsk and by the method of spectral analysis (analysts Shcherban VV, Naumova AV and Vorotynova L.V.). The pictures of lunar milk crystals were done with the application of scanning electron microscope CAMScan in Moscow at the Moscow State University by S.E. Mazina and on a scanning electron microscope VEGA 3 LMH with an X-ray energy dispersive microanalysis system INCA Energy 350 / X-max 20 at the Mining Institute of the Ural Branch of the Russian Academy of Sciences in Perm by analyst Korotchenkova O.V.

**Results and discussion**

In mineral terms, both samples of lunar milk from Botovskaya cave are composed of calcite. The concentrations of the following elements were determined by XRF SR: Ca, Ti, V, Mn, Fe, Cu,

Zn, Ga, As, Br, Rb, Sr, Y, Zr, Mo, Ag (Table 1). The moonmilk showed increased contents of Ca, Ti, Mn, Ba and reduced Sr, Fe, V contents in comparison with the carbonate rocks Clarke content [Interpretation of geochemical data, 2001]. The contents of Mo and Ag do not exceed 0.00002 mass. %, so they were not taken into account. Concentrations of the main rock-forming elements in moonmilk were compared with the concentrations of these elements in the host rock composition (only the elements which content exceeded 0.01 mass. % in moonmilk were taken into account). The results are shown on figure (Fig. 1).

Fig. 1. Botovskaya cave' concentrations of chemical elements in lunar milk, host rocks and clay sediments. 1 - the average composition of lunar milk (2 samples), 2 - the composition of the clay sample, 3 - the average composition of the enclosing rocks (8 samples).

In comparison with the host rocks, the contents of Ti, Mn, Fe, Cu are reduced in the moonmilk and the contents of Ca and Sr are increased. The content of V varies insignificantly. Four main hypotheses of the origin of lunar milk are known [Hill, Forti, 1997], such as: - cryogenic (lunar milk is a residual product from limestone dissolution); - disintegration hypothesis, close to cryogenic (lunar milk is a residual product of dissolution and disintegration of bedrock under the influence of water, respiration of microorganisms and other factors); - biogenic (many bacteria and fungi can precipitate small calcite crystals as a side-product of their activity); - accumulation hypothesis (lunar milk is a product of rapid precipitation substance from a saturated solution simultaneously around many crystallization centers). Taking into account that the samples were selected in the warm part of the cave far from the spread of seasonal and permanent glaciation, the cryogenic formation of these samples is questionable. More over, observed the differences in the morphology of lunar milk crystals and cryogenic calcite powder, taken from the surface of ice bodies (Fig. 2). While cryogenic formations are characterized by lamellar splices and casts, lunar milk is composed of filose crystals forming harp bunches.

Fig. 2. - cryogenic calcite powder, , -lunar milk samples

The lunar milk formation during the disintegration of the bedrock presupposes a similarity of the composition of the enclosing rocks and the moonmilk. Considering the significant differences in the content of Fe, Mn, Ti, we can assume that this milk was formed from a solution saturated with calcium carbonate, and elements mentioned above were removed and precipitated in the residual clay. As the fig.1 shows clay differs in Ti, V, Fe, Cu, Zn contents, which are relatively higher in host rock and moonmilk. If we accept the biogenic hypothesis of mondmill formation, these elements could be absorbed by microorganisms. To prove or disprove this hypothesis, microbiological studies are necessary. This abstract shows the preliminary results of geochemical cave investigations. XRF SR method was not widely used for studies of cave sedimentations, previously. Probably exactly the application of this method will move the geochemical studies of cave sediments towards a new stage. The authors thank the all the speleologists of "Arabica" club and A.V. Osintsev personally for their help in collecting samples, the analysts Samoilenko M., Shcherban V.V., Naumova A.V., Vorotynova L.V., Korotchenkova O.V. for analysts work and Masina S.E. for electron microscope pictures providing.

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## Reflective free-form optical elements for focusing of high-power THz radiation

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Appearance of the sources of coherent and high power THz radiation [1] opened new horizons for investigations in this frequency range [2]. High attention is focused on silicon diffractive optical elements (DOE), which are used for the beam manipulation [3-6]. The lithographic etching of a silicon substrate has been used in [3, 4] to fabricate binary relief of diffractive optical elements. Lithographic etching has disadvantages in the case of multilevel elements, because an expensive and complicated procedure of alignment of photomask is required. Binary (two-level) elements, in turn, have limited energy efficiency. The laser ablation technology has been used in [5, 6] for the fabrication of multilevel diffractive lens with high energy efficiency. However, diffractive optical elements are designed for working with monochromatic radiation of a fixed wavelength [7] only. The present talk is devoted to the fabrication of terahertz reflective free-form elements for transformation of high-power beams. Aluminium elements (spherical and cylindrical mirrors, reflective axicons) were fabricated by technology of micromilling. These optical elements were tested in the beam of the Novosibirsk Free Electron Laser at the wavelength of 129.5  $\mu\text{m}$ . The measured diffractive efficiency of the spherical mirror (>94%) is in good agreement with both numerical calculations and theoretical predictions. Specific features and perspectives of the fabrication of optical elements by micromilling are discussed.

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## Vector beams in the THz range using diffractive optical elements

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Radially and axial polarized beams can be generated by superposition of orthogonally polarized Hermite-Gaussian-like beams which are equal in amplitude. The DOE which transforms Gaussian into Hermite-Gaussian modes was a plate with a phase step equal to half of the wavelength. The combination of the Hermite-Gaussian modes (1.0) and (0.1) gives us the Laguerre-Gaussian mode whose polarization strongly depends on the polarization orientation of the forming beams. Possible applications of these beams in surface plasmon setups are discussed.

## Analysis of suspended layer of alkali ions on a surface of silica sol by synchrotron X-ray reflectivity and scattering

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Solutions of SiO<sub>2</sub> nanoparticles (with typical diameter of 5–25 nm) in water stabilized by a small amount of alkali ions — known as silica sols — exhibit strong gradient of the surface potential at the sol/air interface. It has been shown previously that under such boundary conditions charged silica nanoparticles ( $Q \sim 1000 \bar{e}$ ) form a macroscopically flat layer near the surface, its depth proportional to the Debye screening length within the bulk solution, while alkali cations are accumulated in a thin suspended film directly at the surface [1].

We present new systematic data on the structural properties of suspended cation layers for silica sols enriched with Na<sup>+</sup>, K<sup>+</sup>, Cs<sup>+</sup> and Rb<sup>+</sup> ions, based on the measurements of X-ray reflectivity (XRR), grazing-incidence diffuse scattering (XDS) and small-angle X-ray scattering (SAXS). Measurements were performed at beamline ID31 (ESRF, Grenoble) with photon wavelength  $\lambda = 0.1747 \pm 0.0003 \text{ \AA}$  ( $E \approx 71 \text{ keV}$ ), beam size  $10 \times 250 \text{ \mu m}$ , peak intensity  $I_{max} \sim 10^{19}$  photons/s. SAXS data were analyzed by fitting the particles' structure factors in frames of rigid spheres approximation using ATSAS software suite [2]. XRR and XDS data were analyzed in frames of a self-consistent model-independent approach [3], which allowed us to simultaneously extract depth-graded distribution of volumetric electron density  $\rho(z)$  as well as power spectral density functions of in-plane surface roughness  $\bar{C}(\nu)$ , including "hidden" M<sup>+</sup>-layer/substrate interface, without any a priori assumptions on either sample internal structure or statistical properties of surface roughness.

Fitting of SAXS results indicates that characteristic diameter distributions of SiO<sub>2</sub> nanoparticles increase with dopation by Cs<sup>+</sup> and Rb<sup>+</sup> for  $0.6 \pm 0.1 \text{ nm}$  compared to Na<sup>+</sup>, which indicates additional adsorption of alkali ions at the nanoparticles.  $\rho(z)$  distributions extracted from XRR data show characteristic density peaks near external interface corresponding to the suspended ions layer with thickness  $d \approx 7 \dots 12 \text{ \AA}$ ; estimation of two-dimensional surface concentration of ions yields  $\Theta = (5 \pm 1) \cdot 10^{18} \text{ m}^{-2}$ , which corresponds to known theoretical predictions for two-dimensional Wigner crystal [1,4]. Effective rms heights of surface roughness obtained from XDS data give  $\sigma = (3.0 \pm 0.2) \text{ \AA}$  within spatial frequency range  $\nu = 10^{-5} \dots 10^{-1} \text{ nm}^{-1}$ , which corresponds to theoretical values in frames of capillary waves theory [5]. However, extracted power spectral density functions  $\bar{C}(\nu)$  are found to diverge substantially from the capillary waves-based prediction in frequency range  $\nu < 10^{-4} \text{ nm}^{-1}$ . This can be interpreted according to the "roughness scaling" model [6] as a superposition of two different roughness distribution, which indicates possible presence of at least two different structural phases across the surface. Additionally, preliminary experiments for reproduction of a whispering gallery (WG) phenomenon on liquid silica sol has been conducted; the data obtained in the present work are planned to be applied to further quantitative assessment and modeling of X-ray wave propagation in WG case. The present work has been supported by the Federal Agency of Scientific Organizations (Agreement No 007-/3363/26).

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## Diffraction efficiency of vortex beams in the region of 5-240 $\mu\text{m}$ generated by a binary diffractive axicon

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A Bessel beam with orbital angular momentum, or vortex beam, can be generated using diffraction from an axicon with spiral zones. In this case the Bessel function of the beam doesn't depend on the wavelength of generation and is determined by the topological charge only. Hence, for vortex beam generation with a tunable radiation source, as the Novosibirsk free electron laser (NovoFEL), a diffractive axicon becomes a useful tool. In this work THz Bessel vortex beams with topological charges  $l = 1$  and  $l = 2$  were generated using the NovoFEL and binary phase axicons made from silicon. These axicons were manufactured for 141  $\mu\text{m}$  so that a binary step is equal to  $\pi$ . Their diffraction efficiency is about 30%. Using Fourier filtration one can generate vortex Bessel beams in the full range of NovoFEL (5÷240  $\mu\text{m}$ ). In this paper a calculation of the diffraction efficiency of axicons with Fourier filtration in the NovoFEL generation range is given.

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## Diagnostics of modern synchrotron radiation sources based on refractive X-ray optics

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Recently, the creation of new technologies in the physics of particle accelerators led to a sharp increase in maximum brightness and coherence of synchrotron radiation (SR). The production of low-emittance beams is one of the key techniques for electron accelerators and synchrotron light sources. As a consequence, accurate diagnostics and monitoring of the generated X-ray source have become an important task for the operation of the new generation of synchrotrons.

The pinhole camera (Fig. 1a) is commonly used for monitoring X-ray sources of the third generation, but due to decreasing the size of emittance for SR of the new generation, this approach will limit the possibilities of observation, this means they become less effective. The using of refractive optics, such as Compound Refractive Lenses (CRL) (Fig. 1b) [1] and CRL based interferometers [2], can increase the resulting spatial resolution. On the one hand, CRL produce magnifying source image, which in turn will make it possible to observe it in details. On the other hand, a multilens interferometer can produce an interference pattern that will be more sensitive to the singularity of the source itself and his coherence properties.

The interest of imaging devices using CRLs for beam diagnostics at high-energy storage rings lies in the possibility of choosing the electron beam as the object to image and thus obtaining a direct image of the source with a simple experimental setup [3].

We propose a method for diagnosing new generation of low-emittance SR sources based on refractive X-ray optics. The proposed methods will be in demand, taking into account the construction of new synchrotrons in Russia.

Acknowledgement.

These developments were supported by Ministry of Education and Science of the Russian Federation (Contracts N<sup>o</sup> 14.Y26.31.0002 and 02.G25.31.0086).

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## Beryllium X-ray optical properties: from highly porous to the single crystal material

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Beryllium is an important material for a wide range of scientific applications due to its unique properties. Because of its low atomic number and very low absorption for X-rays, beryllium is widely used for windows of X-ray tubes and synchrotron sources, detectors and as sample holders for special X-ray spectroscopy applications. It is the best material for X-ray refractive optics, which is extensively employed at synchrotrons and X-ray free electron lasers. Beryllium lenses are able to focus high-energy radiation up to micrometer scales, allowing to solve easily different experimental problems arising at synchrotron beamlines. They are used as condensers, micro-radian collimators, low-band pass filters, high harmonics rejecters, beam-shaping elements [1-5]. Two-dimensional Be lenses are the driving force in the development of Fourier optics, coherent diffraction, and imaging techniques [6-12].

However, almost all beryllium grades, used for X-ray optics manufacturing, are sintered materials, which have inevitably internal micro- and nanograined structure and relatively high beryllium oxide (BeO) concentration. BeO forms an inhomogeneous internal structure in beryllium – each beryllium grain covered by thin BeO film which leads to strong small- and ultra-small angular X-ray scattering and losses of intensity [13].

In this paper, we present experimental results of the structure and X-ray optical properties studies of a wide range of beryllium types: sintered beryllium with micro- and nano-grain structure (US and RF production), beryllium dihydride (BeH<sub>2</sub>) and extreme Be form – highly porous beryllium. The obtained results were carefully compared. The influence of the beryllium microstructure on the optical properties of the compound refractive lens was studied and successfully demonstrated for the first time in the coherent transmission X-ray microscopy mode. The direct dependence of the image quality on the average beryllium grain size was found. It allowed us to make recommendations for the use of the nano-grained beryllium as the main material for fabrication of X-ray optics for imaging applications.

In addition, a new promising material for X-ray optical applications was proposed - highly porous beryllium. Due to its low density and high porosity, this material allows manipulating the spatial coherence length, thereby changing the effective source size and removing the undesirable speckle structure in X-ray imaging and microscopy experiments with almost no attenuation of the beam [14].

We are confident that these new beryllium materials are very promising for imaging techniques and this will allow us to use the full potential of novel coherent X-ray sources.

**\*\*Acknowledgments.\*\*** This research was supported by Ministry of Education and Science of the Russian Federation (contract N<sup>o</sup> 14.Y26.31.0002, 16.4119.2017/PCh).

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## Coupled wave equations for acousto-optic diffraction of a divergent light beam in absorbing medium

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Theory of acousto-optic (AO) diffraction of a long wavelength radiation have to take into account divergence of a light beam upon propagation. The effect of a light beam divergence is especially strong in terahertz (THz) range. In this spectral range the most suitable AO media are absorbing and it's light absorption coefficient is about 1 1/cm. Theory of AO interaction taking into account divergence of a light beam and a medium absorbance is developed and coupled wave equations are derived.

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## Computer based test bench for modulation of terahertz FEL radiation power.

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We propose simple method to modulate output radiation power in FEL oscillator which allows to adjust smoothly the average radiation power as well as to obtain short (down to 35 microseconds) radiation macrobunches. This method is based on periodic shift of electron bunch phase with respect to FEL radiation bunch stored in optical cavity which results in lasing suppression. The phase shift frequency required to suppress lasing is relatively small and it does not change significantly electron bunch repetition rate. To demonstrate feasibility of the proposed method we created computer based test bench which is described in this paper. The test bench comprises standard CAMAC blocks: G0609 - modulator timer and G0601 – clock generator. The electron bunches phase shift is carried out by a programmable skipping of the reference frequency periods which are used to start current modulator in electron gun. The skipping is controlled by computer code which runs under real-time operating system LynxOS v3.1. This environment allows to switch modes with an accuracy of 16 microseconds. The clock-generator is used to send synchronization signals to radiation user equipment.

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## Mini-Transfocator for X-ray Microscopy

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X-ray compound refractive lenses (CRL) with parabolic profile became standard components at beamlines of modern synchrotrons. They can be adapted to X-ray energies from 2 to 200 keV to conduct research by modifying their composition and number. In order to simplify alignment and positioning of lens assembly and to provide permanent energy and focal length tunability transfocators were proposed. Used extensively as beam-conditioning devices in optics hutches, transfocators are typically quite bulky because of constructional features: pneumatic actuators for lenses cartridges, cooling and vacuum systems. Moreover, the system of lens cartridges makes it impossible for smooth variation of focus and magnification and therefore does not fully meet the needs of some experimental applications.

In this work we propose a new compact transfocator based on CRL. The Mini-transfocator is designed to combined use of 1D and 2D CRL and can be used to change number of focusing lenses. Small overall size (150x90x100 mm) and weight (approx. 2 kg) of the device allow significantly simplifying the setup of the experiment for any hutches including experimental hutch. In contrast to cartridge-type transfocators the Mini-transfocator change the number of focusing lenses by mechanically moving lenses one-by-one. Thus it provides smooth variation of focus and magnification for applications requiring compact and lightweight zoom-optics (with variable focal length).

The Mini-transfocator was successfully tested for optical accuracy, mechanical performance and repeatability at the Micro-optics test bench in the X-ray optics laboratory of the Immanuel Kant Baltic Federal University and at the PETRA-III P14 beamline. Results indicate that the Mini-transfocator is suitable for a wide range of applications, being either a beam collimation system or a short-focal magnifying objective.

**Acknowledgements**

Ministry of Education and Science of the Russian Federation (contract N<sup>o</sup> 14.Y26.31.0002, 16.4119.2017/PCh) supported this research.

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## Electron Outcoupling System of Novosibirsk Free Electron Laser Facility— Lasing Efficiency Estimations and Schemes Comparison

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The electron outcoupling scheme was proposed for the high power FELs with optical cavities to avoid the power limitation due to overheating of reflecting mirrors. For this purpose, the electron beam is prebunched while passing the first undulator along the optical axis and then is deflected by a small angle, directed to one of the next undulators and emits the high-power radiation. Electron outcoupling system is installed on the third FEL of the Novosibirsk Free Electron Laser facility (NovoFEL). The FEL consists of three undulators, dipole correctors and two quadrupole lenses assembled between them. There are two different configurations of the system since the electrons can be deflected in either the second or the third undulator. The estimates of the lasing power and the comparison of electron outcoupling schemes of NovoFEL are considered. The experimental tests of electron outcoupling are in progress.

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## Propagation and reflection of terahertz surface plasmons through polyimide films

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Terahertz (THz) surface plasmons (SPs) can be effectively used in THz communication lines, information processing devices, spectroscopic measurements of submicron dielectric layers, and metal surface quality control. Such applications necessitate implementation of various optical schemes, including splitting of THz SPs on the guiding surface. This work is the first experimental demonstration of the fact that a polyimide kapton film of submillimetre thickness can effectively split THz SPs. Measurements of the transmission and reflection coefficients of SPs on gold-ZnS layer plane structures have shown good accordance with theoretical calculations.

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## Design features of variable-period wide-aperture undulator

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New undulator for the first stage of Novosibirsk free electron laser (FEL) was developed. Installation of the new undulator will significantly increase radiation tuning range of the first stage FEL due to its features, among them are possibility to vary period of the undulator continuously, possibility to change number of periods and small period to aperture ratio. Mechanical construction is described and some magnetic field simulation results are presented in the article.

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## The radiation monitoring system of the Novosibirsk FEL

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Accelerator recuperator FEL works in the following energy range: 1st FEL to 12 MeV, 2nd FEL - 20 MeV, 3rd FEL - 40 MeV. The electron beam current is about 10 mA. When the beam is lost on the walls of the vacuum chamber, the dose rate of bremsstrahlung reaches several kilo gray per hour. After recuperation, the beam (1.5 MeV) enters into the copper dump, the contribution to the dose rate from the dump reaches several hundred gray per hour. Radiation monitoring system FEL controls radiation in the working rooms and the acceleration hall. The three-meter concrete walls of the acceleration hall provide reliable protection, in neighboring rooms the dose rate does not exceed the maximum permissible levels. TLD dosimeters provide integrated dose monitoring, and ionization chambers provide real-time monitoring. At present, ionization chambers are being replaced by Geiger counters. Part of the sensors is in the acceleration hall. The sensors are placed in lead containers, and installed near the vacuum chamber. It makes it possible to build a profile of radiation levels along the vacuum chamber, and localize the places of beam losses. Several sensors are placed in check points, they are monitoring integral doses. Currently, it is planned to connect sensors that will monitor induced radioactivity. The software part of the system is based on Scientific Linux. It uses a distributed control system EPICS, which makes it easy to create client programs and data acquisition system. The server is implemented on the controller with CANbus interface, which is connected to the block controls radiation sensors.

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## Measurement of hard X-ray spectra on femtosecond laser facility

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Properties of hard x-rays from tantalum targets, irradiated by femtosecond laser, were investigated experimentally. During experiments, focal spot size have been varied from 3  $\mu\text{m}$  to 10  $\mu\text{m}$ , laser pulse intensity on target changed from  $10^{19}$  W/cm<sup>2</sup> to  $10^{20}$  W/cm<sup>2</sup>. Two spectrometers based on gray filters methods were employed for investigation of hard x-ray yield. Spectrometer on image plates was applied for detection in photon energy range  $E=100-600$  keV, scintillation spectrometer was applied for detection in the range  $E>400$  keV. Using scintillation spectrometer, spectrum temperature and relative hard x-ray yield in every laser shot were quantified. By

placing spectrometer at different angles to laser radiation axis, an x ray direction pattern was investigated. All in all it was found that: - x-ray spectrum temperature in intensity range from  $10^{19}$  to  $10^{20}$  W/cm<sup>2</sup> equals to  $\sim 1$  MeV independently on direction; - conversion of laser pulse energy into x-rays in the range from 0.1 to 1 MeV equals to  $\sim 5 \cdot 10^{(-4)}$  1/sr; - X-ray yield has maximum in the direction of laser radiation propagation.

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## Exploration of soft x-ray yield from targets irradiated by ultrashort laser pulses at intensities from $10^{17}$ to $10^{19}$ W/cm<sup>2</sup>

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Hot plasma is formed at irradiation of matter by high intensity laser radiation with ultrashort pulse duration. This plasma is a bright source of continuous and characteristic x-ray radiation. Due to short luminescence time ( $\sim 10$  ps) and micrometer size of luminescence region ( $\sim 10$   $\mu$ m), such sources are applied in different sciences [1-3]. At a picosecond laser facility, experiments were conducted aimed at exploration of soft x ray yield (in the photon energy range of 0.6-4.5 keV) at laser pulse intensities  $10^{17}$  -  $10^{19}$  W/cm<sup>2</sup> from Al, Cu, Mo, W and Pb targets. These experiments allowed to define dependence of electron temperature on laser pulse intensity and dependence of laser pulse energy conversion efficiency into soft x-rays from atomic number of target material. Plasma electron temperature rises from 0.4 keV to 0.6 keV with intensity increase from  $10^{17}$  to  $10^{19}$  W/cm<sup>2</sup>. Conversion efficiency of laser radiation into x-rays changes from  $0.5 \cdot 10^{(-3)}$  to  $1.5 \cdot 10^{(-3)}$  1/sr for Al and Pb targets respectively.

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## A study on structural features of bimetallic Pd-M/C (M: Zn, Ga, Ag) catalysts by EXAFS

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Nowadays, palladium supported on different carriers is one of the most intensively studied catalytic systems. Pd-based catalysts exhibit excellent activity in such important processes as hydrogenation of organic acids and oils, selective hydrogenation of acetylene to ethylene, oxidation of methane and carbon monoxide, etc. An enhancement of catalytic properties is achieved by using

Pd-based bimetallic systems [1]. In the case of bimetallic catalysts for the selective hydrogenation of acetylene to ethylene, a key role is played by a dilution of Pd atoms with atoms of modifying metal [1-2]. Doping the Pd-based catalysts with second metal leads to an increase in the distance between neighboring atoms of palladium and the creation of the new type of active sites that facilitate an improvement of the process selectivity thus increasing the yield of target product. At the same time, the contribution of electron interaction of Pd with modifier is of significant importance. Atoms of second metal act as donors of electron density for vacant d-orbitals of palladium that suppresses an affinity of Pd towards adsorption of unsaturated compounds. It facilitates ethylene desorption and impedes its complete hydrogenation to ethane.

The aim of the present research was to study in detail the local structure of carbon-supported bimetallic Pd-Ag, Pd-Zn and Pd-Ga catalysts by Extended X-Ray Absorption Fine Structure spectroscopy (EXAFS). The EXAFS method offers the possibility to obtain the interatomic distances and near neighbor coordination numbers of the supported metal nanoparticles. The supported bimetallic catalysts were prepared via an incipient wetness impregnation of the pretreated by a solution of nitric acid porous carbonaceous graphite-like material Sibunit used as a support with solutions containing corresponding metal nitrates. Then, the samples were dried in air and reduced in a hydrogen flow at 500°C. The palladium loading was 0.5 wt%, and Pd:M ratio was 1:1. The research was performed at the “Structural Materials Science” beamline of the Kurchatov synchrotron radiation source (SRC “Kurchatov Institute”, Moscow).

According to the EXAFS data, the Pd-Ag/C catalyst contains separate nanoparticles of metallic palladium and silver as well as bimetallic particles of Pd<sub>x</sub>Ag<sub>y</sub> nanoalloy with stoichiometry close to Pd:Ag = 1:1. The analysis of the Pd and Zn K-edge absorption indicates that the Pd-Zn/C catalyst contains the bimetallic nanodispersed PdZn phase, which characterized by the reduced Pd-Pd coordination number; the Zn-doped catalyst contains metallic Pd, PdO and ZnO oxide formed as a result of contact with air. The composition of PdZn phase is deviated from the stoichiometry towards its enrichment with zinc atoms and the surface segregation of zinc from the bimetallic structure. As it follows from the Pd and Ga K-edge EXAFS data, the amount of the joint Pd-Ga phase in the Ga-doped catalyst is quite small. Some part of the alloyed Pd-Ga particles as well as nanoparticles of metallic palladium can be blocked by a layer of oxidized gallium, which goes out on the surface owing to segregation.

This work supported by the Russian Foundation for Basic Research (grant 17-33-50019). Authors are grateful to E. V. Khramov for his support in the XAS measurements.

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## Calculations and measurements of the dose rate of bremsstrahlung for the three operating regimes of the Novosibirsk FEL

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The powerful free electron laser of the Siberian center for photochemical research consists of an injector, an accelerator RF structure, one vertical and four horizontal paths, and dump. The 1st stage of the FEL has one vertical track, the electron energy up to 12 MeV. 2nd FEL has two horizontal tracks on the 1st track of the electron energy 10 MeV. On the 2nd track energy is equal to 20 MeV. 3rd stage FEL has four horizontal tracks, with the passage of each track energy increases by 10 MeV, on the 4th track energy is equal to 40 MeV. The electron beam current is about 10 mA. When the beam is lost on the walls of the vacuum chamber, the dose rate of bremsstrahlung reaches several kilo gray per hour. After recuperation, the beam (1.5 MeV) enters into the copper dump, the contribution to the dose rate from the dump reaches several hundred gray per hour. Levels of neutron dose rate can reach several gray per hour. The high level of

radiation in the accelerator hall leads to the degradation of materials of technological structures and the activation of parts made of stainless steel and copper. The three-meter concrete walls of the acceleration hall provide reliable protection, in neighboring rooms the dose rate does not exceed the maximum permissible levels. FEL radiation monitoring system of the controls radiation in the work's rooms and in the accelerator hall. The system monitors only photon radiation. Photoneutrons are formed in the area of these energies, neutron radiation is not controlled by the system. The system does not control induced radioactivity. The paper presents calculations of the dose rate of photons and neutrons, as well as measurements of the dose near the gate of the hall accelerator, which allows to predict the radiation situation in the acceleration room.

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## Instrumental and methodological provision for structural SAXS/WAXS studies of biological nanosystems using synchrotron radiation

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Until creation of synchrotron radiation (SR) sources in the Siberian and Moscow regions (VEPP-2, VEPP-3, VEPP-4, and Siberia-2), in the country there has not been instrumental and methodological basis for our research in the field of structural biology of tissues. Long-time work of our collective direct on studying the mechanisms of structural and functional stability of the native and transformed tissues of humans and animals under various physicochemical influences. Such heterogeneous multicomponent ensembles of living systems are characterized by high order with structure periods in the nanometer range of the order of 1-100 nm at extremely low concentration in native tissues and weak scattering ability.

The report discusses the instrumental and methodological developments using SR, as well as some test results of our SAXS/WAXS diffraction studies of biological nanosystems in different physiological states. X-ray stations based on monochromatic ( $\lambda=\text{const}$ ) and energy ( $=\text{const}$ ) diffractometry methods were created on the working channels of SR sources. The figure shows their generalized block scheme with fundamental differences for each method in the systems of SR beam formation and registration of X-ray diffraction patterns on the basis of positional-sensitive coordinate or power-dispersion detectors. New approaches to the formation of the SR beam were implemented at the stations. So, there was carried out next operations for the method  $\lambda=\text{const}$ : the reversed version of the arrangement of X-ray optics of the zoom lenses based on the crystal-monochromator and polysectional mirrors; high-speed X-ray diffraction with simultaneous registration of micron periodicity of structure in visible spectrum; distantly controlled methods of combination the SR beam and object, and adjustments of the station at minimal background. There was performed some modifications for the method  $=\text{const}$ : registration in the meridional (interval 0-50 mrad) and sagittal (interval 0-25 mrad) directions at  $5 \times 10^{-2}$  mrad accuracy; collimating device with mirror zoom lens; cooling of object and its scanning. These modifications allowed increasing the signal-to-noise ratio by 6-7 times.

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## FIB diamond micromachining for X-ray applications

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The high degree of coherence, brightness and power of X-radiation from free-electron X-ray lasers (XFELs) and 4-generation synchrotron sources [1] requires the development of new X-ray optics capable of withstanding extreme thermal and radiation loads. Diamond has excellent optical properties for these X-ray sources: high temperature stability and chemical inertness, relatively low absorption of hard X-rays, high thermal conductivity and low coefficient of thermal expansion. Due to its high strength and hardness, the diamond cannot be easily subjected to direct mechanical or chemical machining. Therefore, laser ablation is one of the most suitable methods for manufacturing diamond X-ray refractive optics [2-3]. However, the main disadvantage of this method is the relatively high roughness of the diamond optical surface ( $Ra \approx 0.3 \mu\text{m}$ ). It is necessary to reduce the roughness of the optical elements with high precision for obtaining a high-quality X-ray lenses. The focused ion beam (FIB) method is the best candidate for solving this problem due to its possibility of physically spraying any materials on the micro- and nanoscale [4].

In this work, we present a new approach for final polishing of the diamond surface of refractive lenses by ion beam lithography method. Currently, ion lithography is widely used for the modification and fabrication of new X-ray optical elements [5, 6]. We demonstrate the new results of smoothing two-dimensional parabolic refractive lens surface by the dual-beam system ZEISS Crossbeam 540. This approach has enabled us to reduce the roughness of the diamond single crystal refractive lens with 1mm aperture and 200  $\mu\text{m}$  radius of curvature. The obtained results are very promising and we hope that the diamond optics will be applicable to the tasks of high-resolution microscopy on the extremely bright sources.

**Acknowledgments** This research was supported by Ministry of Education and Science of the Russian Federation (contract N<sup>o</sup> 14.Y26.31.0002, 16.4119.2017/PCh).

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## The dynamics of carbon nanoparticles size at the detonation of TNT-RDX charges

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During the detonation of oxygen-deficient high-explosive nano-sized condensed carbon particles are formed. Studying of the carbon condensation process is necessary to verify the equation of state of the explosion products.

In this work, we carry out the dynamic measurement of small-angle x-ray scattering (SAXS) during the detonation of molten charges of TNT–RDX of different diameter. The dynamics of the average size of carbon particles are recovered from the SAXS data by new multi diameter method.

This work was supported by the Russian Foundation for Basic Research (projects No. 16-29-01050 and 17-03-00251).

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## Further development of the detector for imaging of explosions, present status.

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Detector for imaging of explosions (DIMEX) is successfully exploited at the Siberian Synchrotron Radiation Center for more than ten years. DIMEX can measure spatial distribution of SR after an exploding sample from individual bunches without mixing radiation from them. Recent upgrade of the gaseous version of the detector with custom ASIC DMXG64 allowed to increase the number of stored frames in an experiment from 32 to 100 and improve rate capability from 2 MFrames/s to 8 MFrames/s. The new version of DIMEX-G is operating at two stations at beam line 0 at the VEPP-3 and at beam line 8 at the VEPP-4M storage rings. Further improvement of the parameters of the DIMEX will be achieved with the Si-strip technology. The new prototype detector DIMEX-Si is assembled based on Si-strip sensor with 96 channels with 50 microns pitch and with electronics based on new custom ASIC DMXS6A with 32 analog memory cells in each channel. The new ASIC allows to reach rate capability of 50 MFrames/s and increase maximum registered photon rate by two orders of magnitude as compared to DIMEX-G.

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## Study of main characteristics of the gaseous and Si versions of the detector for imaging of explosions DIMEX.

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Systematic measurements of main parameters of the detectors for imaging of explosions DIMEX-G and DIMEX-Si are described. DQE, spatial resolution and timing parameters are assessed. The methods of DQE and spatial resolution measurements are described in details and sources of systematic errors are determined. Parameters of the gaseous and Si versions of the DIMEX are compared.

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## Powerful and narrowband THz emission from a plasma with counterpropagating electron beams

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Nowadays development of compact and cheap sources of powerful and narrowband THz radiation is highly demanded in science and technology. One of the most perspective nonlinear medium allowing to generate electromagnetic waves in the terahertz frequency range is plasma. It doesn't have damage thresholds and can sustain electromagnetic fields with extremely high amplitudes. However, potential oscillations induced in a plasma by laser or particle beams cannot be directly converted into vacuum electromagnetic emission due to the cutoff at the plasma frequency. To avoid these challenges, we have proposed a new scheme [1] in which nonlinear interaction of counterpropagating laser-induced plasma waves leads to EM emission at the doubled plasma frequency. Such a process can proceed in a uniform plasma without external magnetic fields and is not sensitive to the effect of plasma screening. That is why it becomes possible not only to significantly increase the power and energy of THz pulses, but also to provide a narrow line width of the produced radiation (near 1%). Among other advantages of this generating scheme are possibilities of tuning the central radiation frequency by varying the plasma density as well as a smooth transition to broadband radiation regimes with the increase of plasma waves amplitudes. Physically, generation of EM radiation in the proposed scheme is based on scattering of one Langmuir wave on the periodic perturbation of plasma electron density produced by a counter-propagating Langmuir wave with the same longitudinal wavenumber. The net nonlinear current produced by both waves is not zero only in the case when transverse profiles of their electrostatic potentials differ from each other. In the recent paper [1], we have proposed to drive these colliding waves by short laser pulses. Despite the high absolute values of THz power and energy, which can be achieved with the use of petawatt-class lasers, efficiency of laser-to-THz energy conversion in such a scheme does not exceed the level 0.1%. The main reason for that is the low efficiency of wakefields excitation and short life-time of excited wakes. In this work, in order to drive colliding plasma waves more efficiently and increase the duration of THz generation, we propose to use kiloampere relativistic electron beams of nanosecond duration instead of short laser drivers. Such beams can reach high power (tens of GW) and are able to continuously pump plasma waves at ionic times via the two-stream instability. We carry out particle-in-cell (PIC) simulations and demonstrate that the beams-to-THz power conversion can be highly efficient (up to 7%) if colliding plasma waves are driven by long-pulse electron beams with different transverse sizes. It is shown that low-emittance 1 MeV electron beams capable of focusing in mm-scale spots can generate sub-GW terahertz radiation.

Simulations were performed using resources of Novosibirsk State University. This work is financially supported by the Russian Foundation for Basic Research (grant 18-32-00107).

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## Compensation of undulator focusing variation at FEL radiation wavelength tuning

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Free-electron lasers (FELs) have a key advantage over other types of lasers: the possibility of an operative and smooth tuning of the wavelength of monochromatic radiation. However, this

process causes changing of the focusing strength of the undulator and, consequently, requires the matching of the rest of the FEL magnetic system. As a solution to this problem, two approaches were proposed: 1) conservation of the transport matrix of the section that includes the undulator; 2) matching the input and the output Twiss parameters with the optimal ones at the center of the gap that depend on the current of the undulators. In this paper the process of radiation wavelength tuning of the first stage of the Novosibirsk FEL (the radiation wavelength range: 90-220  $\mu\text{m}$ ) is studied. The variation of the focusing strength of undulators was compensated by the tuning of the currents of the 8 quadrupoles at the undulators track. Two methods to find the rule for tuning the currents of quadrupoles were used: 1) numerical optimization of the transport matrix; 2) solution of the system of the ordinary differential equations for the quadrupole currents as functions of the undulator current. By these numerical methods some example regimes that enable to tune the radiation wavelength in the range 120-130  $\mu\text{m}$  were found. The experimental verification confirmed the validity of our approach. During the experiments the conservation of the transport matrix was monitored by the response matrix diagnostic. The search of new regimes with the larger tunable radiation wavelength range is in progress now.

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## Investigation of Re and Nb doped molybdenum disulfide by x-ray spectroscopy methods

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Transition metals disulfides are currently being actively studied due to their interesting physicochemical properties. Modification of these compounds by doping atoms makes it possible to obtain materials with the necessary electronic properties. In particular, molybdenum disulfide is promising for use in such areas as catalysis [1], transistors fabrication [2], production of lithium-ion batteries [3]. In this work we investigated solid solutions with stoichiometry  $\text{Mo}_{1-x}\text{M}_x\text{S}_2$ ,  $x = 0, 0.05, 0.1, 0.15, 0.2$  based on  $\text{MoS}_2$ , doped with electronically redundant Re atoms and electron-deficient Nb atoms. The structure of the conduction and valence band will depend strongly on concentration and type of doping atoms. The dependence of the band gap on the dopant concentration was experimentally determined from the valence band XPS and the XANES edges. XANES and valence band XPS were obtained on the Russian-German RGLBL beamline at BESSY II synchrotron source. The use of the TEY and PEY modes for XAFS experiments made it possible to obtain surface spectra and the spectra of bulk and surface superposition. The effect of surface defects on electronic structure was evaluated. To complete the study the XPS and the XES were obtained. According to the results of XES and XPS studies, we assume that Re-clusters are formed even at low dopant concentrations in the samples. This assumption was confirmed by the HRTEM method [4].

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## Effect of Terahertz Radiation on the Strength of Binding in Albumin Complexes with Nitrogen Oxide and Oxygen

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The influence of terahertz radiation on the transport characteristics of serum albumin manifests itself as changes in bonding with biologically significant species, such as molecular oxygen and nitrogen oxide. Causing no rupture of covalent bonds, terahertz radiation may affect rotational and rovibrational transitions in biomolecules. To observe the effects of this kind, it is necessary to study their chemical consequences. One of these consequences is a change in the transport characteristics of albumin, namely strengthening or weakening of protein bonding with the species to be transported, in particular O<sub>2</sub> and NO. These changes were successfully followed with the help of in situ spin probing and spin trapping. We demonstrate that irradiation within the terahertz range has a noticeable effect not only on the amount of specific adsorption centers in albumin molecule but also affects the affinity of these centers. After irradiation, albumin binds oxygen to a higher extent than the non-irradiated preparation. This was concluded because we observed an increase in the intensity of the EPR signal of the nitroxide, a spin probe which was formed directly in the system through the oxidation of the precursor nitron molecule at the paramagnetic centers of albumin. In addition, irradiation resulted in a decrease in the mobility of these paramagnetic centers, which was observed as an increase in the width of EPR signals in the spectrum of the sample under investigation. The interaction of preliminarily irradiated albumin with NO caused stronger local acidification than the interaction of non-irradiated albumin. Acidification was measured with the help of a pH-sensitive imidazoline spin trap. This allows us to conclude that NO → NO<sub>2</sub> conversion proceeds at a higher rate in the presence of irradiated albumin, which means that the albumin – NO complex becomes weaker after irradiation. Further studies are necessary to identify the rotational transitions responsible for the observed effects. As the initial step, we carried out the simulation of the interaction of oxygen and nitrogen oxide with the functional groups of albumin molecule. The changes in bond strength in the studies complexes may be due to a conformational rearrangement in the proline-containing fragment. This agrees with the known facts that the terahertz radiation affects the ternary structure in which a special role is played by the proline fragment.

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## The use of X-ray fluorescence analysis using synchrotron radiation to study the relationship between chemical elements and phenolic compounds in the blue honeysuckle plants

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Presently studies of the role of trace and macro elements in the biogenesis of diverse biologically active phenolic compounds (PC) in medicinal plants have become increasingly actual due to PC great diversity and specific functions in physiological processes. Beneficial properties of blue honeysuckle *Lonicera caerulea* L. s.l. (Caprifoliaceae Juss.) are attributed to their PC pool, as well as to the trace and macro elements' content. Earlier research carried out in the *L. caerulea* natural population on the Kurai Ridge of the Altai Mountains (Russia) revealed significant differences in PC content among the plants growing in similar environments. The aim of the study was to reveal the possible effect of soil elemental composition on trace and macro elements' content and on the different PC classes in honeysuckle leaves. The soil and phytomass samples for analyses were collected in 4 sites of the *L. caerulea* population area in the Saryachik River valley on the

Kurai Ridge of the Altai Mountains. The *L. caerulea* leaves were collected at the fruit ripening stage. The total content of trace and macro elements in soil and plants was determined by X-ray fluorescence analysis using synchrotron radiation (XRF SR) at the Elemental Analysis Station of the VEPP-3 storage ring at the Siberian Center for Synchrotron and Terahertz Radiation, Budker Institute of Nuclear Physics. Monochromatized synchrotron radiation with energy of 23 keV was used for fluorescence excitation. The irradiation time of each sample ranged from 150 to 400 s. Individual energy peaks corresponding to the measured elements and the peak area, which in turn corresponded to the concentrations of elements, were calculated for the resulting characteristic X-ray spectrum. Nineteen elements were identified in our studies (K, Ca, Ti, V, Cr, Mn, Fe, Co, Ni, Cu, Zn, As, Br, Rb, Sr, Zr, Nb, Mo, and Pb). The concentration of chemical elements in soil extracts was measured by atomic absorption. Chemical elements' content in soils of the Saryachik River valley, as determined by XRF SR, showed considerable differences among blue honeysuckle habitats in the bulk contents of some macro (Ca, Fe) and trace (Ti, Mn, Sr, Zr, V, Zn, Rb, Ni, Cu, Pb, Co, As, Cr, Br and Mo) elements, as well as in labile forms of K, Ca, Fe, Mn, Sr, Zn, Ni, Cu, Pb and Co. Statistically significant correlation between bulk and labile forms of chemical elements was revealed only for Ca, Fe and Pb. Macro and trace elements' content in honeysuckle leaves, as well as their physiologically informative ratios, also showed significant variation. Statistically significant correlation was found between hydroxycinnamic acids content and Cu, Rb, Mo and V in leaves, whereas decrease in K/Rb ratio in leaves was correlated with increased chlorogenic acid concentration. Significant increase in main PC classes was found in habitats with high soil Sr and Ca content, leading to decreased soil K/Ca ratio. The same habitats were characterized by increased Cu/Zn and Fe/Zn and decreased Fe/Cu Fe/Ni ratios. The obtained results suggest possible participation of biologically active polyphenols in regulating macro- and trace elements' uptake by blue honeysuckle plants.

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## Test results of 6 T superconducting solenoid for THz radiation applications

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This project is related to new spectroscopy method in little-developed THz range. The method is founded on using of a free electron laser with high spectral power radiation which can be smoothly tuned in desirable range of spectrum. The objects of research of this method are fast processes in physics, chemical and biological reactions. Uniform magnetic field of 6 T value in the research area can considerably increase possibilities of this method. The magnetic field will modulate radiation of free molecules induction on characteristic frequencies of the Zeeman splitting that gives more possibilities of identification of molecules having even weak magnetic momentum. Moreover, the use of magnetic field allows essentially increase sensitivity of this method due to almost complete separation of the weak measuring signals from powerful radiation of the laser. A superconducting solenoid was manufactured and tested for such an application. Its design and test results will be presented.

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## Modeling and measuring radiation pattern of thermally stimulated infrared surface plasmon-polaritons diffracting on metal bar with rectangular wedge

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Surface plasmon polaritons (SPPs) on a conducting surface can be generated not only by an external source (such as a free electron laser), but also by thermal fluctuations of the density of conduction electrons within the skin layer of a metal body. This type of SPPs is called thermally stimulated. TSPPs are a kind of evanescent surface electromagnetic waves, which is inherently nonradiative, but can be converted into bulk waves on surface inhomogeneities. In particular, it is known that infrared TSPPs arriving to an edge of the metal body diffract on it and generate directional bulk radiation [1]. Such radiation can be effectively used, for example, to create thermal sources of IR radiation, for heat exchange between metal objects and for passive thermal imaging. In the report we consider the problem of correct calculations and measurements of the TSPP radiation pattern (RP). SPP RP calculations rest on the analytical model obtained by Kotelnikov [2], who applied the Sommerfeld-Malyuzhinets method to calculate SPP RP for the case of rectangular conductive wedge. The model developed by him allows correct RP representation for monochromatic SPPs both in the near and far wave zones. This property of the model enabled us to generalize it to the case of broadband TSPP radiation. In the report we show that due to TSPP existence the edge of a metal body facet is a source of narrowly directed IR radiation with the black-body type spectrum. For correct measurements of RP was proposed a modified scheme of linear scanning by using two additional screens. The results obtained for the maximum intensity of the radiation from TSPPs are consistent with those obtained earlier [3]. Namely: the contribution of TSPPs to the thermal radiation of the optically polished facet due to TSPP scattering by inhomogeneities is negligible, and the maximum of the RP is deviated from the plane of the facet by 2-3 degrees. In addition, it was confirmed that the increase in the radiation intensity at the RP maximum with increasing temperature lags behind the increase in the intensity of the thermal radiation of the given metal body. This work was supported by the Russian Foundation for Basic Research (grant 18-32-00930).

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## Calculations for experiments with vortex beams at the Novosibirsk free electron laser facility

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Beams of photons with an orbital angular momentum (or vortex beams) are of interest both for fundamental physics and for various applications. In the visible range, there are many ways to generate such beams, for example, using spatial light modulators (SLM). In the terahertz frequency range, for which SLM has not yet been developed, diffractive optical elements (DOEs) remain one of the most effective devices for generating vortex beams. For example, in the case of

a high-power terahertz radiation source, such as the Novosibirsk free-electron laser, radiation-resistant silicon phase DOEs are used to transform a Gaussian beam into beams with a specified mode composition. To support the experiments, a program in Matlab environment with an easy-to-use interface has been written to simulate radiation transmission through optical systems consisting of a sequence of amplitude-phase elements. The calculations were performed within the framework of the scalar theory of diffraction. The software calculates the Rayleigh-Sommerfeld integral in the Fresnel approximation using combination of two approaches of the impulse response method and the transfer function method that ensures the solution correctness in the entire Fresnel diffraction region. In this paper we describe the program and present the results of calculations of the formation of vortex beams by binary phase axicons and their transformation after transmission through two-dimensional periodic gratings of round holes. It is found that behind the lattice in the planes corresponding to the planes of self-images of the classical Talbot effect, a lattice of vortex ring beams appears, the topological charge of which corresponds to the charge of the incident beam. The approaches to the analytical solution of this problem are studied.

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## Synchrotron radiation metrological station "Cosmos" at the VEPP-4 storage ring: current status and prospects.

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Metrological synchrotron radiation station "Cosmos" is functioning in the Siberian Centre of Synchrotron Radiation. The purpose of the station is the development and implementation of metrology methods in the soft X-ray (80 – 5000 eV) and vacuum ultraviolet (4 – 100 eV) ranges. In order to provide such a wide spectral range the station has two monochromators, which use multilayer mirrors, diffraction gratings and Si (111) crystals. Also at the station is carry out works on the measurement of the spectral transmission of soft X-ray filters, the definition of the spectral characteristics of the TER mirrors, multilayer mirrors and crystals at the mentioned spectral range. The report presents the main parameters of the station, a description of its layout, of its monochromatic system and its reference detectors. The specification of basic metrological procedures used at the station for calibration of the various detectors is presented too.

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## Polarization dependent X-ray spectroscopy: recent advances

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Development of the third generation synchrotron radiation sources has boosted X-ray spectroscopy, as illustrated by the discovery of a variety of new experimental techniques associated with the exploitation of the polarisation properties of x-rays. The detection of X-ray magnetic linear and circular dichroism in ferro-, ferri- and paramagnetic systems, the discovery of X-ray natural circular dichroism in gyrotropic single crystals as well as the observation of non-reciprocal X-ray linear dichroism and X-ray magneto-chiral dichroism in magnetoelectric systems are particularly interesting. In combination with sum rules these spectroscopies appear as remarkable tools to study fundamental properties of matter via various order parameters, e.g., spin and orbital moments, electric dipole moment, toroidal moments etc. In this talk we report on advanced

instrumentation developments carried out over the past 25 years at the ESRF beamline ID12 which is dedicated to polarization dependent x-ray spectroscopy at photon energies above 2keV. Emphasis is laid on control of the polarization state of the x-ray beam and on the detection systems. We highlight two recent science-driven instrumentation developments initiated by the ID12 team: the first one deals with measurements of x-ray magnetic circular dichroism under multiple extreme conditions (high magnetic field, low temperatures and high pressure); the second examples concerns x-ray detection of optical activity in non-centrosymmetric crystals.

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## X-ray radiography of microjets from a metal surface having constructional elements

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Flash radiography (bremsstrahlung and synchrotron radiation) was used to record micro-cumulation from the surface of flat lead, tin and cooper samples having constructional elements, i.e. a steel tube, joints of members and cavities of the shock-loaded surface. Results of this flash radiography are compared in this report.

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## Near-surface structural changings in paratellurite crystals under external electric field

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This work describes the results of the near-surface structural changes caused by the migration of oxygen vacancies in paratellurite ( $\alpha$ -TeO<sub>2</sub>) single crystals obtained by the in situ X-ray diffraction technique under a constant electric field. This process has a reversible character [1] and its dynamics (duration is about tens of minutes) corresponds with the conductivity kinetics.

When the electric field is applied along the [100] and [110] directions, two types of processes occurs simultaneously which were recorded through measurement of the diffraction curve evolution [2]. The first one is caused by the piezoelectric lattice deformation due to a sharp increase in the strength of the field near both surfaces. At the same time, the change in the lattice parameter near the anode (the surface of a crystal with a positive external charge) was revealed, that is caused by a local rearrangement of the crystal structure due to the counter migration of oxygen ions and oxygen vacancies in the external electric field and their accumulation at the dielectric-metal interface. The velocity of the process depends on the crystallographic direction, however the application of electric field in the non-piezoelectric direction [001] does not affect on the rocking curve shape.

This work was partially supported by the Russian Foundation for Basic Research (RFBR grant N<sup>o</sup> 18-32-00410 mol\_a, 16-32-60045 mol\_a\_dk) and by the Federal Agency of Scientific Organizations (Agreement N<sup>o</sup> 007-/3363/26) in part of crystal growing and sample preparation.

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## Influence of heating rate on oxidation of the powder ASD-4 modified by V2O5

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Aluminum powders, due to their high specific heat of combustion, are widely used in explosive, the production of high-energy combustible mixtures and solid fuels for rocket engines. To solve the problem of high protective properties of oxide film on the surface of metal particles, in recent years much attention is paid to the development of methods for the activation of Al powders and the optimization of their combustion processes. In this work shows the results of studying the oxidation characteristics of the initial and modified Al powder with vanadium pentoxide at a heating rate of 30 and 100 K / min, because exothermic heterogeneous reactions are characterized by a strong dependence on the thermal regime. The vanadium content is about 0.8 wt. %. Carried out a complex study of the oxidation of a modified aluminum powder in air and in a gas mixture flow of 20%O<sub>2</sub> + 80%N<sub>2</sub> up to 1473 K by methods TG, DSC, X-ray diffraction using a synchrotron radiation source. It was found that an increase in the heating rate of the modified ASD-4 powder leads to an active growth of metastable phases of aluminum oxide (  $\gamma$ -Al<sub>2</sub>O<sub>3</sub> and  $\delta$ '-Al<sub>2</sub>O<sub>3</sub>) passing phase  $\gamma$ -Al<sub>2</sub>O<sub>3</sub>. The heating rate of modified aluminum affects the phase composition of the oxidation products, where the basis of the oxidation mechanism is the effect of V<sub>2</sub>O<sub>5</sub> on the increase in the rate of delivery of the oxidant (oxygen) to the metal surface.

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## XAFS- study electronic and structure peculiarities of high-k dielectric non-stoichiometric sub-oxides for memristors

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Neural and neuromorphic networks that mimic brain functions are used in various fields of science and technology. To create more efficient neural networks, it is required to expand the element base with new instruments with a memory effect. This is the property of a memristor, a passive device whose resistance can be changed by passing a certain current pulse through it. The memristor is a metal-dielectric-metal structure with a dielectric with a high dielectric constant (high-k dielectric). In this paper, using the XAFS spectroscopy methods, the microstructural properties



of a number of possible high-k dielectric elements for memristor structures have been studied. These are oxides HfOx, ZrOx, TaOx with different values of “x”. It has been established that the samples ZrOx and HfOx studied are a mixture of the phases of ZrO<sub>2</sub> and HfO<sub>2</sub> oxides and metallic zirconium and hafnium, respectively. The microstructural characteristics for the clusters of both phases in the samples are very close to the characteristics of the pure phases. According to the results of our experiments, the observed samples did not show a noticeable decrease in the coordination numbers for zirconium and hafnium atoms in comparison with the coordination numbers of the massive phases, which allows us to state that the dimensions of single-phase clusters exceed the size of ~ 10 nm. In the XANES spectra for samples of non-stoichiometric TaOx oxides, a long-wavelength shift of the absorption edge relative to the reference oxide is observed, i.e. a decrease in the positive charge on Ta atoms. According to the data of EXAFS, a noticeable increase in the disorder of the structure is observed for the TaOx samples under study as compared to the reference TaO<sub>2</sub> oxide. The observed changes in the interatomic distances and coordination numbers, as well as an increase in the disorder of amorphous films with a change in the parameter x (its decrease), is apparently due to the presence in the samples of several oxides of different stoichiometry (in the absence of a metallic phase). This results are in accordance with known literature data.

This work was carried out with the financial support of the RSF No 16-19-00002

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## Microstructure of multilayer heterosystems containing Ge quantum dots with Mn in Si matrix by XAFS- spectroscopy

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Electric-field manipulation of ferromagnetism has the potential for developing a new generation of electric devices to resolve the power consumption and variability issues in today’s microelectronics industry. To date, in the scientific literature are few examples of the successful synthesis of self-assembled dilute magnetic Mn quantum dots with ferromagnetic order above room temperature. For the first time, a comparative analysis of the microstructure of samples of dilute Mn solutions in quantum dots of Ge in a silicon matrix, obtained as a result of ion Mn<sup>+</sup> irradiation was carried out within the framework of XAFS spectroscopy methods: EXAFS (extended X-ray absorption fine structure) and XANES (X-ray absorption near-edge structure). From the analysis of the GeK EXAFS spectra, a significant presence of Mn in the first sphere of the Ge atoms and coordination numbers and interatomic distances in Ge environment are determined. Thus, for such systems, the direct contacts of germanium and manganese atoms were first discovered and characterized. The features of the microstructure and elemental composition of multilayer Ge/Si systems containing QDs, obtained by the simultaneous molecular beam epitaxy (MBE) of Ge and Mn for various synthesis conditions, in particular, with different impurity content of Mn, are studied. From the analysis of the GeK EXAFS spectra, coordination numbers and interatomic distances were determined, intensive mixing of Ge, Si and Mn atoms was observed in all samples. The degree of diffusion of Ge/Si correlates with the temperature of growth of QD. Correlations of the coordination numbers of Si and Mn in the sphere of Ge surroundings with the amount of manganese in QDs, as well as with the temperature at which quantum dots were grown and with other synthesis conditions were established. The XAFS spectra were obtained on the BM20 channel of the ESRF in Grenoble, and on channel 8 of the Center for Nuclear Research of the Institute of Nuclear Physics of the SB RAS.

This work was carried out with the financial support of the Russian Foundation for Basic Research (No. 16-02-00175-a; No. 16-02-00397-a)

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## Development of hardware-methodological approaches for time-resolved control of synchrotron beam parameters by x-ray acousto-optics based on longwave ultrasound

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Development of the method for time-resolved controlling of x-ray beam parameters by several modes of longwave vibration (longitudinal or bending) will be represented. We suppose that both modes could be useful to provide timeresolved x-ray study. In case of longitudinal vibrations, diffraction conditions changes by modulation of lattice parameters with tension-compression regular deformations. In case of bending vibrations, rotation of reflecting planes of crystal was used.

Based on these researches at Shubnikov Institute of Crystallography of FSRC "Crystallography and Photonics" of RAS and NRC "Kurchatov institute" fundamentally new scientific instrument: the x-ray acoustic laboratory diffractometer as well as x-ray acoustic modules for synchrotron station has been developed and successfully tested. Experimentally achieved space deviation range, time- and angular resolutions are 100-1000 arcsec, 100 ms (theoretically 3  $\mu$ s) and 0.1 arcsec. respectively. It should be noted that mentioned characteristics depends on types of vibration, x-ray optical schemes, etc.

Some results and prospects of implementation of these approaches for studying dynamic of crystal lattice defects structure of the samples under external influences as well as possibility of controlling the wavelength (energy) or angular position of the x-ray beam on laboratory and synchrotron radiation sources will be shown.

Some promising intention of implementation of x-ray acoustic approaches based on combination of different types of ultrasonic vibrations on synchrotron sources, including the 4th generation, and at the XFEL, which could give a significant expansion of the experimental possibilities, will be discussed.

These researches was performed using the equipment of the Shared Research Center of FSRC "Crystallography and Photonics" of RAS and Unique scientific installation "Kurchatov Synchrotron Radiation Source" and partially was supported by the Russian Foundation for Basic Research (proj. № 16-29-14057 ofi\_m, 18-52-05024 Arm\_a) and the Council on Grants of the President of the Russian Federation -2451.2018.2.

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## Time range extension from nanoseconds to hundreds nanoseconds in NFEL pump-probe experiments

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Pump-probe experiment is widely used spectroscopic method. In case using different frequencies and different sources for pumping and probing there practically are no limitations on time duration of phenomena under investigation. But if using the same source for pumping and probing time range is limited by length of optical scheme from picoseconds to few nanoseconds. By using NovoFEL emission time range of pump-probe experiments can be extended up to hundreds nanoseconds in optical region from infrared to terahertz.

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## Polymethylmethacrylate double compression registration using synchrotron radiation

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Experiments with single and double shock compression of the materials provide background for the development of equation of state (EOS) of the material under high pressure. Double compression compared to single compression provides smaller growth of the internal energy, the states that occur due to this phenomenon are observed below Hugoniot curve, approaching to isotherm. The fact helps to understand the value of the experiments with double compression conducted in order to investigate into the states of matter and its EOS under high pressures. The work describes the setup and results of the experiments where two modes of shock-wave collision were observed. Synchrotron radiation was used to register processes of incident shock wave propagation and collision, as well as reflected shock wave formation. The experiments were conducted using accelerator of the Budker Institute of Nuclear Physics SB RAS.

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## Multilayer X-ray imaging optics in IPM RAS

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Owing to the high peak and integral reflection coefficients, multilayer interference mirrors (MIMs) have unique X-ray optical characteristics in the extreme ultraviolet (EUV) and X-ray wavelength range of 0.01-60 nm. On their basis, experimental a new generation nanolithographs for a wavelength of 13.5 nm, microscopes for the transparency windows of silicon (13 nm region), carbon (5-6 nm) and water (2.8-4 nm) have been manufactured. The greatest amount of information about the physical processes occurring on the Sun is obtained from investigations of the solar corona in the EUV and X-ray ranges. Recently, in connection with the advent of super-power femtosecond lasers, great interest arose in aperiodic MIMs, which allows transporting, focusing, and spectral analysis of atto- and even sub-atto- second pulses of electromagnetic radiation. The wide bandwidth of such MIMs allows one to control these beams without “blurring” the wave packet, or even to shorten it in time. The short wavelength and pulse duration allow to increase by 3-5 orders of magnitude the power density of the radiation in the focusing spot and to come close to reaching the “vacuum breakdown” values of 1028-1030 W/cm<sup>2</sup>. MIMs are widely used in synchrotron centers both in the EUV and in the hard x-ray range, up to 100 keV. By providing collimation and focusing of X-ray radiation, due to the large operating angles and the controlled spectral bandwidth of the MIMs, they outperform the traditional grazing incidence mirrors, both in terms of the aperture and in the diffraction distortions of the reflected wave fronts. The latter is especially important for synchrotrons of the third and fourth generation. On the basis of the MIM’s technology, recently a new X-ray optical element has appeared: a free-standing (without substrate) multilayer structure. It can be used as a polarizer, phase shifter, beam splitter and spectral filter with a given bandwidth. The use of this element on synchrotrons makes it possible to carry out interference experiments, to certify the polarization composition of the probe and secondary (scattered, reflected, X-ray luminescence) X-ray beams. To use the potential of the MIMs for imaging, focusing, collimating and transportation of the beams without distortion of the wave fronts in full, diffraction quality optics for the X-ray range is required. In comparison with traditional optics, its accuracy should be at least 2 orders of magnitude higher: subnanometric shape accuracy and angstrom roughness are required. Traditional methods of manufacturing and examination the mirrors do not meet these requirements. The report describes the main scientific and technological directions and methods developed in the Institute for Physics of Microstructures of the Russian Academy of Sciences for solving the problems noted above, and the main results obtained in recent times are given. Considerable attention will be paid to the works on the development of diffractive quality optics for the EUV and X-ray ranges. Research includes metrology of shape and roughness, ion-beam polishing and the substrates surface shape

correction techniques, deformation-free fixing substrates in frames, searching for new materials and deposition of “stress-free” MIMs, and other aspects.

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## Conceptual design of "MicroFocus" beamline of Siberian Circular Photon Source (SKIF)

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The recent development of low-emittance storage rings promises particular benefits for techniques that require tightly focused and brilliant beams of synchrotron radiation, such as XRF/XRD/XAS micro- and nanomapping, high-pressure research \*etc.\* The “MicroFocus” beamline was designed to use all benefits of low-emittance storage ring enhanced by modern refractive X-ray optics, and provide a ‘multi-tool’ infrastructure for investigation of geo-, bio-, and functional materials, samples related to forensic science and cultural heritage \*etc.\*

The beamline will use undulator radiation converted into a slightly convergent beam by diamond refractive optics installed in the front-end. An optic hutch will contain two diamond monochromators, reflecting 12.6 keV and 14.4 keV harmonics aside for use at (I) macromolecular crystallography and (II) nuclear scattering stations, respectively. The remaining part of the spectrum will be monochromatized by adjustable channel-cut Si monochromator with zero offset for use at (III) nanomapping and (IV) extreme conditions stations. Each monochromator will be followed by a transfocator (adjustable array of Be lenses) in order to provide an X-ray spot of necessary size on the sample. The following techniques are planned to be realized at the stations:  
**\*\*I. Macromolecular crystallography station:\*\*** Se-SAD protein crystallography, small-molecule crystallography  
**\*\*II. Nuclear scattering station:\*\*** <sup>57</sup>Fe nuclear inelastic/forward scattering and synchrotron Mössbauer source  
**\*\*III. Nanomapping station:\*\*** combined X-ray fluorescence (XRF), diffraction (XRD) and absorption (EXAFS/XANES) mapping with submicron spatial resolution  
**\*\*IV. Extreme conditions station:\*\*** XRF, XRD, EXAFS/XANES in a diamond anvil cell (up to 3 000 000 atm) including laser heating (up to 6 000 K) and cryostat options

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## Scanning XRF microanalysis of iron meteorite Sikhote-Alin

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The first results of the investigation of microparticles of the Sikhote-Alin iron meteorite are shown. Measurements was carrying out on experimental module X-ray Confocal Microscopy [1] situated on NRC “Kurchatov Institute”. The experimental parameters are excitation energy 20 keV, the size of the excitation radiation spot on the sample is 15 μm, the scanning step is 20 μm, the measurement time at the point is 10 sec. Two-dimensional maps of the distribution of rock-forming (Ti, Mn, Fe, Ni) and microelements (V, Cr, Cu, Zn, Ga, Ge, As, Rb, Sr, Y, Zr, Mo)

in the samples were obtained. This work was partially supported by Comprehensive program of fundamental scientific research of the SB RAS project N°0330-2018-0031.

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## Analytical microstratigraphy of annual layers in frozen bottom sediments of Lake Shira

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Samples of modern bottom sediments of Lake Shira (Khakassia) was obtained in the form of frozen bulk contained the upper 20cm of bottom sediments using special sampling equipment (ice-corer). This procedure guaranteed the receiving of the unmixed and undamaged upper sediment layers. The preparation of solid samples for measurements was carried out by a vacuum drying technique and impregnated with epoxy resin [1]. Measurements of the distribution of rock-forming and trace elements in cores of bottom sediments were carried out on XRF station at VEPP-3 and by method described in the article [1] at an excitation energy of 22 keV with a scanning step of 200  $\mu\text{m}$ . Investigations of individual annual layers with a width of 0.5 - 1.5 mm were carried out on experimental module X-ray Confocal Microscopy [2] situated on NRC “Kurchatov Institute”. The experimental parameters are excitation energy 20 keV, the size of the excitation radiation spot on the sample is 20  $\mu\text{m}$ , the scanning step is 20  $\mu\text{m}$ , the measurement time at the point is 10 sec. Each investigated annual layer was crossed by a profile of 25-75 points which made it possible to create a model for the accumulation of microelements during the annual cycle of sedimentation. This work was partially supported by Comprehensive program of fundamental scientific research of the SB RAS project N°0330-2018-0021.

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2. Sorokoletov D.S., Rakshun Y.V., Darin F.A. // The spread function of a polycapillary lens and a confocal x-ray microscope in retuning its confocal volume// Optoelectronics, Instrumentation and Data Processing. 2015. . 51. N° 3. . 293-301.

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## Application of short-wave diffraction of synchrotron radiation for X-ray diffraction study of the structural features and phase transitions during hydrogenation of titanium Ti-6Al-4V alloy additively produced by the method of electron beam melting

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Additive manufacturing of titanium alloys is an advance and intensively developing field of modern material science due to the wide application of titanium alloys in various industries. One of the important and interesting aspects of the study of additive materials is the establishment of regularities in the interaction of hydrogen with titanium alloys produced by the method of electron beam melting (EBM). On the one hand, this is due to the fact that EBM is considered as the main method of manufacturing titanium alloys for aircraft building, engine building and rocket construction; on the other hand, hydrogen treatment of additively produced titanium alloys, under certain conditions, can improve the physical and mechanical properties of products. In this connection, it is important to study the structural features and phase transitions during hydrogenation of titanium alloys produced by the method of electron beam melting. In the present work, hydrogen sorption by titanium Ti-6Al-4V alloy produced by the EBM method was investigated as well as structural-phase transformations in the alloy during hydrogenation was studied with the help of in-situ X-ray diffraction. The joint application of the X-ray diffraction method and hydrogenation from the gas-atmosphere made it possible in one experiment to determine the relationship between the phase composition of the alloy and its sorption properties, and thereby broaden the understanding of the mechanisms of interaction of hydrogen with titanium alloys produced by additive technology. Previously, this approach was used only to study the interaction of hydrogen with titanium and zirconium alloys manufactured by traditional methods [1-2].

This research was funded by Russian Science Foundation, research project No. 17-79-20100. The authors thank Tomsk Polytechnic University Competitiveness Enhancement Program.

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## Investigation of the structure and composition of annual layers in varve sediments of glacial lakes Donguz-Orun (Caucasus) and Kucherlinskoye (Altai)

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Models of the annual cycle of sedimentation in glacial lakes are necessary for constructing paleoclimatic reconstructions with high time resolution. Samples of bottom sediments of two glacial lakes - Donguz-Orun (Caucasus) and Kucherlinskoye (Altai) - were studied for obtaining the

necessary information. The bottom sediments cores were received during field work using special samplers (box-corer). The preparation of solid samples for measurements was carried out by a vacuum drying technique and impregnated with epoxy resin [1]. Measurement of the elemental composition was carrying out on experimental module X-ray Confocal Microscopy [2] situated on NRC “Kurchatov Institute”. The experimental parameters are excitation energy 20 keV, the size of the excitation radiation spot on the sample is 20  $\mu\text{m}$ , the scanning step is 20  $\mu\text{m}$ , the measurement time at the point is 10 sec. Each investigated annual layer was crossed by a profile of 25-75 points. This investigation result in creation a model for the accumulation of microelements during the annual cycle of sedimentation. Additional information on the structure of annual layers was obtained using the X-ray microtomography method on the experimental station “RT-MT” at KSRS [3]. This work was partially supported by RFBR project N<sup>o</sup> 18-55-53016 and N<sup>o</sup> 17-35-50115.

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## The Influence of the Synchrotron Radiation on the Exothermal Effect of Aluminum Micron Powder Oxidation

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The influence of the synchrotron radiation on the exothermal effect of aluminum micron powder oxidation was investigated. It was founded, that exothermal effect of oxidation increased on 8.0 % (107.5 kJ/mol) after irradiation. It is the result of particles core charging due to ionization treatment of the synchrotron radiation.

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## X-band (9 GHz) Electron Paramagnetic Resonance station at the NovoFEL facility: sensing the spin dynamics induced by high-power THz pulses

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Large-scale facilities such as neutron research reactors, synchrotron light sources and free electron lasers offer natural science experimentalists the opportunity to perform unique experiments which determine the cutting edge of experimental data accessible. Novosibirsk Free Electron Laser (NovoFEL) was originally developed to fit the requirements of physicists, chemists and biologists [1] and featured ultrawide range of radiation wavelength (THz, far- and mid-IR ranges) and very high average power (up to 400 W at 70 cm<sup>-1</sup>). International Tomography Center SB RAS is constructing two user stations at NovoFEL aiming to utilize the unique radiation applied to the field of molecular magnetism: SQUID magnetometer station and continuous-wave (CW) and Time-Resolved (TR) X-band (9 GHz) Electron Paramagnetic Resonance (EPR) station.

In this work we present the current status of CW and TR EPR station at NovoFEL. The detailed layout of the experimental user station is shown and discussed. While the CW and TR EPR X-band (9 GHz) spectrometer used is almost the same as in many EPR laboratories, it was built in the NovoFEL beamline and allows performing the unique EPR experiments with simultaneous irradiation of the sample by common UV-vis and exceptional NovoFEL light. For this purpose, multimodal THz waveguide allowing to feed NovoFEL radiation directly into the EPR resonator is used. Laser radiation of NovoFEL is passed through the collimator based on two off-axis parabolic mirrors which compress the beam diameter by a factor of 15 adjusting it to the aperture of THz waveguide used in EPR spectrometer. Mechanical optical chopper is used to decrease the average power by a factor of 100 to the safety level while keeping the maximum accessible peak power. Passed through the collimator and optical chopper, the NovoFEL radiation can be readily directed to the sample into EPR resonator. Different detection schemes of experiments conditioned by the initial time profile of NovoFEL radiation, optical chopper modulation and the capability (CW and TR) of EPR spectrometer used are discussed. Special attention is drawn to the heat effect and its practical use for the temperature-modulated detection of very broad EPR spectra [2].

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Acknowledgements: This work has been supported by the Russian Science Foundation (no. 17-13-01412).

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## Magnetic measurement 22-pole 7 Tesla wiggler for DELTA synchrotron radiation source

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The Dortmund Electron Accelerator (DELTA, Germany) operated as 1.5 GeV synchrotron radiation source requires a superconducting wiggler as an insertion device for three x-ray beamlines with photon energies up to more than 30 keV. The article describes all the stages of magnetic measurement procedure. During preliminary tests in a submerged cryostat the magnetic system was trained and the magnetic field integrals were evaluated. Based on the measurement results, the final correction of the geometry of the wiggler was made by adjustment of the distances between the inner and the end poles. The whole cycle of magnetic measurements was carried out in the own cryostat and the table of working currents was prepared.

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## Employing a highly-sensitive synchrotron XRF technique for obtaining multielement geochemical records: the first



## continuous ~10-kyr record from a remote Caucasus Mountains lake

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Lake sediments offer valuable highly-resolved continuous records of past climatic and environmental changes. Temporal coverage of these records typically constitutes centuries to millennia. Over past decades many indicators have been explored in the sediments and used as proxies for past climatic and environmental conditions. One powerful technique for paleoreconstructions, that has been perfected at the Siberian Synchrotron and Terahertz Radiation Centre, SSTRC (by A. V. Darin, I. V. Rakshun, and others) is based on employing synchrotron radiation. The technique (described in detail previously by A. V. Darin \*et al.\*) supplies essentially continuous (resolution up to 20  $\mu\text{m}$ ) profiles of over 20 elements along the vertical axis of the sediment core. Employing a synchrotron beam allows to significantly extend the possibilities of the ordinary XRF analysis. In this work, we present the first continuous geochemical record for the Holocene for the Caucasus region from a remote lake in the Caucasus Mountains (Lake Khuko, 43°56'N 39°48'W). Specially prepared samples from the ~2-meter sediment core were scanned at the X-ray Fluorescent Elemental Analysis station at the SSTRC at the 23-keV energy. The increment step during the overview scanning was set to 250  $\mu\text{m}$ . The full interpretation of the new record will be possible only when the geochemistry data will be combined with other indicators (pollen, organic matter content, etc.). Here we present the data on selected elements (Br, Sr, etc.) displaying the most distinctive patterns, signifying the past changes in climate and environment.

This work was supported by the Russian Foundation for Basic Research (Project leader: O. N. Solomina, grant No. 17-05-01170).

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## In situ X-ray diffraction study of Pr(2-x)CaxNiO4 at low oxygen pressure

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This work was aimed at structural studies of mixed oxygen–electron conducting Pr(2-x)CaxNiO4(x=0.1-0.6) oxides synthesized by the coprecipitation method with the final sintering temperature of 1150-1250C. Structural changes of samples during heating in the excess/lack of oxygen were characterized by X-ray diffraction (XRD) method using synchrotron radiation. The heating rate and temperature range were 10C/min and from room temperature up to 900C, respectively. We also performed the thermogravimetric experiments in similar conditions to connect the phase transformations with the sample oxygen loss. The synchrotron XRD studies were performed using the infrastructure of the Shared-Use Center “Siberian Synchrotron and Terahertz Radiation Center(SSTRC)” based on VEPP-3 of BINP SB RAS.

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## The nickel hydrogenation in situ investigation by X-ray diffraction using synchrotron radiation

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The process of nickel hydrogenation was studied in situ by X-ray diffraction using synchrotron radiation. It is generally believed that the occurrence of defects in the metal during hydrogenation is due to the fact that hydrogen, meeting pores, cracks and other discontinuities, gathers in them, recombining into a molecular form. As a result of a constant influx of hydrogen, a high pressure is created in the cavities, leading to deformation and local destruction of the lattice of the crystal. Almost simultaneously with the beginning of the change in the X-ray diffraction pattern of nickel, the formation of the metastable phase of nickel hydride begins - reflexes appear from the planes (111), (200), (220), (311), (222), (400). Reflexes of nickel hydride are shifted towards smaller diffraction angles with respect to nickel reflexes, since the lattice parameter of the new phase is 6% larger. At the initial stage of the formation of the new phase, the reflexes of nickel hydride are strongly broadened, which probably indicates the small size of the nuclei of the new phase in the nickel matrix. In the process of hydrogenation, nickel hydride nuclei increase in size, and approach  $L = 200 \text{ \AA}$ . At the same time, it was found that at the initial stages of the formation of a new phase, the lattice parameter of the nickel hydride differs from the lattice parameter of the parent metal by only 3%, and not by 6%, as measured after the cathodic hydrogenation process was completed. Analyzing the data on the change in the lattice parameter of nickel and the dynamics of the formation of nickel hydride, it can be concluded that the lattice parameter changes faster than a new phase is formed. This indicates that hydrogen first penetrates into the interstices of the crystal lattice, creating an excessive concentration there (it is known that the concentration of hydrogen in the interstice of the matrix of the metal is linearly related to the change in the lattice parameter), and only then the formation of nickel hydride begins. Thus, during the first cycle of hydrogenation in 40 minutes the concentration of hydrogen in the matrix reaches 90% of its limit value, while nickel hydride forms only 69% of its limit value.

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## Insertion devices for national sources of synchrotron radiation with extremely low emittance

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It is planned to build new national sources of synchrotron radiation with extremely low emittance in Russia in the nearest future. The program is also known as SSRS-4 (specialized synchrotron radiation source of the 4-th generation). This paper describes modern and promising methods of synchrotron radiation using in relation to insertion devices for synchrotron radiation generation.

It was analysed users' needs and wishes working on over 100 user stations all over the world: Siberian Synchrotron and Terahertz Radiation Centre (Russia), ESRF (France), SPring-8 (Japan), PETRA III (Germany), NSRRC (Taiwan) etc. Classification work was performed, and insertion devices were grouped in following way: multipole wigglers with extremely high field level (7...7.5 T) and long period (140...200 mm); multipole wigglers with medium field level (3.5...4.2 T) and short period (48...60 mm); multipole wigglers with short period (30...34 mm) and low field level (2...2.2 T); superconducting undulators; permanent magnet undulators; permanent magnet helical

undulators; APPLE-II undulators with variable polarization. As an example, the paper describes 6 proposed user station for Siberian Circular Photon Source ("SKIF"), which is planned to launch in 2023.

The work was supported by The Ministry of Education and Science of The Russian Federation project RFMEFI61617X0087.

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## Operational Status of SOLARIS Storage Ring

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Solaris – the first Polish synchrotron light source built in Krakow is in operation since mid-2015. The storage ring is a twin brother of MAXIV 1.5 GeV storage ring installed in Lund, Sweden. The lattice is composed of double bend achromat (DBA) cells with zero-dispersion straights and exhibits 12-fold symmetry. With the circumference of 96 m and energy of 1.5 GeV, its strong focusing and ultra-compact lattice enables to reach the natural emittance of 5.98 nm-rad. It also provides twelve 3.5 m long straight sections and ten of them can host the insertion devices (IDs). The injection septum and diagnostics is located in the 1st straight section (SS) whereas the RF cavities occupy the 12th SS. In the 3rd SS the injection dipole kicker is installed, therefore in this location only short (up to maximum 1.6 m) ID can be installed. Performance of position monitoring devices has proven essential for the successful optimization of beam parameters such as: closed orbit, tune, chromaticity, and dispersion. Now, the effort is focused on fine-tuning the machine by implementing the linear optics from orbit correction (LOCO) and reducing the disparity between model and measured results revealed by the phase advance analysis and dispersion measurement. Moreover, during daily operation the main task is to maintain long-term stability of the circulating electron beam allowing for beamlines commissioning. Within this presentation the current status of the Solaris storage ring and the commissioning results will be reported.

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## Premiere study of iron Sikhote-Alin meteorite by XAFS

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This work is devoted to present of premiere results of the study of microparticles of the Sikhote-Alin iron meteorite by XAFS spectroscopy method. All XAFS (Ni-, Fe- K edges) spectra, using  $\mu$ - and standard modes, of the microparticles and reference samples were recorded both on experimental module X-ray Confocal Microscopy situated on NRC "Kurchatov Institute" (Moscow) and at XRF-, EXAFS- stations of Siberian Synchrotron Terahertz Radiation Center (SSTRC, Novosibirsk). Some changes of the phase compositions and local structure arrangements

of the studied microparticle samples were characterized in detail. The interatomic distances and corresponded coordination numbers were revealed. All possible structural models were discussed. Additionally, morphology and composition of the microparticlesamples were studied by the SEM, EDX and XRF methods. This work was conducted within the framework of SB RAS program II.1 (project 0303-2018-0010, -17-117112840087-0)

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## Bottom sediments of the Lake KarakyoI reveal a 2000-year climate variability in the Western Caucasus

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Two sediment cores were retrieved from the Lake KarakyoI (Teberda, Western Caucasus) during the field expeditions of 2010 and 2014. The cores (depths 20-85 cm and 0-60 cm, respectively) were combined to create a joint master-chronology using the results of high-resolution scanning SR XRF analysis carried out within the facilities of BINP SB RAS. In addition, traditional sedimentary analyses (loss-on-ignition, grain size, magnetic susceptibility) were performed at an increment of 1-2 cm. The chronology was constrained by the eight <sup>14</sup>C AMS dates that ensured a reliable age-depth relation for the last 2000 years. The results witness several distinctive warm and cold periods in the Caucasus during the last two millennia and help to constrain regional timeframes of such events as the Medieval Warm Period and the Little Ice age. The research funded by the RFBR grant # 17-35-50134

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## The project of end-station for high-resolution soft X-ray emission spectroscopy on VEPP-4M storage ring

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Modern X-ray emission spectroscopy (XES) is a powerful and effective method for studying the elemental composition, electronic and atomic structure of various substances and materials. The XES data allows comparing features of the electronic and atomic structure of the studied substances with their physicochemical properties to predict on this basis properties of new substances and materials. Despite the highly informative XES method, in our country specialized end-stations for studying electron and spatial structure with soft X-ray emission spectra using synchrotron source is lacking. In this connection, the Nikolaev Institute of Inorganic Chemistry of SB RAS and the Budker Institute of Nuclear Physics of SB RAS are working to create a high-resolution X-ray emission spectroscopy end-station for the study of the elemental composition and electronic structure of light-element compounds in "Siberian Synchrotron and Terahertz Radiation Center (SSTRC)" based on VEPP-4M. As expected, the station will use the "Cosmos" beamline based on the VEPP-4M storage of Budker Institute of Nuclear Physics of SB RAS and the ultra-soft X-ray spectrometer "Stearat", developed at the Institute of Inorganic Chemistry of SB RAS. The report presents the main parameters of the planned station and the current status of the work on its creation.

The reported study was funded by RFBR according to the research project N<sup>o</sup> 18-03-01061 A. The work relating to the development of the station is carried out using the infrastructure of the Shared-Use Center “Siberian Synchrotron and Terahertz Radiation Center (SSTRC)” based on VEPP-4M of BINP SB RAS.

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## X-ray refractive lenses made by ultra-deep LED lithography

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X-ray refractive objective lens and condenser lens were developed for hard X ray transmission microscopy with synchrotron beam at photon energies 15-35 keV. The lenses are rows with large number of separate bi-concave parabolic elements with periodic spaces in order to cross perpendicularly 2 linear rows forming point focusing. The maximal raw length is 10 cm. The material of the X-ray refractive lenses is lithographic SU-8 polymer. It is a multi-element composition based on the monomer of diglycidyl ether of bisphenol-A novolack with small amounts of elements of a photo-acid generator, for example: C : O : H : Sb : F : S = 72.3 : 18.2 : 6.9 : 0.9 : 1.2 : 0.6. At 25 keV photon energy, the real and imaginary parts of the refractive index decrement of the SU-8 polymer are equal to 4.32E-7 and 2.61E-10, respectively. We control the current element compositions of the material by means of X-ray fluorescence analysis using INCA X-act system with the HITACHI S 3400N tip II E-beam microscope. The effective apertures are up to 1 mm for the designed X-ray condensers and they are up to 100 microns for the X-ray objectives. The focal distances of the objective lenses are within 30-60 mm and 1-3 m for the condenser lenses. The calculated focal spot (full width at half maximum) of the X-ray objective lenses with ideal parabolic profiles is less than 50 nm. In order to achieve the ideal parabolicity the technological design of the photomask lens elements included size deviations of SU-8 microstructures during photo- and X-ray lithography processes. Photolithography enables to produce X-ray mask for next step of X-ray lithography. The possibility of fabrication of planar X-ray refractive lenses focusing in two directions was demonstrated in previous works with the use of X-ray lithography [1]. In this work, we studied ultra-deep photolithography with the use of 400 nm light-emitting diode (LED) as a point light source, to make linear X-ray lenses on planar surfaces and on butt ends of CuZn substrates employing one lithography process. The exposure area of the compact photolithography setup was within 8-15 cm in diameter. SU-8 resist layer is photoactive for the LED wavelength showing the specific absorbance of 1.7 l/cm [2]. The material transparency results to ratio closed to unity for doses of the absorbed LED radiation at the bottom and at the top of the SU-8 layers of 1 mm thicknesses. The photolithographic dose ratios are comparable with X-ray lithography dose conditions and provide small diffraction distortions with high aspect ratios in the resist layer depth. In the paper, we describe the fabrication process and properties of the X-ray refractive linear lenses with vertical sidewalls of their elements made by the ultra-deep LED lithography as well as the crossed X-ray refractive lenses, which satisfy the X-ray microscope application purpose.

[1] E. Reznikova, T. Weitkamp, V. Nazmov, A. Last, M. Simon, V. Saile. Investigation of phase contrast hard X-ray microscopy using planar sets of refractive crossed linear parabolic lenses made from SU-8 polymer. *Phys. Stat. Sol. 204 (a)* 2811–2816 (2007). [2] E. Reznikova, J. Mohr, H. Hein. Deep photolithography characterization of SU-8 resist layers. *Microsys. Technol.* 11 (4-5) 282-291 (2005)

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## Development of method for creation a LIGA-raster for correcting the spatial distribution of radiation from flash X-ray generator

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To explore the inner structure of fast moving objects, an X-ray flash generator in energy range from 30 to 200 keV is used. Since the X-ray radiation is scattered by the object, X-ray grids are used to collimate the beam of radiation near the X-ray tube and dampen the scattered radiation before detector. The development of microcollimators using LIGA based on synchrotron X-rays was performed. Using anti-scatter, X-ray golden grids with a pitch size of about 40 microns will improve the signal-to-noise ratio, increase the spatial resolution, and reduce the undesired scattered radiation. LIGA technology, which combines deep X-ray lithography at VEPP-3 storage ring and electroforming, was used for manufacturing X-ray anti-scatter grids. The technique of fabrication of intermediate X-ray masks based on titanium membrane and working masks on glassy carbon membrane is described. The influence of LIGA rasters on the radiation spatial distribution for a pulsed X-ray apparatus experimentally researched. The work was done using the infrastructure of the Shared-Use Center “Siberian Synchrotron and Terahertz Radiation Center (SSTRC)” based on VEPP-3 of BINP SB RAS (Novosibirsk, Russian Federation). The anti-scattering rasters were created using the infrastructure of the Shared-Use Center “Siberian Synchrotron and Terahertz Radiation Center (SSTRC)” based on VEPP-3/VEPP-4M of BINP SB RAS The work was supported by the Russian Science Foundation, project 14-50-00080.

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## The project of new synchrotron radiation technological station at VEPP-4M

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Synchrotron radiation (SR) is a successfully used tool of analytical techniques for studying the composition and structure of matter. Methods of X-ray fluorescence, diffraction analysis have been successfully used at the shared-used center (SUC) “Siberian Synchrotron and Terahertz Radiation Center (SSTRC)”. Prospects for the development of research infrastructure with the help of SI, the creation of a new SUC “Siberian circular photons facility” ("SKIF") necessitates the expansion of the training program for young researchers working with synchrotron radiation. To solve this problem, in order to expand the laboratory practical work on the beamline #1 of the SR source VEPP-4M storage ring, a synchrotron radiation technological station is create. The mission of new station is a practical training of students on the basics of working with synchrotron radiation. The modular concept of the station makes it possible to implement all the main experimental methods of SR researches. The station can also be used to test new experimental equipment for SR (detectors, positioning monitoring systems, etc.). The work was done using the infrastructure of the Shared-Use Center “Siberian Synchrotron and Terahertz Radiation Center (SSTRC)” based on VEPP-3 of BINP SB RAS (Novosibirsk, Russian Federation).

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## Application of X-ray lithography for fabrication microstructures with 3D-continuous-relief.

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In the classical scheme of X-ray lithography, the planar pattern is transferred from the X-ray mask to the resist layer. The depth of the resulted structures is determined by the irradiation spectrum and absorbed dose and mode of development. The vertical smooth walls along the entire depth of the structure is advantage of X-ray lithography. On the other hand, in some tasks of manufacturing micromechanical systems or micro-optical systems (MEMS or MOMS), the formation of microstructures with a smooth continuous profile of a given shape is required. X-ray lithography can be used to solve this problem. This can be achieved using gray-scale X-ray mask, where the thickness of the X-ray opaque coating changes along the surface of the template. In this case, during the exposition time, different sections of the resist will receive different exposure doses. And with simultaneous development, the depth of structures will correspond to the distribution of the dose across the surface of the resist. However, making gray-scale X-ray masks is a hard technological challenge. An alternative to making gray-scale X-ray patterns is the use of a specific method of dynamic X-ray lithography. This is the process in which the resist moves relative to the X-ray pattern during exposition. The resulting exposure dose is determined by the shape of the transparent areas on the mask, taking into account the characteristic curve for the resist used. The choice of X-ray pattern topology for obtaining a given curvature of microstructures in PMMA resist is considered in the article. Samples of the microstructures obtained are shown. The work was done using the infrastructure of the Shared-Use Center "Siberian Synchrotron and Terahertz Radiation Center (SSTRC)" based on VEPP-3 of BINP SB RAS (Novosibirsk, Russian Federation).

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## The electronic structure of binuclear transition metal complexes with disulfide bridged ligands studied by X-ray spectroscopy

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Transition metal chalcogenides (TMC) are interesting and practically important classes of inorganic substances. TMC compounds are of great interest due to their chemical, electrophysical, magnetic and optical properties. From a fundamental point of view, TMC are convenient model systems for investigating new physical phenomena and the physicochemical properties inherent in metal cluster compounds and systems with reduced dimensionality.

An important feature of the trichalcogenides (MX<sub>3</sub>), as well as chain structures [MnX<sub>4</sub>(n-1)] of IV-VII group transition metal thiocomplexes is the dichalcogenide group X<sub>2</sub> (where X = S, Se, Te). Discrete binuclear niobium chalcogenide complexes are convenient model structures for studying the nature of the electronic interaction between the dichalcogenide group (X<sub>2</sub>) and transition metal atoms in the case of 1D/2D TMC materials. A typical structural fragment of this compounds is the binuclear cluster {Nb<sub>2</sub>(μ-S<sub>2</sub>)<sub>2</sub>}<sup>4+</sup>, containing the disulfide group (S<sub>2</sub>) as bridging ligand. Detailed XES, XAS, XPS and quantum chemical study of the electronic structure of binuclear complexes [Nb<sub>2</sub>S<sub>4</sub>(acac)<sub>4</sub>], K<sub>4</sub>[Nb<sub>2</sub>S<sub>4</sub>(ox)<sub>4</sub>], TBA<sub>4</sub>[Nb<sub>2</sub>S<sub>4</sub>(SCN)<sub>4</sub>], Nb<sub>2</sub>S<sub>4</sub>Br<sub>4</sub> was carried out. SKβ, NbLβ<sub>2,15</sub> X-ray emission spectra (XES) were obtained on the X-ray spectrometer "Stearat". NbM<sub>2,3</sub>, SL<sub>2,3</sub>, OK, CK and NK X-ray absorption spectra were obtained at the RGLB beamline at BESSY II. Nb3d, S2p, O1s, C1s, N1s X-ray photoelectron spectra were obtained on the spectrometer SPECS (Germany).

Based on the X-ray spectra and quantum chemical calculations, the following data were obtained: 1) HOMO and LUMO energy position and partial molecular orbital compositions in the complexes under study; 2) the electron density distribution in the studied complexes and the atom charges

in binuclear clusters  $\{\text{Nb}_2(\mu\text{-S}_2)_2\}^{4+}$ ; 3) the character of molecular orbitals (bonding, anti-bonding).

The reported study was funded by RFBR according to the research project N<sup>o</sup> 18-03-01061 A. The Siberian Branch of the Russian Academy of Sciences (SB RAS) Siberian Supercomputer Center is gratefully acknowledged for providing supercomputer facilities.

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## Study on secondary electron emission of a quarter wavelength coaxial cavity for triode type thermionic RF electron gun

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Free Electron Lasers (FEL) require generation of electron beam of high quality. Some infrared FEL facilities apply thermionic RF guns due to their advantage of compactness and cost effectiveness as compared to RF photo-cathode electron guns. Other remarkable advantages are the long lifetime and moderate vacuum condition requirements. On the other hand, thermionic RF guns are suffering from back-bombardment phenomena, which limit the bunch charge and macro-pulse duration. In order to overcome this disadvantage a novel configuration named the triode type thermionic RF electron gun, which has an additional small pre-bunching cavity around the cathode, was developed at the Institute of Advanced Energy (IAE), Kyoto University. The triode concept has already been verified by numerical simulation. However, the tests of the prototype cavity have revealed the appearance of the secondary electron emission, which makes the operation of the cavity as a pre-buncher impossible. The objective of this study is the investigation of the origin of secondary electron emission and discussion of possible solution methods.

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## Fabrication of silicon LIGA masks

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The design and method of manufacturing high-contrast LIGA masks for deep X-ray lithography in the spectral range  $0.5 \div 3 \text{ \AA}$  are described. Gold was used as the material of the masking layer. Creation of this layer on the surface of the silicon plate was carried out by electro forming through a resistive mask. The bearing membrane and the support ring of the X-mask were formed by removing the central part of the plate by ion-beam etching it from the back side. Testing the quality and contrast of the finished LIGA masks was done at the X-ray microscopy station VEPP-3.

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## Methods for counteracting of back-bombarding effect for thermionic RF guns

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Thermionic RF guns find application as high brightness electron sources for RF linacs or as pre-injectors for synchrotron facilities. The thermionic RF guns have advantage over photo-cathode RF guns in their compact structure and simplicity of operation. However, for the applications where long electron macropulse duration (several  $\mu\text{s}$ ) is required, such as oscillator type FELs, the thermionic RF guns have a big disadvantage due to the back-streaming electrons. Those electrons are accelerated in the negative phase of an RF period backwards to the cathode. The back-streaming electrons hit the cathode and increase its temperature during the macropulse. This causes a ramp in thermionic current and limits usable pulse width. The reduction of the impact of the back-streaming electrons would advance further development of accelerator technologies using the advantage of compact structure of thermionic RF guns. This work presents a short overview over concepts for reduction of the back-bombardment effect for thermionic RF guns and some examples of technical realization respectively.

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## The station XRFA-SR on storage ring VEPP-4M

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The synchrotron radiation station XRFA-SR on storage ring VEPP-4M is functioning in the Siberian Centre of Synchrotron Radiation. The purpose of the station is the development and implementation of XRFA-SR methods in the hard X-ray (40 - 100 keV) range. The article presents the main parameters of the station, a description of its layout, of its monochromatic system and detector. The obtained spectra of XRF at the station is presented.

The work was done using the infrastructure of the Shared-Use Center “Siberian Synchrotron and Terahertz Radiation Center (SSTRC)” based on VEPP-4M of BINP SB RAS (Novosibirsk, Russian Federation). This work was funded by the RFBR and Government of the Novosibirsk Region within the framework of the research project 17-45-540618.

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## Absolute calibration of the spectral sensitivity of detectors in the VUV range. easurement procedure.

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Metrology synchrotron radiation station “Cosmos” is functioning in the Siberian Centre of Synchrotron Radiation and uses radiation from the VEPP-4 storage ring. As part of the methodological support of the station’s operation, a methodology for measuring the spectral sensitivity of detectors in the VUV range was developed and validated. The calibration is carried out using the reference detector method using a certified silicon photodiode. The report contains a description of the main stages of the procedure, such as obtaining metrologically pure monochromatic radiation, statistical processing of the obtained data, etc.

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## Fabrication X-ray masks with multilayer bearing membranes

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The design and manufacturing method of x-ray masks, which have high-contrast in x-ray spectral range of shorter wavelengths ( $\lambda \approx 3\div 7 \text{ \AA}$ ), are described. These masks can be a tool to form a resistive mask with a thickness of  $\sim 20\div 150$  microns and also can be used as intermediate mask for the fabrication of LIGA-masks for deep X-ray lithography. The manufacturing method is based on silicon planar technology. Two types of masks were made with mainly aluminum membrane. The method ensures the manufacture of x-ray masks with multilayer (titanium, aluminum and silicon layers, the thickness of which can vary in a fairly wide range) bearing membranes.

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## The project of the beam-line "Structure investigation" at synchrotron radiation source SKIF in Novosibirsk

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A new beamline "Structure investigation" will be put into operation in the test mode at the SKIF synchrotron radiation source in 2024. The beamline will have stations: 1) "Precision diffraction / anomalous scattering"; 2) "In situ diffraction"; 3) "Single crystal diffraction"; 4) «USAXS / reflectometry». The stations will supervise accordingly: 1) Institute of Catalysis of the SB RAS; 2) Institute of Solid State Chemistry and Mechanochemistry of the SB RAS; 3) Institute of Inorganic Chemistry of SB RAS; 4) Shubnikov Institute of Crystallography RAS. The beamline will use the radiation from a 300-pole superconducting undulator with a 1.2 T field. At a photon energy of 7 keV, the brightness will be  $4 \cdot 10^{21}$  phot / (sec mm<sup>2</sup> mrad<sup>2</sup> 0.1% BW). The energy range will be from 5 to 50 keV. Required channel element will be beam monitor. The monitor will determine both the intensity of the beam and its position with an accuracy of 1 micron. The read out frequency of monitor will be 100 MHz, which will allow to determine the position of the radiation from each bunch of electrons. A dual monochromator with a fixed output can provide monochromatization from  $10^{-2}$  to  $10^{-4}$ . If necessary, a monochromator can skip a white beam. Focusing mirrors will be used, which will ensure focusing and cutting of high harmonics if necessary. To work in a high energy area, refractive optics will be used. Both focusing and defocusing lens will be used. A fast chopper will be used, for investigating of fast

processes, with exposure from a single bunch with continuance near 73 ps. The main detector at the new station will be the coordinate-sensitive detectors DIMEX [1], whose use at the VEPP-3 source and showed great potential possibilities associated with the use of synchrotron radiation for the study of fast processes [2]. Detectors OD-3M [3] and PILATUS will also be used.

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## Synchrotron radiation investigation of bismuth potassium citrate water solutions with linear and branched polysaccharide

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The study of water solutions of linear (dextran) and branched (arabinogalactan) polysaccharides with Denol substance (Bismuth potassium citrate, ISSCM SB RAS) using SAXS, nuclear magnetic resonance (NMR), transmission electron microscopy (TEM) and gel permeation chromatography (GPC) were performed. It was shown the difference in the solubility of polysaccharides in mixtures with Denol. GPC were confirmed the existence of large Denol molecules in aqueous solutions (result of previous investigations of Denol). NMR investigation (<sup>1</sup>H and <sup>13</sup>C), TEM and SAXS confirm a presence of interaction between Denol and linear polysaccharide. The authors thanks Prof. Yu.I. Yukhin and Dr. E.S. Naydenko for providing of a Denol samples. This work were supported of Russian Foundation for Basic Research (grant 15-29-01297 ofi\_m).

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## Current status of EXAFS station of SSTRC. Possibilities of XAFS method to investigate challenging functional nano-materials. Reality and perspectives in future.

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At present time XAFS method, using of SR sources, in a various methodical modes and options is a well-known powerful tool to investigate the state of the elements and the local structure of the diverse nanosized systems having different kinds of aggregation: high dispersed material, semiconductors, solutions, nanoalloys, glasses and others. In this work, on example of studies carried out at the EXAFS station of SSTRC for a variety of nanosized systems used in materials science, catalysis, biology, the possibilities of XAFS spectroscopy are shown as an independent method and in combination with other physical methods of research, such as HRTEM, XRD, XPS, XRF. Possibilities of an integrated approach to study nanostructured systems having

complex composition: ordered semiconductor nanostructures, nanocomposite catalysts, biological nanomaterials and others are demonstrated. Some conceptual aspects of new EXAFS beamline of Siberian Circular Photon Source (SKIF) are discussed. This work was supported by the Russian Foundation for Basic Research (projects No. 16-03-01139, 17-33-50198, 18-03-01251).

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## New manufacturing method of a stamp for fabrication biochips

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A new manufacturing method of a stamp or a mold is described. They can be used to mold biochips of various plastics. A typical sequence of operations in LIGA technology is as follows: the formation of a thick resistive mask by deep x-ray lithography, the creation of a stamp (or mold) by galvanoplasty and the manufacture of plastic products using a stamp (or mold). This report describes a different sequence of operations: the formation of a thick resistive mask by deep photolithography on the working surface of blank of stamp (or mold), creating a relief of stamp by ion-beam reactive etching through a resistive mask, stamping (or casting) of plastic products, using the resulting stamp (or mold).

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## Synchrotron radiation investigation of PVC transformation to carbon structure under electron beam treatment

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Were performed the decomposition of PVC under electron-beam irradiation (ILU-6, BINP SB RAS) without high temperature heating and melting as usually take place during pyrolysis. The product of PVC radiation treatment is carbon material (data of element analysis and Raman spectroscopy). This material has a resistance to short-term exposure to oxidizing atmosphere at temperatures above 600 oC. The morphology of the particles of carbon material analogically of the samples of initial polymer which is very unusual for pyrolysis. Transmission electron microscopy and SAXS investigations was showed the formation of ordered structures with particle sizes of 25-30 Å.

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## Method of fabricating LIGA masks of a tungsten foil

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The design and fabrication method for self-supporting mesh structures, used as high-contrast LIGA-masks in x-ray lithography of the wavelength range  $\lambda \approx 0,5 \div 3 \text{ \AA}$ , are described. The LIGA-masks are produced by through-thickness patterning a solid tungsten foil fixed on a supporting ring using reactive ion-beam etching through a resistive mask of a corresponding topology. The LIGA-masks of this type are intended for manufacturing afterwards thick planar metal microstructures playing a role of quasi-optical filters in the terahertz range of the electromagnetic spectrum.

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## **X-ray lithographic fabrication of thick metal microstructures for terahertz applications**

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The design and fabrication method for thick (with thickness up to  $\sim 1 \text{ mm}$ ) planar metal microstructures of subwavelength topology, intended to control electrodynamic characteristics of radiation beams in the terahertz (THz) range of the electromagnetic spectrum, are described. Such structures have a number of advantages as compared to pseudometallic microstructures made of polymethylmethacrylate (PMMA) with metallized surface. The latter are inferior to the former ones due to such drawbacks as shorter life (as a result of natural aging of the polymer), restrictions on the radiation power density for the impinging THz beams, etc. The structures made entirely of metal do not exhibit the aforementioned disadvantages.

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## **The application hard X-rays of synchrotron radiation for determination of the minimum detection limits of heavy platinumoids (Os,Ir,Pt) and Au by the XRFA-SR method**

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The experiment on analysis of the elemental composition of samples by their X-ray fluorescence spectra were performed at the synchrotron radiation station using radiation from the 9-pole wiggler on VEPP-4M at the Siberian Center for Synchrotron and Terahertz Radiation (SSTRC). The samples Russian and International standards were used (G-3, KN-1, KM-1, VP-2, GMD-4, GMD-5, COG-13-3). The results on the minimum detection limits (MDLs) of heavy platinumoids (Os,Ir,Pt) and Au based on the lines of the K series of the characteristic radiation excited by photons with energies of 100 KeV were obtained.

The work was done using the infrastructure of the Shared-Use Center “Siberian Synchrotron and Terahertz Radiation Center (SSTRC)” based on VEPP-4M of BINP SB RAS (Novosibirsk, Russian Federation). This work was funded by the RFBR and Government of the Novosibirsk Region within the framework of the research project 17-45-540618.

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## Orbital changes of climate in the northwestern part of the Pacific Ocean according to the data of the study of bottom sediments by XRFA

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The chemical composition of the deep-sea sediments of column LV 76-18-1 (north-west of the Pacific) was measured at the collective station, VEPP 3 (Institute of Nuclear Physics, SB RAS). Coordinates of the column: 49 ° 03.2 'N, 168 ° 32.3'E, depth of the sea 2683 m, column length 810 cm.

The aim of the study is to study climate change in the northwestern Pacific Ocean, both on the scale of the orbital changes, and in millennia changes in the global climate in the past.

This region of the Pacific is extremely poorly studied, and therefore our results will reveal new patterns in climate change in this region and understand the mechanisms of their connection with global climate changes based on well-studied and dated data of the North Atlantic (Sarnthein et al., 2007), ice cores of Greenland (NGRIP members, 2004) and Antarctica (Jouzel et al., 2007) and monsoon southeast Asia (Wang et al., 2008). The preliminary age scale of this column with Isotope Oxygen Precipitation (BKS) boundary boundaries according to Bassinot et al. (1994) was established based on a study of sediment lithology, tephrochronology, color characteristics of sediments (clarity of sediment and parameters b), element composition and magnetic properties of sediments. Changes in the content of individual elements allow us to reconstruct the changes in biological productivity, the intensity of ventilation of deep waters and other parameters of the medium (the content of Ca, Br and As, U, respectively).

In the spectra of the elements of K Rb Ca Br Nb, etc., there are lines with a periodicity of 19 and 24 thousand years and 40 thousand years, and in the spectra of Br and Nb there are lines of more high-frequency. This is evidence of the impact on the regional climate and environment of both orbital changes in the parameters of the Earth's rotation, and the millennial climate processes. This work was supported in the framework of the joint work of the Far Eastern Branch of the Russian Academy of Sciences with the National Science Council, Taiwan for 2017 (project number 17-NSN-003 “High-resolution records of responses of the paleoceanology of the Bering Sea and the northwestern Pacific to global climatic changes in the late Pleistocene-Holocene”).

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## Time-resolved X-ray diffraction experiment investigations of ultrafast processes in BINP SB RAS Novosibirsk. Status and perspectives.

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Solid state chemistry studies several irreversible fast processes that require a single - bunch mode of investigation and are initiated by various external influences: detonation, shock waves, laser heating. As a result of this action, processes are initiated in the sample (adiabatic compression and heating, development of thermal stresses, increase of pressure in the local region), which creating extreme conditions (temperature of 10000 C, pressure of 1 million atmospheres), leading to irreversible structural changes associated with chemical reactions, phase transitions, the destruction of the material. These processes are so unusual, and difficult for research because of the lack of appropriate instruments, that the nature of these phenomena occurring only under extreme conditions is unknown to date. Therefore, it is extremely important to develop new installations on synchrotron radiation channels in order to uncover the secrets of nature. Only with the help of synchrotron radiation can the most effective advance in this area. Wiggler. In BINP SB RAS, for single-bang experiments, a VEPP-4 collider is used at an energy  $E = 4.5$  GeV and a 9-pole wiggler with a field of 2 T. The current of one bunch is 20 mA. The number of bunches from 1 to 16. The length of the bunch is 78 ps. The minimum interval between bunches is 76 ns. X-ray optics. Refractive optics is manufactured by LIGA technology [ 1]. One lens consists of 150,000 microelements made of SU-8. On the VEPP-4, such lens compresses the beam 10 times. On a new source of synchrotron radiation SKIF, which began to be designed, the beam can be compressed 1000 times. Detectors. A fast one-coordinate X-ray detector was developed for single bunch experiments [ 2]. The detector enables fast recording of 100 diffraction frames with an exposure time of 73 ps, space resolution 100 micron and a periodicity of 100 ns. Thus, we can record X-ray “movies” with high time resolution, which store information about the dynamics change of structure of the object under external action. Explosion chamber. Currently, our team uses two blasting chambers in which it is possible to blast an explosive charge of 15 g and 200 g, which have diameters of 60 cm and 120 cm, respectively. At present, work has begun on the design of an explosive chamber for the charge of an explosive weighing 2000 g. Gas dynamical gun. To study shock waves, a cannon is made that has the following parameters: bullet velocity – 600 m / s, bullet diameter – 20 mm, bullet weight – 500 g. The cannon with a bullet velocity of 12 km / s is designing for study the impact of micrometeorite on the surface of a space apparatus. Methods of research. Three research methods were implemented in a single-banshee regime: the X-ray imaging, the small-angle X-ray scattering (SAXS), and the X-ray diffraction. The X-ray imaging is used to tomographically reconstruct the density distribution of detonation products during an explosion (of particular interest is the reaction zone and the structure of the detonation front) [ 3], to study shock waves and ejected products density distribution in spallation processes [ 4, 5]. Small-angle X-ray scattering is used to study the nucleation and growth of nanoparticles [ 6, 7]- the products of a chemical reaction during detonation (for example, nanodiamonds), is used to analyze the size of the particles formed during ejected in spallation processes. X-ray diffraction is used to study the change in the crystal structure of the material of the first wall of a thermonuclear reactor during the ELM discharge, simulated by laser pulse heating with parameters:  $E = 100$  J, pulse duration 120 microseconds [ 8].

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## Study of Phase Transformation of Iron Containing Catalyst in Ethylene-Ammonia Mixture Decomposition Reaction

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Nitrogen doped carbon nanomaterials (N-CNM) currently attract a lot of interest due to their special physical, chemical and functional properties [1]. Nitrogen doped carbon nanotubes (N-CNT) look most investigated in details. It is generally recognized that N-CNT growth proceeds over iron containing catalysts with mixture of hydrocarbons and ammonia, as well as organic compounds, as a source of carbon and nitrogen. So the aim of this work is the study of the transformation of iron containing catalyst phase composition during the reaction of N-CNT growth in ethylene-ammonia mixture. The reaction of ethylene-ammonia decomposition over iron containing catalyst leads to formation of bamboo-like N-CNT with nitrogen content up to 7 at. %. Pure ethylene decomposition over the same catalyst gives CNT with coaxial cylindrical graphite layers packing. To explain the difference of CNT morphology the investigation was carried out by means of Ex Situ and In Situ X-ray diffraction. According to Ex Situ XRD data the initial sample of catalyst consists of two phases – metal iron and iron-alumina spinel Fe[Fe,Al]<sub>2</sub>O<sub>4</sub>. The preliminary reduction of the catalyst at hydrogen flow at 650-700°C makes the iron content increased that was demonstrated by In Situ experiments. Under consequent exposure of reduced catalyst to ethylene-ammonia mixture at 650°C the spinel phase almost disappears and iron carbide phase arises. In contrast, the phase composition of catalyst doesn't changes while the reaction with pure ethylene. However, at 700°C the catalyst demonstrates similar behavior under both pure ethylene and ethylene-ammonia mixture.

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## Study of the reduction process of Co<sub>3</sub>O<sub>4</sub> in supercritical isopropanol using In Situ SR XRD

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Because of the development of chemical technology, the emergence of new materials and the improvement of engineering solutions, supercritical (SC) fluid technologies become more and more economically attractive for carrying out various chemical processes (extraction, separation, chemical reactions). Approaches using the SC state of the substance, allow the creation of unique functional materials that are difficult, and sometimes impossible to obtain using traditional approaches. It is known that isopropanol in the SC state ( $T_{crit} = 235^{\circ}\text{C}$ ,  $P_{crit} = 53$  bar) allows the reduction of cobalt oxide (Co<sub>3</sub>O<sub>4</sub>) at temperatures lower than when hydrogen is used as a reducing agent [1]. Thus, it was shown by the ferromagnetic resonance method that this feature of the isopropanol SC avoids the high-temperature sintering of metal particles upon reduction and makes SK isopropanol a promising reducing agent for the preparation of dispersed metallic Co catalysts [2]. This work is dedicated to the studying of the reduction process of Co<sub>3</sub>O<sub>4</sub> cobalt oxide by SK-isopropanol by In Situ Synchrotron Radiation X-ray diffraction (In Situ SR XRD). The experiment was carried out at Beamline No.8 of VEPP-4M storage ring. Since the sample is placed to glass capillary of 1 mm diameter with thick walls of about 0.1-0.15 mm the radiation of high energy is necessary to minimize radiation absorption. The SR XRD patterns were registered with Toshiba FDX4343R Flat Panel detector. Time resolution was 4 sec/frame.

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## A family of precision switch-mode power supplies designed for highly inductive superconducting magnets

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This report presents a family of precision switch-mode power supplies specially designed for highly inductive superconducting magnets used in particle physics experiments. The power supplies have an output current range from 500 to 1300A and are adapted to non-linear load behaviour while maintaining high stability of the output parameters.

Circuit designs aimed at reducing switching losses and increasing the overall efficiency are presented.

Some aspects of superconducting magnets quench protection systems and save energy extraction have also been considered.

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## Hard X-ray operation mode at VEPP-4M

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There is synchrotron radiation experimental program at electron-positron collider VEPP-4M. Taking to account maximum beam energy, hard X-ray experiments at the storage ring seems rather attractive. The paper describes VEPP-4M performance at high energy with high current. New hybrid magnet wiggler developed recently specially to increase photon flux in the range of 50÷100 keV is also discussed.

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## Synchrotron Radiation Activity in the Novosibirsk Scientific Center

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The Siberian Synchrotron and Terahertz Radiation Center provides possibilities for the number of the researcher groups to use modern synchrotron radiation applications and research techniques. A big part of the activity is devoted to developing new original approaches for synchrotron radiation usage. The report covers activity on synchrotron radiation applications at the Siberian Synchrotron and Terahertz Radiation Center, as well as some bright results of recent research.

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## Project of Transmission X-ray Microscope-Interferometer for 15-35 keV Synchrotron Radiation

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A new Transmission X-ray Microscope-Interferometer, which is based on X-ray refractive lenses and diffractive gratings, was developed for 15-35 keV photon energies in order to create the X-ray optics at the Siberian Center of Synchrotron and Terahertz Radiation and to set up the TXMI at the front-end #2 of storage ring VEPP-4M. Unlike with diffractive zone plates, which are applied at present as objectives in modern X-ray microscopes, there is no a requirement of a high spatial transverse coherence for X-ray refractive lenses. In case of zone plate, the coherence should be comparable with 50-100 microns of a zone plate diameter, that leads to a pure photon flux at the object plane and, respectively, to a necessity of its cryogenic treatment of a specimen and its fixation at vacuum conditions. X-ray refractive optics enable to use hard X-rays, providing X-ray microscopy imaging at air environment and with low radiation damages. In order to increase sensitivity of the X-ray microscopy phase contrast imaging of the objects, which have homogeneous X-ray absorption or transparency (for an example, living biological cells in their habitat, i.e. in the open air, in a water solution), the X-ray interferometric gratings are set nearby the object zone. The local coherent zone for the interferometric gratings is formed by means of special X-ray refractive condenser lens with a long-length focal waist. The intensity changes of

moiré phase function from X-ray gratings because of X-ray phase structural heterogeneities of the micro objects are revealed typically by computed analysis of the X-ray microscopic image. Previous result [1] of computed X-ray phase contrast tomography using X-ray amplitude and phase gratings in the geometry of Talbot interferometer have demonstrated X-ray imaging visibility of internal structure of biological object with homogeneous X-ray absorption and phase structural non-uniformity with extremely low changes of X-ray refraction index decrement. And also, X-ray imaging of micro objects by means of a transmission X-ray microscope based on X-ray refractive objective and condenser lenses have been demonstrated previously [2] with spatial resolution up to tens of nanometers using monochromatic synchrotron radiation of 15-30 keV photon energies. The scientific novelty of the project is the combination of X-ray microscopy based on X-ray refractive long lenses for hard X-rays with X-ray interferometry and using X-ray gratings in the local coherence zone, which is formed by X-ray refractive condenser. In the paper, we describe the operations flowchart of the TXMI in detail. The block sequence on the SR beam course is the following: 1) A forvacuum chamber includes horizontal and vertical slits, a multilayered X-ray mirror-monochromator, a detector of the SR beam position and a blocker absorbing the direct beam. 2) The block of the X-ray refractive condenser lens has the air atmosphere. 3) Further, a forvacuum tube for the beam inflight after the condenser. 4) The block of a research object and the X-ray refractive objective lens with X-ray diffractive gratings is in the air environment. 5) Further, a forvacuum tube for the beam inflight after the objective lens. 6) The air block of the TXMI detector is disposed at the end of the beamline station. It includes an X-ray scintillator and an optical microscope with a planapochromate objective, inclined mirror and digital camera. [1] G. Schulz, T. Weitkamp, I. Zanette, F. Pfeiffer, F. Beckmann, Ch. David, S. Rutishauser, E. Reznikova, B. Mueller. High-resolution tomographic imaging of a human cerebellum: Comparison of absorption and grating based phase contrast. *Journal of the Royal Society Interface*, 7 (2010) 1665 – 1676. doi:10.1098/rsif.2010.0281. [2] E. Reznikova, T. Weitkamp, V. Nazmov, A. Last, M. Simon, V. Saile. Transmission hard X-ray microscope with increased view field using planar refractive objectives and condensers made of SU-8 polymer. *J. Phys.: Conf. Ser.* 186, 012070 (2009).

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## Beamlines and laser beam propagation on the NovoFEL

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Design of extended optical beamlines, calculated and measured beam parameters of three Novosibirsk free-electron lasers are presented.

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## Radioprotective effects of manganese oxide nanoparticles in the mice exposed to high doses of $\gamma$ -radiation.

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Nowadays, malignant brain tumors are one of the most complex oncological diseases for therapeutic intervention. While radiotherapy is a powerful anticancer treatment approach, it is associated

with severe side effects that drastically limit its therapeutic capacity. Since, the killing of cells by radiation is mediated by the ionization of irradiated matter, development of radioprotective agents which would be substantially improved the outcome of radiotherapy without an increase in the radioresistance of tumors is a one of the most important problems in the conduct of radiation therapy. Previously, it was showed the ability of nanoparticles of manganese oxide to accumulate in the animal's body. Toxic effects of nanoparticles were shown to be in a high dependence on particle size and morphology. For example, particles of a cubic shape and sizes of 50-200 nm have a minimal toxic effect and are able to accumulate in the brain. So, the aim of this study was to investigate the effects of nanoparticles on living tissues exposed to ionizing radiation. Using irradiation in doses close to clinical and several times exceeding them, in mouse model it was shown the high radioprotective activity of manganese oxide nanoparticles. In group of animals with head-only irradiation in a dose of 100 Gy, intraperitoneal injection of manganese oxide nanoparticles increase the survival rate from 69% to 100%. With the increase in the irradiation dose to 200 Gy, the protective effects of the nanoparticles are also preserved, but after head-only irradiation in a dose of 400 Gy all animals died within a few hours. Thus, we hypothesized that the mechanism of protective action of manganese oxide nanoparticles is associated with free radicals scavenging. Using hyperoxia animal pretreatment for enhancement of superoxide radical production during irradiation, ROS-scavenging properties of manganese oxide nanoparticle were confirmed. In group of animals exposed to hyperoxia before head-only irradiation in a dose of 200 Gy, intraperitoneal injection of manganese oxide nanoparticles increase the survival rate from 16% to 67%. Thus, we are the first ones to show in vivo the radioprotective effects of manganese nanoparticles.

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## Conceptual design of synchrotron beamline combined Photoelectron, Emission and Absorption Spectroscopies and Reflectometry for studying Electronic Structure in soft and tender x-ray range

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An undulator beamline with working name "Electronic structure" at the new synchrotron center SKIF (Novosibirsk, Russia) will be dedicated to advanced X-ray spectroscopies for studying electronic structure of the surface layers and in the bulk of different classes of compounds and functionalized materials with a high energy resolution in the x-ray range from 50 to 10000 eV. The beamline will consist of three beam stations. The first beam station will be based on X-ray Photoelectron Spectrometer making use such techniques as angle-resolved X-ray Photoelectron Spectroscopy (AR-XPS), HARD X-ray PhotoElectron Spectroscopy (HAXPES) and X-ray absorption near edge structure (XANES) under different conditions. The AR-XPS station will provide information about band structure, element composition, chemical states and local geometry of each elements which are present in the sample under study. Thanks to non-destructive HAXPES, one is indispensable in characterization of bulk materials, buried interfaces of multi-layered functional materials or as-grown samples. The second beam station will be based on a crystal spectrometer intended for x-ray emission spectroscopy (XES), which is also used for Resonant Inelastic X-ray scattering (RIXS), High Energy Resolution Fluorescent detected XANES or partially fluorescent yield (PFY) XANES. By combining the tunable incident energy with an x-ray emission spectrometer, resonance effects can be used for providing detailed information on the bulk electronic structure: studying spin-orbital interaction between electrons, configuration of electronic orbitals participating in chemical bonds as well as local structure of the probed element. The third beam station named as X-ray Reflectometry is dedicated to determine the optical properties of samples in transmission or reflection. The work will present a conceptual design of Electronic Structure Beamline and advanced application fields.

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## Structure and Phase Transitions Study of New Semiconducting Organo-Mineral Perovskite

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In a last few years a semiconducting material methylammonium lead triiodide CH<sub>3</sub>NH<sub>3</sub>PbI<sub>3</sub> with perovskite-like structure which has got general name Organo-Mineral Perovskite (OMP) attracts growing interest of researchers. Based on OMP Thin Film Solar Cells for several years of development demonstrate outstanding growth of efficiency of solar light to electricity conversion (from 4 up to 22%) [1]. Such a great performance becomes possible due to unique properties of OMP – optimal for solar cell energy gap width (1.5 eV), high solar light absorption coefficient (105 cm<sup>-1</sup>), low exciton decay energy (16 meV), and large path length of charge carriers (1 μm in thin film and 175 μm in single crystal). OMP may be applied as well for lasers, LEDs, X-ray and [U+<sup>235</sup>F067]-ray detectors. We have developed synthesis method and synthesized OMP single crystals. To produce OMP powder two precursors were used: lead iodide and iodine methylamine. Single crystal growth was carried out from saturated solution with temperature decrease. Iodine hydride acid was used as a solvent. During the growth the temperature of solution was decreased under control from 65°C down to 21°C with step size of 0.1° for 40 min. The growth proceeded for 12 days. The chemical composition of single crystals was confirmed by means of energy dispersive spectroscopy (EDX) and photoelectron spectroscopy (XPS). Reflection High Energy Electron Diffraction (RHEED) indicates high quality of surface of single crystals synthesized. The crystal structure of CH<sub>3</sub>NH<sub>3</sub>PbI<sub>3</sub> was investigated by means of synchrotron radiation XRD within wide temperature range allowing to detect phase transitions of OMP at ~160 and ~330 K  
W.S. Yang, et al, Science, 356, 1376-1378, (2017)

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## New light source for Novosibirsk Scientific Center, machine review

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The main problem of the Siberian Synchrotron and Terahertz Radiation Center (SSTRC) is absence of the modern light source. In spite of intensive exploring of SR beams and SR analytic techniques of the SSTRC by a large number of user groups, the SR parameters of the currently used beams strongly restrict capability of the used techniques. Budker INP has rich experience and technologies for developing and building complex accelerator facilities. Moreover, the unique scientific environment of the SSTRC provides a high demand for modern light source in the Novosibirsk Scientific Center only. Thus, the creation of the new light source is highly relevant issue. After analysis of the user requirements and modern accelerator approaches for design of magnetic lattice for high brightness light source the Budker team proposed an option of the magnetic structure of a new light source for Novosibirsk Scientific Center. The project was named “SKIF”. Current report describes the main parameters for the proposed light source as well as common design of the accelerator complex, magnetic structure of the main ring and buster accelerator. The preliminary schedule and systems cost analysis are included.

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## Development of XRFA-SR method with the hard X-ray (65 keV) range for paleoclimate reconstruction (Lake Baikal region) on the storage ring VEPP-4M

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The Selenga River is the main inflow of Lake Baikal and its suspended material penetrates away into the lake. The catchment of the Selenga River is 447000 km<sup>2</sup> and water content of the river is a proxy of moisture of the south part of East Siberia and the Northern Mongolia. In this reason, studding of geochemical pattern of bottom sediments of Lake Baikal we can reconstruct water content of the Selenga River in the past.

The analysis of bottom settlings by the scanning X-ray fluorescence spectrometry method (SR-XRF scan) is carried out in the context of paleoclimate research.

All measurements were carried out on the station of XRFA-SR on the storage ring VEPP-4M. The work was done using the infrastructure of the Shared-Use Center “Siberian Synchrotron and Terahertz Radiation Center (SSTRC)” based on VEPP-4M of BINP SB RAS. The work was supported by The Ministry of Education and Science of The Russian Federation (project RFMEFI62117X0012).

The conditions of VEPP-4M were the following: Ee = 4.5 GeV, SR beam from 9-pole wiggler with B = 1.9 T, and Ie = 20 mA. The excitation energy for the determination of concentrations in practically all bottom sediments was 65 keV. The new XRFA-SR station on the storage ring VEPP-4M allowed to move to a new level of research on paleoecological reconstructions, by increasing the number of chemical elements. The work is supporting by the RFBR grant No. 17-29-05016.

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## The application of synchrotron radiation of the VEPP-4M storage ring for determine the concentrations of rare-earth elements in geological samples by the XRFA-SR method

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Rare-earth elements contain valuable information on rock forming geological processes that is used in geochemical research. The hard X-rays of synchrotron radiation of the VEPP-4M storage ring for determine concentrations of rare-earth elements in geological samples was used. The excitation energy for the determination of rare-earth elements (La-Lu) was 57-85 keV. The Compton and the multiple scattering of radiation from geological samples also was analysed. The article presents the main parameters of the minimum detection limits of rare-earth elements in geological samples by the XRFA-SR method.

The work was done using the infrastructure of the Shared-Use Center “Siberian Synchrotron and Terahertz Radiation Center (SSTRC)” based on VEPP-4M of BINP SB RAS (Novosibirsk,

Russian Federation). This work was funded by the RFBR and Government of the Novosibirsk Region within the framework of the research project 17-45-540618.

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## Hybrid 9-pole wiggler as a source of «hard» X-ray radiation at the VEPP-4M accelerator complex

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A hybrid 9-pole wiggler was developed in the Budker Institute of Nuclear Physics and successfully installed at the VEPP-4M accelerator complex. This wiggler consists of 9 poles, which are electromagnets with an iron core. In order to achieve the greatest value of the field in the gap, permanent magnets with magnetization of 1.2 T are set between the wiggler poles. This combination of electromagnets and permanent magnets made it possible to achieve a maximum field of 1.9 T, with an inter-pole gap of 30 mm. At present time, several research methods have been developed using the “hard” X-ray range (50-250 keV) based on the SR beam line from a 9-pole wiggler. On the beamline, were carried out range of investigations with application X-ray imaging of fast processes, X-ray computed tomography, XRD and XRF analyzes. The best results are shown in the report.

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## Synchrotron based investigations of ZnS:Cu(Mn);Cl nanocoatings on porous alumina

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Synchrotron facilities give more precise and reliable information about structure and electronic properties of materials. For instance EXAFS and X-Ray Diffraction techniques are the powerful methods of obtaining of structural information and XANES allows to investigate an electronic structure of materials. During last decade materials science developing the methods of obtaining and studying of new materials for light emitting devices, particularly, electroluminescent emitting panels based on nanostructured thin films and nanostructures [1]. Traditional materials for electroluminescent light sources are zinc sulfide doped with copper, chlorine, manganese and other elements. Although these materials are well studied but some information about the local environment of dopands elements is still unknown. In this work we propose templating approaches based on the formation of doped by Cu(Mn);Cl ZnS nanocoatings on the surface the porous alumina films with highly ordered and controlled diameter channels. Porous alumina were obtained by anodization process of aluminum films thermally deposited on glass/ITO/SiO<sub>2</sub> substrates when 100 nm thick SiO<sub>2</sub> film were used as a buffer layer. We trying to identify the influence of synthesis conditions on the structure, electrical, including light emitting, properties of materials. All of

the above defines the scope of work: EXAFS, XANES and XRD investigations of doped ZnS nanostructures for the creation of a new class of fluorescent materials to form the basis of their phosphor layers for high-performance and high-brightness light-emitting electroluminescent panels. This work is supported by Russian Foundation of Basic Research (Project N<sup>o</sup> 16-48-180303).

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## SR XRD Phase Analysis of Kidney Stones within Model Object at VEPP-4M Storage Ring

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The results of the study of kidney stones of different mineral composition are presented. Previously, studies were conducted of human kidney stones obtained from patients, both after lithotripsy and after abdominal operations [1]. The experiments mentioned above were carried out at beamline No.4 of VEPP-3 storage ring at Siberian Synchrotron and Terahertz Radiation Center (SSTRC), Novosibirsk, Russia, using photon energy  $E=33.7$  keV. Diffraction patterns were registered by Image Plate area detector MAR345, the thickness of model sample was  $\sim 20$   $\mu$ m, exposure time 2 min. Definite limitations on object thickness as well as exposure time appear to be crucial for this photon energy. It is clear that to decrease radiation loading and to remove limitation on the sample size (patient weight) the higher radiation energy is to be used. The experiments described here were executed at beamline No.8 of VEPP-4M storage ring at SSTRC. The electron energy of this storage ring is 4.5 GeV and the radiation is emitted by 11-pole wiggler. Such source provides essential photon flux at photon energy about 100 keV, which is well suitable for In Vivo kidney stones diagnostics. 2D XRD pattern of kidney stones of different phase composition were registered by Toshiba FDX4343R Flat Panel detector. The 1D pattern can be obtained from 2D pattern by integration of diffracted intensity over azimuthal angle. 1D XRD patterns of free kidney stone and stone in model object which was cylindrical plastic vessel of 17 cm diameter filled with water were recorded and discussed.

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## SR XRD Functional Materials Diagnostics

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X-ray diffraction studies of structure, structural and phase transformations of functional materials under different external conditions are carried out at SR beamlines of VEPP-3 and VEPP-4M storage rings at Siberian Synchrotron and Terahertz Radiational Center. Beamline No.2 of VEPP-3 storage ring is dedicated for high angular resolution and anomalous scattering experiments under normal temperature and pressure. The available photon energy range is 5-20 keV, diffraction patterns are recorded in step-by-step scanning mode within angular range  $2\theta=0.5-140^\circ$ . Time-resolved experiments with samples being under high temperature and gaseous reaction mixture are conducted at Beamline No.6 of VEPP-3 storage ring. It operates at three fixed photon energies, 7.16, 7.56 and 12.3 keV, diffracted intensity is registered by OD-3M position sensitive detector. Three different high temperature XRD chambers are available for experiments. High photon energy experiments are executed at Beamline No.8 of VEPP-4M storage ring. The source provides photon energy up to 100-120 keV with sufficient intensity which allows to investigate structure and phase composition of functional materials placed to autoclave or electrochemical cell. The diagnostics of materials by means of Total X-ray Scattering is also available. The latest activities of Beamlines and results of XRD experiments will be presented.

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## Calibration of the spectral properties of crystals in the soft X-ray range

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The calibration of the spectral properties of the crystals (quartz, mica and potassium biphthalate) were carried out at the station "Cosmos" using synchrotron radiation from the the VEPP-4 storage ring. The measurements were carried out by the rocking curve method at the 1-6 keV spectral range. The report describes measurement conditions, data processing procedures and the experimental results. The work relating to the development of the station is carried out using the infrastructure of the Shared-Use Center "Siberian Synchrotron and Terahertz Radiation Center (SSTRC)" based on VEPP-4M of BINP SB RAS.

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## Synchrotron radiation induced photo etching of cesium iodide

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Synchrotron radiation induced photo etching of cesium iodide (CsI) was investigated. The model for photo etching was proposed and compared with experimental data. Dependence of the etching rate on the SR spectrum and on the substrate temperature were revealed. We estimate possibility of manufacturing of high aspect ratio microstructures of CsI scintillation material.

The work was done using the infrastructure of the Shared-Use Center “Siberian Synchrotron and Terahertz Radiation Center (SSTRC)” based on VEPP-3 of BINP SB RAS (Novosibirsk, Russian Federation).

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## Complex study of test samples to develop combined SR techniques

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The first results of the study of test samples of technological and natural origin having complex composition to develop combined SR techniques (XAFS, XRF) are shown. The main purpose of investigation is to pass developed approach for SR studies of microinclusions and microparticles in future. All spectra, using  $\mu$ - and standard modes, were recorded both on experimental module X-ray Confocal Microscopy situated on NRC “Kurchatov Institute” (Moscow) and at XRF, EXAFS stations of Siberian Synchrotron Terahertz Radiation Center (SSTRC, Novosibirsk). The test samples having complex composition (such as low-percentage metal-oxide nanocomposites, nanoalloys, specimens of natural origin) were synthesized and/or selected. The test samples and references necessary for testing the SR techniques were performed by a set of methods (HRTEM, XRF, XPS, SEM, EDA, etc.). For the test systems under investigation, new information will be obtained on the phase and elemental compositions, atomic structure, morphology, structural parameters. The prospects of the proposed approach for future research are shown. This work was conducted within the framework of SB RAS program II.1 (project 0303-2018-0010, -17-117112840087-0)

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## The emission of microparticles from metal joints under shock wave influence

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The output of a strong shock wave on the free surface (SP) of the metal plate leads to the development of instabilities and the subsequent formation of a flux of a finely dispersed fraction

(shock-wave “dusting”). The flux of particles has a distribution in space in size and velocity. The development of the instability growth process on the metal SP and, accordingly, the characteristics of the flow of microparticles depends on the degree of plate inhomogeneity (the size of the grooves and protrusions), the phase state of the material, the loading conditions, etc. To study the dusting process, the method of pulse radiography, the method of piezoelectric sensors, optical methods (including holographic methods) are traditionally used. The measured sizes of the microparticles range from a few microns to hundreds of microns. It is assumed that there are smaller particles in the stream, but the existing methods can not solve them yet. We present the results of a study of dusting processes using synchrotron radiation (SR). The experiments were carried out at the VEPP-3-VEPP-4 accelerator complex (INP SB RAS), the SR registration was carried out with a precision high-speed DIMEX detector. Flows of microparticles from a free surface of copper and tin were investigated. Mass distributions along micro-jets formed from micron-sized grooves were obtained.

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## The project of the beam-line "Fast processes" at synchrotron radiation source SKIF in Novosibirsk

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A new beamline “Fast processes” will be put into operation in the test mode at the SKIF synchrotron radiation source in 2024. The beamline will have stations: 1) “Plasma”; 2) “High temperature”; 3) “Extreme conditions”. The stations will supervise accordingly: 1) Institute of nuclear physics of the SB RAS; 2) Institute of Solid State Chemistry and Mechanochemistry of the SB RAS; 3) Institute of Hydrodynamics of SB RAS. The beamline will use the radiation from a 100-pole superconducting wiggler with a 5 T field. At a photon energy of 30 keV, the brightness will be  $10^{19}$  phot / (sec mm<sup>2</sup> mrad<sup>2</sup> 0.1% BW). The energy range will be from 5 to 200 keV. X-ray optics. A dual monochromator with a fixed output can provide monochromatization from  $10^{-2}$  to  $10^{-4}$ . If necessary, a monochromator can skip a white beam. Focusing mirrors will be used, which will ensure focusing and cutting of high harmonics if necessary. To work in a high energy area, refractive optics will be used. Both focusing and defocusing lens will be used. Refractive optics is manufactured by LIGA technology [ 1]. One lens consists of 150,000 microelements made of SU-8. On the VEPP-4, such lens compresses the beam 10 times. On a new source of synchrotron radiation SKIF, which began to be designed, the beam can be compressed 1000 times. A fast chopper will be used, for investigating of fast processes, with exposure from a single bunch with continuance near 1 - 73 ps. Detectors. A fast one-coordinate X-ray detector was developed for single bunch experiments [ 2]. The detector enables fast recording of 100 diffraction frames with an exposure time of 73 ps, space resolution 100 micron and a periodicity of 100 ns. Thus, we can record X-ray “movies” with high time resolution, which store information about the dynamics change of structure of the object under external action. Detectors OD-3M [ 3] and PILATUS will also be used. Required element will be beam monitor. The monitor will determine both the intensity of the beam and its position with an accuracy of 1 micron. The read out

frequency of monitor will be 100 MHz, which will allow to determine the position of the radiation from each bunch of electrons. Explosion chamber. Currently, there are two blasting chambers in which it is possible to blast an explosive charge of 15 g and 200 g, which have diameters of 60 cm and 120 cm, respectively. At present, work has begun on the design of an explosive chamber for the charge of an explosive weighing 2000 g. Gas dynamical gun. To study shock waves, a cannon is made that has the following parameters: bullet velocity – 600 m / s, bullet diameter – 20 mm, bullet weight – 500 g. The cannon with a bullet velocity of 12 km / s is designing for study the impact of micrometeorite on the surface of a space apparatus. Methods of research. Three research methods will used: X-ray imaging, the small-angle X-ray scattering (SAXS), and the X-ray diffraction. The X-ray imaging will used to tomographically reconstruct the density distribution of detonation products during an explosion (of particular interest is the reaction zone and the structure of the detonation front) [ 4], to study shock waves and ejected products density distribution in spallation processes [ 5, 6]. Small-angle X-ray scattering will used to study the nucleation and growth of nanoparticles [ 7, 8]- the products of a chemical reaction during detonation (for example, nanodiamonds), will used to analyze the size of the particles formed during ejected in spallation processes. X-ray diffraction will used to study the change in the crystal structure of the material of the first wall of a thermonuclear reactor during the ELM discharge, simulated by laser pulse heating with parameters: E = 100 J, pulse duration 120 microseconds [ 9].

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## XAFS study of platinum–gold alloyed nanosystem

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Recently, new functional materials containing nanosized particles consisting of two or more precious metals attract great interest, because ones are known to exhibit unique chemical and physical properties, which differ from that of monometallic particles. Amongst this new class of materials, metastable nanoalloys should be mentioned especially. The present work is devoted to the study of Au-Pt alloyed nanoparticles by XAFS spectroscopy method. The Au-Pt alloyed nanoparticles were prepared via decomposition of double complex salt  $[\text{AuEn}_2]_2[\text{Pt}(\text{NO}_2)_4]_3 \cdot n\text{H}_2\text{O}$  containing

both inorganic and organic ligands. Decomposition of the precursor was performed in a reductive atmosphere with varied temperature ramping rates. All XAFS spectra of the Pt-L3, Au-L3 edges were recorded at Siberian Synchrotron and Terahertz Radiation Center (SSTRC, Novosibirsk). The study of the genesis of the state and local structure of the Au-Pt formed nanoparticles, including the study of initial precursors were carried out. According to XAFS data, both the ramping rate and the final temperature of decomposition strongly affect the Pt–Au ratio in alloyed nanoparticles which are being formed. The interatomic distances and corresponded coordination numbers were established. All possible structural models were discussed. Additionally, the samples of catalysts were characterized by the TEM, EDX, XRD methods. The data obtained by all the methods are in a good agreement. The work has been supported by grant of Russian Science Foundation (project №16-13-10192). Vladimir Kriventsov thanks to the Russian Foundation for Basic Research for support of the analysis of the local structure (projects No. 16-03-01139, 17-33-50198, 18-03-01251).

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## Features of stabilization of Pd and Rh in model low-percentage supported catalysts

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Increased requirements for environmental safety and the introduction of new standards for the content of harmful impurities in waste gas mixtures led to significant progress in the development of devices to neutralize automobile exhausts and emissions of industrial gases based on catalytic systems containing noble metals, especially palladium and rhodium, allowing the transition to processes of low-temperature oxidation. The present work is devoted to studying the features of the state of palladium and rhodium and its stabilization centers in the active component of model low-percentage (less than 1%) palladium-rhodium catalysts supported on oxide carriers using XANES/EXAFS methods. All XANES/EXAFS spectra (Pd-K, Rh-K) of the studied samples were recorded at Siberian Synchrotron and Terahertz Radiation Center (SSTRC, Novosibirsk). The state and local structure arrangement of supported metal nanoparticles of the studied catalytic nanosystems was characterized in detail. Correlations between their catalytic properties, local structure distortions and state of metal components were demonstrated. The interatomic distances and corresponded coordination numbers were calculated by fitting. All possible structural models were discussed. Additionally, morphology and composition of the samples of catalysts were studied by the HRTEM, EDX and XPS methods. The data obtained by all the methods are in a good agreement. The work has been supported by grant of Russian Science Foundation (project №16-13-10192). Vladimir Kriventsov thanks to the Russian Foundation for Basic Research for support of the analysis of the local structure (projects No. 16-03-01139, 17-33-50198, 18-03-01251).

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## XAFS study of dispersed Pd catalytic nanosystem stabilized on oxide support

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It is well-known fact that Pd catalytic systems are very promising for processes of oil refining and petrochemistry. A direct structural XRD study of such systems is difficult due to methodological limitations, while the use of XAFS spectroscopy can provide the necessary data. The first results of the XAFS study of low-percentage Pd catalytic nanosystem stabilized on oxide support are shown. Synthesis of catalysts was carried out by chemisorption impregnation of supports with palladium acetylacetonate from benzene. The impregnation temperature was 20C. The content of palladium in the catalysts was 0.5 wt.%. Before the tests, the samples were subjected to a preliminary oxidation-reduction treatment at 400C. All XAFS (XANES/EXAFS) spectra of the Pd-K edge were recorded at Siberian Synchrotron and Terahertz Radiation Center (SSTRC, Novosibirsk). The study of the state and local structure of the active component were carried out. It was shown that, depending on the prehistory, the formation of different (metals, metal-oxides, oxides) nanosized forms of Pd, which are located on the surface of the supports. The interatomic distances and corresponded coordination numbers were established. All possible structural models were discussed. Additionally, the samples of catalysts were characterized by the HRTEM, EDX, XPS methods. The data obtained by all the methods are in a good agreement. The work is performed according to the Russian Government Program of Competitive Growth of Kazan Federal University. Vladimir Kriventsov thanks to the Russian Foundation for Basic Research for support of the analysis of the XANES (projects No. 16-03-01139, 17-33-50198, 18-03-01251).

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## Application of XAFS for study of nano-dispersed Pd catalysts

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This work is devoted to a study of the state and local structure of original nano-dispersed catalytic low-percentage systems Pd-M, M=Zn, Co, Mn, Ag used for production of alkanes or olefins fractions 4-12+ which components of biofuels obtained from biomass fermentation products. Nowadays great attention is attracted to development of highly effective approaches of converting renewable biomass into energy resources as biofuels. Some spirit compounds, such as ethanol, butanol, pentanol, glycerine, being biomass fermentation products, may be used as fuel components, both without any treatment and after catalytic converting, that provides production of more effective fuel components. This work is devoted to a study of the state and local structure of original nano-dispersed catalytic low-percentage systems Pd-M, M=Zn, Co, Mn, Ag used for production of alkanes or olefins fractions 4-12+ which components of biofuels obtained from biomass fermentation products. Initial samples of catalysts were prepared by impregnation and zol-gel methods from original mono- and hetero-metallic precursors located on the oxide supports. All XANES and EXAFS spectra of the studied samples were recorded at Siberian Synchrotron and Terahertz Radiation Center (SSTRC, Novosibirsk). Genesis of the local structure of the studied catalytic nanosystems was characterized in detail. The interatomic distances and corresponded coordination numbers were revealed. All possible structural models were discussed. Relations between their catalytic properties, local structure distortions and state of metal components were demonstrated. It was found out that active components of all studied systems were highly dispersed onto the oxide support surface and strong interaction with the support took place, resulting in formation of mixed surface MeOx - support oxides. Additionally, the samples of catalysts were characterized by the HRTEM, EDX, XPS methods. The data obtained by all the methods are in a good agreement. This work was conducted within the framework of the budget project #-17-117041710078-1 for Boriskov Institute of Catalysis. Vladimir Kriventsov thanks to the Russian Foundation for Basic Research for support of the analysis of the XANES data (projects No. 16-03-01139, 17-33-50198, 18-03-01251).

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## Structural study of PdM/C and PtMe/C supported catalytic systems

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In recent years, few new types of carbon supported catalysts, such as activated carbon fibers, Busofit and CNF carbon nanofibers-based catalysts, inspire a growing interest. As rule, Busofit and CNF based catalysts have improved catalytic selectivity, activity and thermostability as compared to the traditional carbon supported catalysts using carbon-black or Sibunit as supports. This modification of catalytic properties is believed to result from higher accessibility of the deposited active component to the reactants and from strong interactions between the catalyst particles and the support surface. This work is devoted to the comparative structural study of few promising Pd, Pd-M, Pt, Pt-M carbon-based catalytic systems using different carbon supports by the XAFS methods. All EXAFS spectra of the studied samples were recorded at Siberian Synchrotron and Terahertz Radiation Center (SSTRC, Novosibirsk). The local Pd, Pt and M (Zn, Ag, Ga, Fe, Cu, Co) arrangements of all the samples studied were established and the phase compositions were determined. It was shown that there are some differences of the metal arrangements for the studied systems. All possible structural models were discussed. In addition, these samples were studied by the TEM, EDX and X-ray diffraction methods. The data of all methods are in a good agreement. This work was conducted within the framework of the budget project #-17-117041710078-1 for Boreshkov Institute of Catalysis. Vladimir Kriventsov thanks to the Russian Foundation for Basic Research for support of the analysis of the XANES data (projects No. 16-03-01139, 17-33-50198, 18-03-01251).

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## The structural determination of gold catalytic nanosystems by XAFS spectroscopy method

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The structural determination of gold catalytic nanosystems by XAFS spectroscopy method

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This work is devoted to a detailed structural study of the nanosized Au, AuMe stabilized forms located on oxide supports by XAFS method. It is a well known fact that metallic gold is most inert among the precious metals due to its electronic structure, nevertheless the nanosized Au, Au-Me forms located on oxide supports may be used as very active catalysts for numerous industrially important reactions. These systems are very promising for complex organic synthesis, selective isomerization reactions and for processes of environmental catalysts, such as, CO oxidation and selective conversion of secondary alcohols. Reliable analysis of the nature of gold forms is extremely important for design of new effective gold based catalysts for different

industrial applications. Synthesis of low-percentage  $\sim 0.1\text{-}2\%$  Au, Au-Me samples was carried out under varying preparation methods (impregnation, deposition-precipitation with urea, direct ion exchange), calcination temperatures and activation methods. All XAFS spectra (Au-L3 edge) of the studied samples were recorded at SSTRC, Novosibirsk. The state of metal components and the local Au structure arrangements of the prepared catalysts were studied. The Au-O, Au-Au, Au-Me interatomic distances and corresponded coordination numbers were established. All possible structural models were discussed. It was shown that different stabilized gold forms located on oxide supports were formed, most probably depending on a sample prehistory: Au(3+) cations, having octahedral oxygen surrounding and metallic Au-Au, Au-Me nanoparticles  $\sim 10\text{-}100\text{\AA}$ . Some correlations between catalytic activities and structural functional properties of the studied samples were shown. Additionally, samples of catalysts were characterized by the HRTEM, EDX, XRD, XPS methods. The data of all methods are in a good agreement. This work was conducted within the framework of the budget project #-17-117041710078-1 for Boreskov Institute of Catalysis. Vladimir Kriventsov thanks to the Russian Foundation for Basic Research for support of the analysis of the XANES data (projects No. 16-03-01139, 17-33-50198, 18-03-01251).

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## Application of XAFS for the study of state and genesis of nanosized forms of precious metals stabilized on the support of the different nature

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Application of XAFS for the study of state and genesis of nanosized forms of precious metals stabilized on the support of the different nature

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This works demonstrated the results of a study of the genesis of nanosize forms of precious metals – Ir, Pt, Pd, Au, Ru and others, existing in mono- and bimetallic supported catalysts, stabilized on the oxide and carbon supports of different nature by XANES/EXAFS spectroscopy. It is well known that nanosize forms of noble metals exhibit a high catalytic activity in various industrial important processes and also ones are promising for applications of alternative energy. Development of methods of synthesis can provide to a significant economical profit, due to lower metal content in the active component and optimization of the catalytic properties. Detailed study of the nature of different stabilization forms of the supported metal, it is necessary to create new effective catalysts for various industrial applications. Samples of supported catalysts were prepared under varying preparation methods (zol-gel, impregnation, deposition-precipitation) from different precursors. All XANES/EXAFS spectra of the Pt-L3, Ir-L3, Au-L3, Pd-K, Ru-K edges were recorded at Siberian Synchrotron and Terahertz Radiation Center (SSTRC, Novosibirsk). The study of the genesis of the active component of the local structure, including the study of initial precursors and catalysts after reduction and change the state of the active component were carried out. It was shown that, depending on the prehistory, the formation of different (metals, metal-oxides, oxides) nanosize forms of precious metals, which are located on the surface of the supports. The interatomic distances and corresponded coordination numbers were established. All possible structural models were discussed. Additionally, the samples of catalysts were characterized by the HRTEM, EDX, XRD, XPS methods. The data obtained by all the methods are in a good agreement. The data of all methods are in a good agreement. This work was conducted within the framework of the budget project #-17-117041710078-1 for Boreskov Institute of Catalysis. Vladimir Kriventsov thanks to the Russian Foundation for Basic Research for support of the analysis of the XANES data (projects No. 16-03-01139, 17-33-50198, 18-03-01251).

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## Development of X-ray coherent optics for fourth generation synchrotrons and XFELs.

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New ultimate parameters of the beam provided by the diffraction-limited sources will open up unique opportunities to build up a new concept for the beam-transport and conditioning systems based on in-line refractive optics [1]. Taking an advantage of the reduced horizontal source size and divergence, the refractive optics integrated into the front-end can transfer the photon beam almost without losses from the source directly to the end-stations. In this regard, development of diamond refractive optics is crucial [2,3]. In addition to traditional focusing applications, the refractive optics can provide the various beam conditioning functions in the energy range from 3 to 200 keV: condensers, micro-radian collimators, low-band pass filters, high harmonics rejecters [4], beam-shaping elements [5].

The implementation of the lens-based beam transport concept can significantly simplify the layout of majority of new beamlines, opening novel opportunities for the protein crystallography [6] and for the material science research under extreme conditions [7-8]. The versatile beam conditioning properties of refractive optics enable to develop and implement new X-ray coherence-related techniques including interferometry [9-11], phase contrast imaging [12-14] and dark field microscopy [15] using light polymer micro-objectives made by additive technology [16].

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## BESSY VSR Project: design status and project challenges

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Complementary to many other upgrade programs, BESSY VSR strives to provide a train of ultra-short bunches while maintaining a large average beam current and thus high brilliance to the users. This upgrade program requires installing a set of superconducting cavities into the machine next to improvements and refurbishments in the existing accelerator chain.

In this presentation we present the current project status, describe the different preparation steps and tests currently being executed next to a summary of the challenges to tackle.

Научное издание

**Synchrotron and Free electron laser Radiation:  
generation and application (SFR-2018)**

June 25 – 28, 2018

Budker INP

**Book of Abstracts**

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Ответственный за выпуск М.В. Кузин

Сдано в набор 24.05.2018 г.

Подписано в печать 1.06.2018.

Формат 60x90 1/16 Объем 6.5 печ.л., 5.2 уч.-изд.л.

Тираж 140 экз. Бесплатно.

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Отпечатано в типографии Alexpress  
Новосибирск, пр. Лаврентьева, 6/1, оф. 104