Synchrotron and Free electron laser Radiation: generation and application (SFR-2016)

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Book of Abstracts
**Poster Session**

**In situ X-ray diffraction studies of Ce0.9Y0.1O2-δ, Ce0.65Pr0.25Y0.1O2-δ and Pr6O11**

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Ce0.9Y0.1O2??(YDC), Ce0.65Pr0.25Y0.1O2??(YPDC) and Pr6O11 were synthesized by Pechini technique and sintered at 1100 C. The samples were characterised by X-ray diffraction (XRD) and TEM methods.

It has been found that oxides behave differently after change of the ambient oxygen partial pressure (pO2). In situ XRD experiments showed that YPDC cell reversibly expands after change to the lower pO2 at 500-600C due to the oxygen loss while YDC didn’t show any significant structure changes. For the Pr6O11 the following structure changes were observed at the same conditions: monoclinic > cubic > trigonal.

We also estimated the oxygen mobility coefficients(kchem - oxygen surface exchange coefficient and Dchem - oxygen chemical diffusion coefficient) of all samples by analysis of the cell volume relaxation (CVR) curves obtained from the XRD data after abrupt change of the oxygen partial pressure from 100 mBar to 10 mBar. Since no relaxation was observed for YDC, kchem and Dchem could not be calculated. For YPDC process is limited by surface exchange thereby only kchem could be calculated. The following kchem values were determined for YPDC $1.3 \times 10^{-4}$ cm/s at 500 C and $1.6 \times 10^{-4}$ cm/s at 600 C. For Pr6O11 both kchem and Dchem could be calculated simultaneously and were about $5.0 \times 10^{-4}$ cm/s and $5.0 \times 10^{-7}$ cm$^2$/s at 500 C. In situ XRD experiments were carried out with involvement of equipment belonging to the shared research center 'SSTRC'.

**X-ray apparatus**

**Metrological station "Cosmos". Current status.**

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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 (80-2000 eV) and vacuum ultraviolet (10-100 eV) ranges. 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 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.

**Radiation sources and Centers**

**Status of Siberian Synchrotron and Terahertz Radiation Center. Activity review.**

**Author(s):** Dr. ZOLOTAREV, Konstantin
The Siberian Synchrotron and Terahertz Radiation Center is the oldest Russian center for synchrotron radiation applications. The center combines the research efforts of numerous scientific groups, mostly from institutes of the Novosibirsk Scientific Center, as well as groups from institutes and universities of other Russian cities. The center provides research groups with access to most popular 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.

Radiation sources and Centers

**NSLS-II Booster**
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The National Synchrotron Light Source II is a third generation light source, which was constructed at Brookhaven National Laboratory. This project includes a highly-optimized 3 GeV electron storage ring, linac preinjector, and full-energy synchrotron injector. Budker Institute of Nuclear Physics built and delivered the booster for NSLS-II. The commissioning of the booster was successfully completed.

**FEL-based study and THz radiation application**

Intensity and timing jitter compensated ultra-fast experiments at accelerator-driven photonsources

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Timing jitter and power instabilities are crucial parameters which greatly reduce the applicability of accelerator driven light sources for time-resolved experiments. In this contribution we present a technique that allows achieving few 10 fs time-resolution in experiments operating at cw repetition rates of up to 100 kHz at presents of timing jitter in order of ps. As we show on the example of a benchmark experiment based on THz driven magnetization dynamics, our method provides a sensitivity or dynamic range that is comparable to all-laser based techniques. Moreover we show how the time and power instabilities in combination with a high repetition rate can be turned from a problem into a favor an advantage and can be utilized as an ultrafast delay line and attenuator. Our method employs a fs-level arrival time monitor based on electro-optic sampling of residual pulses from a coherent diffraction radiator and a fast THz detector allowing for pulse to pulse detection of arrival time and pump pulse intensity. The monitor can operate at high repetition rates (presently up to a few 100 kHz) and low electron bunch charges (sub pC). The prototype device has been tested at the quasi CW linear RF electron accelerator (ELBE).
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timing jitter of 1.5 ps (FWHM) and 15% intensity fluctuations of the pump pulse intensity, a
temporal resolution of around 20 fs (peak-to-peak) and a dynamic range of 100 dB was achieved.
Our method has high potential to provide few fs level synchronization on next generation large
scale X-ray photon sources based on high repetition rate electron accelerators such as LCLSII. A
demonstrator aiming at operation up to 4.7 MHz is under development for the European X-FEL
within the frame of a recently granted EU project within the HORIZON2020 program.

X-ray structural analysis

MECHANISMS OF COMBUSTION AND STRUCTURE FORMATION IN SHS- SYSTEMS WITH PARTICIPATION
OF TWO AND MORE CHEMICAL REACTIONS

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The mechanisms of combustion and structure formation in perspective systems Ta-Ti-C, Mo-
Si-B, Ta-Zr-C, Zr-Si-B, Ti-C-Ca3(PO4)2, Cr-Al-Si-B, Si-C-B were well characterized using a
combination of various techniques including dynamic XRD, stop combustion front, SEM, TEM,
Raman spectroscopy, etc. It was established that gas transport reactions is to control the
combustion in some cases. In system (100%-X)(Ti+0.5C) + X(Ta+C) with X= 10 and 30% an
abrupt increase of Uc and Tc occurs as a result of the transfer from the splitting to merging
mode, which is accompanied by an increase in heat release as a result of two parallel chemical
reactions. In the case of X= 50%, dependences Tc(T0) and Uc(T0) are linear over an a wide
range of T0. The following processes are defined the SHS for Si-rich Mo-Si-B compositions: silicon
melting, its spreading over the surfaces of the solid Mo and B particles, followed by B dissolution
in the melt, and formation of intermediate Mo3Si-phase film. The subsequent diffusion of silicon
into molybdenum results in the formation of MoSi2 grains and molybdenum boride phase forms
due to the diffusion of molybdenum into B-rich melt. The formation of MoB phase for B-rich
compositions may occur via gas-phase mass transfer of MoO3 gaseous species to boron particles.
The stages of chemical interaction in the combustion wave are also investigated. The obtained
results indicate the possibility of both parallel and consecutive reactions to form molybdenum
silicide and molybdenum boride phases. Thus the progression of combustion process may occur
through the merging reaction fronts regime and splitting reaction fronts regime. Molybdenum
silicide formation leads the combustion wave propagation during the splitting regime, while
the molybdenum boride phase appears later. Kinetics of the SHS process, stages of chemical
transformations and structure formation of ceramic materials in the Cr-Al-Si-B system were
investigated. The effect of green mixture composition and initial temperature on the combustion
rate Uc and combustion temperature Tc, which reduce with increasing Al content, was studied.
An increase in the initial temperature of the SHS process causes a linear increase of Uc and Tc
in the range of T0 = 290-750 K. This is a fact that each composition is characterized by the
similar combustion mechanism, when the stages of chemical reactions of product formation remain
unchanged. However, an increase in T0 above 750 K, probably, may lead to exponential character
of Uc growth. Furthermore, an increase in Al content increases the proportion of the Al-Si
eutectic melt. The stages of chemical transformations and the mechanism of structure formation
in the combustion wave were studied. Dependences Tc(T0) and Uc(T0) of mechanically activated
(MA) Ta-Zr-C exothermic mixtures were determined. The self-heating phenomenon is observed in
argon atmosphere at T0 > 380 K due to zirconium particles oxidation by adsorbed oxygen. ZrO2
was formed in the combustion zone at the initial stage of chemical interaction; it is subsequently
transformed into ZrC. TaC was formed in the combustion zone, while the single-phase (Ta, Zr)C
with the lattice parameter of 0.4479 nm was formed closer to the post-combustion zone. Kinetics
and mechanism of SHS process, stages of chemical transformation and structure formation in
systems Zr-Si-B, Ti-C-Ca3(PO4)2, Si-C-B were also discussed.

Poster Session
Beam parameter measurements and demonstration of UED at the KAERI ultrashort pulse facility

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We developed an ultrashort pulse facility for pump-probe experiments at the Korea Atomic Research Institute (KAERI). This facility composed two ultrafast electron diffraction (UED) experiments for gas target and solid target. We measured beam parameters and demonstrated UED. We will present on results of beam parameter measurements and UED experiments.

Poster Session

Analytical study of terahertz spoof surface plasmons on corrugated metal-dielectric structures

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Results of analytical and numerical analysis of characteristics of spoof surface plasmons on corrugated metal-dielectric structures in terahertz region will be presented.

FEL-based study and THz radiation application

Terahertz surface plasmons on real metal-dielectric structures: comparison of theory and experiments

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Co-author(s): Prof. KNYAZEV, Boris; Prof. NIKITIN, Alexey; LEMZYAKOV, Aleksey; Ms. BULGAKOVA, Vladislava

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Terahertz surface plasmons propagated along real metal-dielectric structures, as well as their propagation through the macroscopic air gaps between two conducting surfaces, have been studied. The experimental results obtained using terahertz radiation of Novosibirsk free-electron laser and comparison with theory will be presented.

Poster Session

Interaction of monochromatic terahertz surface plasmons with plane mirrors

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Co-author(s): Prof. KNYAZEV, Boris; Prof. NIKITIN, Alexey
Experimental results of study of reflection and diffraction of surface plasmons generated using monochromatic terahertz radiation of Novosibirsk free-electron laser will be presented.

X-ray structural analysis

Methods of angular scanning in imaging and topography

Author(s): Dr. PODURETS, Konstantin
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In an imaging experiment using the topography or phase contrast techniques, generally, the result is a single image, which represents the internal structure of the object. However, identification of the features of the observed structure is a challenge. In these methods, an important parameter is the scattering angle, and methods of using its variation are being actively developed [1,2], making possible to obtain a scattering curve for each local region of a sample. The report discusses the results of applying the methods of the angular scanning experiments in the synchrotron topography and analyzer based imaging. The curved silicon single crystals are currently used at the big accelerators (LHC etc) as deflectors for the extraction and collimation of the proton beams. Using the method of topography with angular scanning at synchrotron radiation such a deflector was studied, the curvature of its elements was measured and its performance at the passage of the proton beam was understood. A wide range of objects was studied with the help of analyzer based imaging with angular scanning. Several samples of opals of natural and artificial origin were investigated. Images of all the samples were obtained in a wide angular range from the peak of crystal reflection to the distant tail at about 102 of FWHM. The majority of the samples displayed the inhomogeneous structure with regions that changed their brightness, relative to adjacent regions, during the rotation of the analyzer crystal. Another kind of a sample was an ancient parchment, where the features of its structure on tens micrometers scale can provide a valuable information of the parchment manufacturing technology.


FEL-based study and THz radiation application

Synchrotron radiation X-ray tomographic microscopy, noble gas mass spectroscopy, infrared and Raman microscopy correlated study of the Itokawa asteroid particles returned by the Hayabusa space probe

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The analysis of asteroid’s surface grains allows to match remote observations and modeling of its history and current properties with laboratory experiments. Mass spectrometry and electron microscopy of the dust enables detailed study of the history of asteroidal regolith and compare the results with theoretical considerations of the evolution of rubble piles in the inner solar system. A correlated study including consequential Raman and infrared microspectroscopy, synchrotron radiation X-ray tomographic microscopy (SRXTM) and noble gas mass spectroscopy has been implemented for the determination of cosmic-ray exposure ages and trapped Xe content in a few particles returned by the JAXA’s Hayabusa space probe from the near-Earth asteroid 25143 Itokawa, the first successful sample return mission to an asteroid. New data support the uniformly short exposure to cosmic rays of the Itokawa regolith of <8 Ma suggesting a freshly rejuvenated regolith.

FEL-based study and THz radiation application

Raman scattering at terahertz frequencies enabled by an infrared free electron laser

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In the last decade the use of infrared free electron laser facilities enabled observation of inelastic light (Raman) scattering in THz frequency range. Raman-active intracenter donor transitions in silicon fall into the THz range and serve as outgoing resonances in electronic Stokes scattering. At photon fluxes above 1E24 photon/cm²/s donor-related Raman stimulated emission occurs in the range 4.2-6.5 THz from natural and isotopically enriched silicon crystals with various dopants while the free electron laser wavelength was varied between 18 and 41 mkm (7.5-16.5 THz). Study of dynamics of the observed emission shows a transient picosecond-micropulse mode that indicates on significantly larger Raman gain realized in THz Raman silicon lasers. This research has been...
partly supported by the EC CALIPSO project for the Transnational access to the European FELs and Synchrotron facilities as well as joint German-Russian program "Research on technological advances of radiation sources of photons and neutrons based on accelerators and neutron sources in cooperation with research organizations and universities of the Federal Republic of Germany" (InTerFEL project, BMBF No. 05K2014 and the Russian Ministry of Science and Education (No. RFMEFI61614X0008).

X-ray structural analysis

Zero-area THz optical pulses in gases.

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Transformation of the short optical pulses in a medium with a high optical density is described. In such conditions an intense free-induction signal is formed, which has a specific type of the oscillations. These oscillations were observed experimentally in rotation spectra of HCN and HBr molecules using a terahertz free electron laser as a source of optical pulses and ultra-fast Schottky diode as detector.

Poster Session

XANES investigation of the chromium dichalcogenides CuCr_{1-x}M’S_{2} and MCrX_{2}

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Transformation of the short optical pulses in a medium with a high optical density is described. In such conditions an intense free-induction signal is formed, which has a specific type of the oscillations. These oscillations were observed experimentally in rotation spectra of HCN and HBr molecules using a terahertz free electron laser as a source of optical pulses and ultra-fast Schottky diode as detector.

FEL-based study and THz radiation application

Formation of nanosized metal hydrosols 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 metal containers used in the experiment. Fractional and elemental composition of the particles was determined by atomic force (AFM), scanning electron microscopy with elemental analyzer (SEM EDAX), and the results of mass spectrometry with inductively coupled plasma (ICP-MS). 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.

FEL-based study and THz radiation application

High temperature quasi stationary terahertz optical discharge on NovoFEL

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Threshold conditions, plasma parameters, autooscillations and their stabilization in terahertz optical discharge on NovoFEL are considered in the report.

FEL-based study and THz radiation application

Single-pulse high-resolution spectroscopy on NovoFEL: methods, applications and development

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Methods of single-pulse high-resolution spectroscopy based on powerful tunable radiation of terahertz NovoFEL, ultra-fast technique, and strong magnetic field of superconducting solenoid are presented in the report.

X-ray structural analysis

Synchrotron radiation methods for registration of the ejection of particles from the free surface of shock-loaded metals

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When a strong shock wave leaves a metal, flows of particles of different sizes are ejected from the wave’s free surface (shock-wave "dusting", ejecta) [1,2]. Unlike a cumulative jet, such a flow consists of separate particles having a size of a few microns to hundreds of microns. It was assumed that there are also finer particles in such a flow, but the existing techniques are not able to resolve them.

This report presents the results of experiments using SR from the colliders VEPP-3 and VEPP-4 at BINP. Precision measurement of transmitted SR (of an energy of 2 GeV on VEPP-3) was applied to exploration of microparticle flows from a free surface of various materials (copper, tin, and tantalum). Mass distributions along microjets originating from micron-sized slits were obtained. Dynamic detection of small-angle X-ray scattering (SAXS) of synchrotron radiation (SR) from the collider VEPP-4M (energy of 4 GeV) was implemented on the facility SYRAFEEMA (Synchrotron Radiation Facility for Exploring Energetic Materials). A technique of SAXS measurement on this facility enables detection of nanoparticles ranging in size from 4 to 200 nm. Flows of nanoparticles of about 100 nm in size from a surface of smooth foil (tin and tantalum) affected by compressed HMX were detected for the first time.

X-ray apparatus

Current driven wire based magnetic measurement systems

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Manufacture of magnetic devices like wigglers and undulators requires magnetic measurements. There are well-known Hall probe and moving wire based methods. This paper describes current driven wire based method. A thin nonmagnetic wire is strained within the wiggler. It is secured and tighten on supports located in some distance from the wiggler. Wire position sensors are located on the wiggler sides. Current applying to the wire deflects it according to the magnetic field inside the wiggler. There are a number of current modes giving different information. Constant current method gives 1-st and 2-nd magnetic integrals, median plane position, sextupole integral. Vibrating current method (multiple by wire self-resonant frequency) gives any integral with higher sensitivity. Pulse wire method is alternative to Hall-probe method.

Poster Session

Vibrating wire magnetic field measuring technique

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Vibrating wire magnetic field measuring technique is a kind of current driven wire based magnetic measurement system. Harmonic current is applied to thin wire strained within the wiggler. The current frequency is multiple by wire self-resonant frequency. If the magnetic field integral is
non-zero, the wire vibrates. Wire position sensors measure the deflection. The technique is very sensitive and allows to measure many integrals simultaneously.

**Poster Session**

**Pulsed wire method for magnetic field measurements**

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Pulsed wire method is a kind of current driven wire based magnetic measurement system. A current pulse is applied to thin wire strained within the wiggler. Wire position sensors measure the deflection. This method may take the place of Hall-probe method, because it shows magnetic field characteristics in every point inside the wiggler, not only integral characteristics. And it requires only few milliseconds for measurement.

**Poster Session**

**System for diagnostics of local electron beam losses in microtron-recuperator at Novosibirsk Free Electron Laser beamline via registration of induced X-rays.**

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The Novosibirsk Free Electron Laser (FEL) is based on a 4-turn microtron-recuperator. To ensure its stable operation and radiation generation, it is necessary to provide a stable mode of electron beam recirculation and minimize beam losses on the vacuum chamber wall on all the way of beam in the accelerator beamline. To fulfil this task it is necessary to know the longitudinal distribution of these losses along the beamline. To this end, a system for registration of beam losses was created. The system applies optical fibers placed along the full length of the vacuum chamber and nearby it. In case of local electron beam losses somewhere in the vacuum chamber, electrons falling to the vacuum chamber wall cause generation of X- and gamma rays. This radiation in turn causes generation of optical radiation in the optic fiber nearby the region of electron "precipitation" on the chamber wall. Then, executing some transformation and processing of the time dependencies of signals from these optic fibers, one can obtain the longitudinal distribution of electron beam losses along the whole accelerator channel in all its 4 turns.

**Radiation sources and Centers**

**Accelerator development at NSRRC-from the first 3rd-generation light source in Asia to a low-emittance photon source**

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Taiwan Light Source (TLS) was the first third generation light source in Asia and served to users in 1993 with 1.3 GeV beam energy and 200 mA design current. The requests for higher
photon energy and flux emerged from x-ray users’ community. After series upgraded, TLS has the capabilities running in top-up injection with beam energy increased to 1.5 GeV and 360 mA of beam current. A six-fold symmetry TLS storage-ring has 9 insertion devices installed, which included 5 Super-Conducting (SC) wigglers and 4 conventional wiggler and undulators. In order to improve the instabilities threshold and the photon flux, higher-order-modes (HOM)-free SC cavity was installed to replace primitive Doris cavities in the ring. Academic and operation statics will be presented.

Taiwan Photon Source (TPS) was designed to have extreme low emittance, 1.6 nm-rad., to push the x-ray brightness into world frontier in ring-based facilities. The ring is DBA lattice with 3 GeV beam energy and 500 mA of design beam current in 24-cells. Top-up injection with HOM-free SC cavities are the target mode and power sources for day-one operation. The design, construction and commissioning results will be addressed in this presentation.

Poster Session

Small angle X-ray scattering on RE31 aptamer

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This work shows aptamer RE31 (5′-GTGACGTAGGGTTGGTGTGGTTGGGGCGTCAC-3′) by small angle X-ray scattering (SAXS) measuring results, wich obtained on DIKSI station in the National Research Center ‘Kurchatov Institute’. Also the aptamer structure model is presented, wich was processed in DAMMIN software. According to obtained model, the molecular simulation was constructed with known nucleotides considering interatomic interactions. We have compared aptamer model from SAXS with crystallographic method and confirmed, that SAXS can provide us the information about the size and native shape of bioaptamers in solution. However SAXS method doesn’t require the crystal growing and high concentrations for measuring.

Radiation sources and Centers

Superconducting 72-pole indirect cooling 3Tesla wiggler for CLIC dumping ring and ANKA image beamlne

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One of the directions of BINP activity is the creation of multipole superconducting wigglers with the magnetic field levels from 2 T to 7.5 T which are installed on many SR sources. Special efforts were made by BINP to develop the cryogenic system with zero liquid helium consumption. The next significant step became the design and creation of superconducting full size prototype of dumping wiggler for CLIC project where supposed the installation of 104 wigglers. For operation of CLIC dumping wiggler it is required the magnetic field of 3 T and the period about 50 mm with a beam vertical aperture of 13 mm. Design features of the wiggler which was proposed and created by BINP is the application of the indirect cooling method. In this case Nb-Ti magnet with the length of 1.8 m and the weight of 700 kg is located in a vacuum and is cooled by four Gifford-McMahon cryocoolers. To maintain the temperature about 4.2 K on the magnet it is
used the tubes with circulating liquid helium as the heat conducting elements. To increase the efficiency of pre-cooling down of the magnet it is used nitrogen-based heat pipes of siphon type. The features of the magnetic and cryogenic systems of CLIC dumping wiggler full size prototype and test results are presented in this article.

Radiation sources and Centers

Progress in the design and related studies on the High Energy Photon Source

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The High Energy Photon Source (HEPS), a kilometre-scale diffraction limited storage ring light source, with energy of 5 to 6 GeV and emittance of a few tens of pm.rad is to be built in Beijing and now is under design. In this paper we reported the progress of the physical studies for the HEPS project, covering issues of storage lattice design and optimization, resonance effect evalution, booster design, injection design, collective effect study, error study, etc.

Influence of terahertz laser irradiation on the morphology of chicken hepatocytes

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Previously, we have discovered the erythrocyte membrane disruption in aqueous solution under the influence of high-power pulses of the Novosibirsk free electron laser operated at the wavelength of 120-170 microns, with the average power density of 10W/cm2. This fact was explained by the emergence of powerful ultrasound caused by water absorption of terahertz radiation. In this report hepatocytes were studied under the same conditions. The aim of the study was to evaluate the effects of radiation on normal hepatocytes in comparing with previous studies of red blood cells. Liver fragments were sampled and transferred into 500ul of aqueous suspension and irradiated with non-focused beam at different exposure time. Samples were examined histologically using the atomic force microscopy (tapping mode) in the native form. Exposure of 2-4 seconds resulted in single membrane injuries and a slight change in the shape of hepatocytes. Exposure of 5-10 seconds or more leads to significant damage of hepatocyte membranes. Results have fully confirmed the earlier experiments. Destructive effects of high-power THz pulses on biological objects in the aquatic environment were proven.

Biomedical application of SR and THz radiation

Study of the THz response of protein solutions at different stages of glycation

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Protein glycation is accelerated under hyperglycemic conditions resulting in loss of the structure and biological functions of proteins. This process plays a significant role in diabetic complications in organism tissues. THz spectroscopy is a unique tool that can be used for analysis of biological tissues and solutions because changes in relative proportions of free and bound water and in relaxation times for either of these states can all be observed in THz range. A distinctive feature of this method is the possibility of measuring directly the refractive index, absorption coefficient, and hence complex permittivity spectrum of the sample in a single scan and in a broad frequency range. The THz time-domain spectroscopy has been used for measuring of bovine serum albumin (BSA) and monosaccharides solutions and studying of glycation dynamics for 0-96 h incubation BSA with sugars. To detect small-scale changes in solutions and to increase the significance of observations, we have performed measurements using both transmission and attenuated total internal reflection (ATR). By combining the results obtained in both configurations, the reliable range of the spectrum can be considerably broadened (0.05-2.5 THz). The THz time-domain spectrometer used in the study was described previously [1]. The Fourier transform of the measured pulse time profile provides the complex spectrum comprising information on the refractive and absorption indices of the medium where the pulse has been reflected or transmitted. To recalculate the transmission spectrum into the absorption and refraction spectra, we used the Fresnel formulas and the methods described in [2]. We analyzed the reasons for the THz transmission changes of BSA and monosaccharides solution during incubation. The differences were analyzed by comparing experimental spectra to the model dielectric function of water. THz absorption coefficient of glycated albumin solution increases during first several days of incubation. Fructose demonstrated stronger and/or faster influence on THz absorption of BSA than glucose. We consider that glycation of BSA results in a change the parameters of slow Debye relaxation. These results show that THz spectroscopy is a useful tool for study of protein glycation in time.

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**EXPERIENCE OF USE OF X-RAY ENERGY-DISPERSIVE AND MONOCHROMATIC DIFFRACTOMETRY FOR STRUCTURAL RESEARCHES OF BIOLOGICAL SYSTEMS USING SYNCHROTRON RADIATION**

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The important role in the study of the structural mechanism of the functional activity of biological systems belongs to the instrumental and methodical developments intended for the energy-dispersive (\(\lambda=\text{const}\)) and monochromatic (\(\lambda=\text{const}\)) diffractometry with synchrotron radiation (SR) usage. Results of our long-term developments are considered. At one method the collimated X-ray beam of polychromatic radiation ("white spectrum") is directed on the object, and the diffraction pattern represent the intensity distribution as function of a wavelength at the fixed scattering angle (\(\theta\)), i.e. lattice-plane spacing of object are uniquely determined by a discrete set of the photon energy. Note the spatial resolution of different zones of the X-ray pattern can...
be changed by varying $?\text{=}\text{const}$ value that allowed to record weak reflections and to register X-ray pattern with high resolution for material with large spacings of identity. The $?\text{=}\text{const}$ method determines by dedicated monochromatic wavelength from continuous SR spectrum, and then the X-ray beam is collimated by X-ray optical zoom lenses; obtained diffraction patterns demonstrate the intensity distribution as a function of scattering angle. The $?\text{=}\text{const}$ method provides increasing intensity of the X-ray beam on 2-3 orders, however earlier during creation of the first SR sources these advantages were unattainable because of under quick-action of the detection system. The $?\text{=}\text{const}$ method is traditionally used with position sensitive coordinate detectors.

Now power-dispersion detectors with high performance of registration and energy resolution are developed, therefore it is very inviting to use polychromatic radiation. Various X-ray diffraction stations created by us (KEMUS, FRAKS, DICSI) on the operating channels of the storage rings VEPP-3, VEPP-4 (Siberian Center of Synchrotron and Terahertz Radiations, SCSTR, Novosibirsk) and Siberia-2 (Kurchatov Center of Converging of Nano-, Bio-, Information-, and Cognitive Sciences and Technologies, NBIC, Moscow) are presented. This equipment based on block-modular configuration, i.e. the main devices of stations could be used as independent modules for any experimental scheme. Despite different character of current tasks, a many things in the experimental technique were unified, for example, such blocks as monochromatic and reflecting zoom lenses, collimators of the primary beam, and also the vacuuming device, remote control of the optical system components and software. Results of the SAXS/WAXS diffraction investigations of biological tissues at different physiological states are considered. There are presented also the first experimental results of X-ray diffraction study of test objects (collagen and muscle) received by the $?\text{=}\text{const}$ method with using SR of the VEPP-3 storage ring. The work is supported by the RFBR grant # 14-44-03667.

Poster Session

Study of Electronic Structure and Magnetic Properties of Manganese Sulfide Solid Solutions Doped With Rare Earth Elements

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Solid solution containing chemical rare-earth elements (such as Yb or Tm) have a number of unique properties, which is commonly undergo a phase transitions having a purely electron nature and related with the change in filling of 4f electron levels [1, 2] along with magnetic-nonmagnetic state transitions [3]. These systems are also possess thermoelectric properties. Invention of a highly efficient thermoelectric materials is one of the important direction of modern materials science. One of the directions on finding of new thermoelectric materials is the study of systems, in which metal-dielectric transition is observed. The systems of such kind could be solid solutions based on the transition metal sulfides Mn1-xMxS (M=Cu, Fe, Co) and possess high values specific of thermoelectric power [4-5]. Electrophysical properties of such functional materials is primarily determined by features of its electronic structure. In this regard, current work involves a comprehensive study of the interrelationship between electronic structure and magnetic properties (i.e. magnetic susceptibility) of the Mn1-xLnS (Ln=Dy, Tm, Yb; x=0;0.01;0.05). X-ray absorption near edge spectroscopy (XANES) provides information about electronic structure (K-absorbtion spectra of Mn, S; L-spectra of lanthanides). Measurement of the chemical shifts of (Mn, S)K1,2-lines and ratio of MnL1,2-,L3- emission spectra allows to determine the charge and the electron density of metal and sulfur atoms in investigated solid solutions. In order to investigate electronic structure XAS and XES spectra figured in unified energy scale. Experemental spectra were compared with quantum chemical calculations in the FDMNES and ADF BAND software packages.

A set of algorithms and procedures of a crystal loop centering during X-ray high-throughput protein crystallography experiment has been designed and developed. A simple algorithm of the crystal loop detection and preliminary recognition has been designed and developed. The crystal loop detection algorithm is based on finding out the crystal loop ending point (opposite to the crystal loop pin) using image cross section (digital image column) profile analysis. The crystal loop preliminary recognition procedure is based on finding out the crystal loop sizes and position using image cross section profile analysis. The crystal loop fine recognition procedure based on Hooke-Jeeves pattern search method with an ellipse as a fitting pattern has been designed and developed. The procedure of restoring missing coordinate of the crystal loop is described. Based on developed algorithms and procedures the optimal autocentering procedure has been designed and developed. A procedure of optimal manual crystal centering (Two Clicks Procedure) is described. Developed procedures have been integrated into control software system PCCS installed at crystallography beamlines Photon Factory BL5A and PF-AR NW12, KEK.

Process of gelation and thermal decomposition at different temperatures of the Zr/Ti sol-xerogels, chemically identical but prepared by different methods (mixed and binary), was studied by SAXS/WAXD with the use of SR. Primary particles in mixed and binary Zr/Ti sol-xerogels have different structure. Using the qualitative contrast variation method it was concluded, that primary particle in mixed system consists of Ti nuclear surrounded by Zr atoms in the outer layer of the particle. In contrast, primary particle in binary system consists of Zr nuclear surrounded by Ti atoms in the outer layer of the particle. Multistage thermal decomposition of the Zr/Ti xerogels proceeds through an intermediate phase formation. In the case of the mixed Zr/Ti xerogel the intermediate phase has a distorted (amorphous) structure of anatase (TiO2) which is formed, probably, inside the primary particle of the xerogel. In this case, during the thermal...
decomposition the interatomic distance in the intermediate phase is similar to the interplane
distance of metal planes in final product (crystalline ZrTiO4 powder). In the case of a binary
Zr/Ti xerogel, the intermediate phase has an amorphous structure consisting, probably, of the
disordered metal atoms (probably, in an oxide state) surrounded by water molecules.

Radiation sources and Centers

Fabrication of high effective power silicon diffractive optics of terahertz range by femtosecond laser ablation of silicon surface

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The emergence of coherent, high power sources of THz radiation [1] has formed a need for optical
elements to control this radiation. It is known that high-power THz beams can be controlled by
silicon diffractive optical elements (DOEs) [2,3,4]. The lithographic etching of a silicon substrate
[2,3,4] has disadvantages: fabrication of multi-level elements by lithographic etching requires an
expensive and complicated procedure of photomask alignment, and binary (two-level) elements
have limited energy efficiency. A possibility of the microfabrication of a high-effective power
silicon diffractive optics for terahertz range by laser ablation is investigated [5]. A four-level
silicon diffractive THz Fresnel lens has been fabricated for the first time by femtosecond laser
ablation [5]. High repetition (f = 200 kHz) femtosecond Yb:YAG laser was used for the formation
of micro-relief at the silicon surface. Features of the lens were investigated in the beam of the free
electron laser at the wavelength of 141 mkm. The measured diffusive efficiency of the lens is in
good agreement with the theoretical prediction [5]. The experiments have shown the feasibility
of laser ablation of the silicon surface to fabricate effective multilevel THz DOEs. Improving
the proposed technology of forming a silicon microrelief, in particular increasing the number of
its quantization levels, will eventually increase the energy efficiency of silicon DOEs designed to
control beams of THz radiation.

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933-936.
Poster Session

The research of the superconducting undulator prototype with neutral poles and features of the magnetic field distribution in it.

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The article describes superconducting undulator prototype with neutral poles designed and fabricated in Budker Institute of Nuclear Physics. In the upper half of the undulator even-numbered poles have a core with a winding and odd - core without winding. In the lower half vice versa. This design allows to provide more accurate period and reduces the probability of mechanical movements caused by the action of ponderomotive forces leading to quench of superconductivity. The article also describes the features of the magnetic field distribution in such windings configuration.

In situ determination of the active phase in palladium nanocatalyst by X-ray absorption spectroscopy and X-ray diffraction

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Synchrotron and Free electron laser Radiation: generation and… / Book of Abstracts

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After severe plastic deformation under the high pressure torsion technique a number of brittle refractory metals such as Mo, W, Re and Ni₃Ge intermetallic compound with L1₂ structure have been investigated. These studies are of great interest, since the effects of shear deformation under high pressure have not been previously studied on these materials. In all cases the grain refinement caused by dislocations mobility has been observed. The Ni₃Ge demonstrates the decrease of the degree of the long-range order after large plastic strain. Synchrotron radiation source has been used in order to determine the level of internal stresses, crystallite size and other parameters of the plastically deformed materials. The models of the influence of the mobility of dislocations to crystallite size and fracture surfaces have been considered. The reported study was partially funded by RFBR according to the research project No. 16-03-00182-

Biomedical application of SR and THz radiation

Nanoradiator therapy and synchrotron X-ray imaging of malignant brain tumor

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Glioblastoma malformation is typical intractable malignant brain tumor that has less than 2 years as mean survival due to infiltrative nature in conjunction with microvascular malformation (MVP). Conventional drug delivery is limited by intact BBB in normal tissue surrounding MVP, and current radiotherapy is not indicative due to infiltration of tumor cell into normal brain. Therefore, tumor-infiltrating MVP should be therapeutic target. Recently we imaged MVP with tumor infiltration in rat glioma model using synchrotron X-ray DEI CT technique. High-Z nanoparticles play a role as nanoradiator, burst release of low energy electrons and characteristic X-ray fluorescence, when subject to X-ray photoelectric ionization or high-energy ion beam-irradiated Coulomb scattering. During last decade we have developed dosimetry of nanoradiator effect and medical application of novel site-specific nanoradiator therapy using either monochromatic synchrotron X-ray or traversing Bragg-peak proton beam. Eventually nanoradiator therapy provide a new platform of therapeutic nanobeacon technology by converting prodrug inert gold/iron nanoparticle into electron-emitting drug. Realization of this concept in oncology and non-oncology would be demonstrated in this presentation. In addition, high-resolution X-ray diffraction CT imaging of skull-based brain is challenging and discussed with our resolving efforts.

X-ray structural analysis

The high-pressure diffraction studies of potassium and complex carbonates: structural trends and stability

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The interest to carbonates is determined by their obvious practical importance, as well as their abundance in the Earth's interior and a key role in magma generation, plume upwelling and diamond growth [1]. Despite the structural trend of the high-temperature phases of all alkaline carbonates is well understood [2], almost nothing is known about high-pressure behavior of these compounds. In this work we use a combination of the high-pressure diffraction experiments in diamond anvil cell (4th beamline of the VEPP-3 storage ring of the SSTRC, Novosibirsk) and ab-initio calculations to determine the crystal structure of the high-pressure phase of K2CO3 at 3.1 GPa. Among the structures founded in our calculations, the $\text{U+FFFD}$ structure shows relevant matching with the experimental pattern (Fig. 1). The comparison with the experimental data on Li2CO3 allows to reconstruct the common structural trend, which is consistent with the simple rule that the structure of the high-pressure polymorph is the same as the ambient structure of a heavier element compound from the same group of the periodic table. We also report on the compressibility and structure behaviour of thaumasite Ca3Si(OH)6(CO3)(SO4)12 [U+FFFD] [U+FFFD] a unique known mineral that possesses Si coordinated by six hydroxyl groups and stable at ambient P/T conditions [3]. Our data show a strong dependence of its high-pressure stability limit on the nature of compressing medium. This work is supported by the Russian Foundation for Basic Research (grant No 15-55-45070) and the Ministry of Education and Science of Russian Federation (# 14.B25.31.0032).


Poster Session

SRXRF STUDY OF CHEMICAL ELEMENTS CONTENT IN THE Atherosclerotic PLAQUE OF HEART VESSELS

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The main problem of cardiology, that society faces, is the acute heart failure (AHF). Many factors have effect on the development of cardiovascular diseases. The primary reasons of myocardial infarction and stroke are hypertension and sclerosis that lead, with time, to calcification and contraction of arteries which prevents blood to enter heart and brain. In heart vessels an atherosclerotic plaque forms which closing a vascular lumen, causes IDH, and myocardial infarction. Therefore, studying the deep mechanisms of plaque formation is the focus of attention. The atherosclerotic processes in heart vessels may be explained by several theories that account for the complex processes of atherogenesis. Many papers are devoted to the cellular composition of plaques. Little, however, is known about the element composition of the atherosclerotic nidus of coronary vessels. Elucidating the content and ratio of chemical elements upon plaque formation in heart vessels may substantially supplement the missing stages of this process. The goal of this study is to determine and estimate a complex of chemical elements in the substrates of the atherosclerotic nidus of human heart vessels. The method of X-ray fluorescence analysis with synchrotron radiation (SRXRF) (VEPP-3, store ring) was used in Center “Siberian Synchrotron and Terahertz Radiation Center (SSTRC)” to study the content of chemical elements in vascular walls and in atherosclerotic plaques (stable and unstable) drawn from men after operative therapy. As the amount of material for studying is limited, we have developed a special technique for preparing the samples of vascular and plaque tissues. We measured the concentrations of K, Ca,
Cr, Mn, Fe, Co, Ni, Cu, Zn, Br, Sr, Zr, and Pb. A comparison was made of the content of elements in the tissues of heart vessels with atherosclerotic plaques and of the element compositions of stable and unstable plaques. In all the samples studied, prevailing is Ca, particularly in unstable plaques. The concentrations of K, Ca, Cr, Mn, Fe, Ni, Zn, and Br in atherosclerotic plaques are much higher than in the vascular tissues of heart. At the background of predominant Ca no reliable difference has been revealed in the elements measured in stable and unstable plaques. A high association of Ca with Fe, Zn, and Sr has been recorded in the atherosclerotic plaques. Thus, the SRXRF method has first allowed one to determine the multielement composition in the substrates of the atherosclerotic nidus of human heart vessels.

Poster Session

Superconducting solenoid for superfast THz spectroscopy

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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 developed for this method. Its design and peculiarities are described in this presentation.

Poster Session

X-ray diffraction data for the study of the multilevel nanostructures in Ni3Fe deformed single crystals

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X-ray analysis has been applied for the study of the formation of nanostructures as the result of large plastic deformation in Ni3Fe single crystals ordered alloy with the L12 structure. It was shown that the main structural changes in ordered alloys had the features similar to pure metals and the disordered solid solutions. However, in the ordered Ni3Fe single crystals the formed nanostructure was more complex. Structure consists of nano-crystallites divided by the antiphase boundaries forming the elements having size a ten times smaller than the dimensions of the crystallites. The antiphase domain structure was not homogeneous. After the strain of 0.18 together with the domains, the average size of which is equal to the original 14 nm, there were revealed a small fraction of domains with an average size of 10-11 nm. After the strain of 0.38 or more, the average size of the antiphase domains was reduced to 5-2 nm. However, in the material, a small amount of antiphase domains whose size was close to the original (13 nm) also was revealed along with the small domains. This indicates that the deformation was carried out inhomogeneously and there remained the sites of material almost unaffected by deformation processes. Analysis of the research points to the emergence of multilevel nanostructure with a clearly expressed of the two scale levels. The reported study was partially funded by RFBR according to the research project 16-03-00182- [U+FFFD]
Poster Session

The X-FEL quadrupole with gradient 100 T/m

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BINP

Abstract - The paper describes X-FEL quadrupole with gradient 100 T/m. These quadrupole magnets are designed for installation in the free space between the XFEL undulators. In order to maintain the high quality of the electron beam at an energy of 10 - 20 GeV, requiring high stability of the magnetic axis of the lens. The available size of the aperture of the lens is R = 8 mm. Stability of the quadrupole magnetic axis better than 5 um for 10% of a gradient range, good quality of the magnetic field $\approx 10^3$.

Poster Session

The application coated NEG as main pumpdown facility in the narrow extensive chambers of the particle accelerators

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Abstract - The paper describes in detail the design and manufacturing of prototypes with coated non-evaporable getter (NEG) as main pumpdown for NSLS-II third generation synchrotron light source and for Coherent electron Cooling of eRHIC. TiZrV NEG sputtered film in the narrow extensive chambers is a vacuum technology for obtained ultra-high vacuum which is used in many particle accelerators worldwide. The experimental results of NEG film sticking probability for hydrogen are presented here.

Invited Talks

My profession is using of synchrotron radiation in the structural biology of tissue

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In this report the results of long-term works - more than 40 years - in the field of structural biology of tissue with synchrotron radiation (SR) usage will be presented. We were pioneers in the field of SR use for nanostructural researches of biological tissues. C.S. Holmes was the first to demonstrate the advantages of SR in studying the structure of biological objects in 1971 on the DESY station (Hamburg). We began to conduct systematic experimental works with SR in 1972. At that time, there were only two accelerators available for use as sources of SR in the X-ray range: the ARUS synchrotron at the Yerevan Institute of Physics, and the VEPP-3 storage ring at the Budker Institute of Nuclear Physics in Novosibirsk, which is still a world-class experimental SR site. The creation and development of a method for high-speed small-angle X-ray diffractometry must be considered a fundamental methodological success of this period, since the minimum exposition time was reduced to a fraction of a second. Diffraction cinema for the first time has been created by the large team of scientists from Novosibirsk and Pushchino, which for many years ranked ahead of other laboratories abroad. Last decade works are conducted on
the small-angle stations DIKSI (Siberia-2, NRC Kurchatov institute, Moscow). SR is promising for application in structural biology. Specific features of SR (relatively high intensity, broad radiation spectrum, and coherence) allow us to obtain structural data on objects whose sizes range from fractions of a nanometer to several centimeters. In living systems, we can study the functional range from single molecules to cells, tissues, and even organs, so that the object can be characterized comprehensively. The research was supported by RFBR-Moscow region Grant #14-44-03667.

Poster Session

Nanostructural study of human tumour transformed tissues by X-ray diffraction methods using synchrotron radiation

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The work is dedicated to investigating the nanostructural organization of proteoglycans in different physiological states of biological tissues of human and animals. Experimental samples of human tissues obtained during biopsies, surgeries and pathologoanatomic revisions; samples of breast cancer tissue were obtained from mammory gland division of Blokhins Onkological Scientific Center of Russian Ministry of Health. We used also unique collection of tumor transformed epithelial tissues from respiratory tracts of liquidators engaged in elimination of the disaster sequels on the Chernobyl A-plant obtained from Pulmonogical Research Institute. X-ray diffraction and fluorescence study of biological tissues with using synchrotron radiation were carried out on the channel K1.3 of the Siberia-2 storage ring at NRC Kurchatovsky Institute, Moscow (on the constructed and created by us small-angle stations DICSI) and on the channel 3b of VEPP-3 at INP SD RAS, Novosibirsk, and also on SAXS/WAXS station of European Synchrotron Radiation Facility at Grenoble, France. Small-angle X-ray patterns of epithelial tissue, both intact and pathologically transformed, display a large number of sharp diffraction rings at spacing of 4.5 nm and its higher orders caused by the proteoglycans of extracellular matrix of tissue. The period of identity at spacing of 4.65 ± 0.15 nm is attributed by us to regular attachment of olygosaccharide chains to the protein core of giant proteoglycan molecules [A.A.Vazina et al., NIM, 2005, A543, 297-301]. A correlation between the integrated intensity of X-ray patterns and elemental content of tissue is observed. It is experimentally shown that the structure of proteoglycans can be reversibly transformed by metal cations and chelating agents, and that calcium is the major element in the mineral composition of tissue. In cancer transformed tissues intensity of diffuse scattering and diffraction rings of 4.5 nm varies in a wide range and correlates with change of calcium content in the diseased tissues, a significant change in elemental content is detected, up to 200 times increase in the concentration of calcium is found. The final stage of disease is characterized by a significant increase in small-angle diffuse scattering; Debye rings are never registered. Treating the tissue with chelating agents such as EGTA and EDTA, in this case turns out to be ineffective, suggesting the irreversibility of the structural transformation of the extracellular matrix. Thus, the problem of cancerogenesis should be looked at in two aspects: investigation of the mechanisms of normal cell transformation which is initiated by DNA damage (mutations), and the stages of tumor progression, processes of malignant cell proliferation, invasion and metastasis which lead to numerous malignant tumors. X-ray study of tissues indicates that it may be possible to use diffraction characteristics of proteoglycan structures as novel markers of pathological transformation of tissues. The research was supported by RFBR-Moscow region Grant #14-44-03667.
Nanostructural ordering of epithelial tissue of silk gland of Antheraea mylitta silkworm

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X-ray diffraction study of epithelial tissue of silk gland of Indian domestic Antheraea mylitta silkworm was carry out on the small-angle station DIKSI of the Siberia-2 storage ring (NRC Kurchatov institute, Moscow). The silk gland is a unique pair organ of internal secretion in diameter 2 mm and length about 1 metre, that ten times surpasses the size of the larva of a silkworm. In an active phase of cocooning all three departments of a glan (anterior, middle, and posterior) are filled by jelly-like substratum from fibroin and sericin proteins which form a silk thread under the influence of mechanical forces (pulling) and specific geometry of keratin segment of gland. Samples of various departments of a silk gland have been allocated from a larva by soft preparation methods, and after that dried up at a room temperature under cargo. X-ray patterns of all departments of a silk gland are characteristic for epithelial tissues: in a range of 1-5 nm its show several Debay rings with the basic period of identity of 4.72 nm. In our earlier investigations of epithelial tissues of animals and human the series of diffraction Debay reflections at the spacing of 4.65 (± 0.15) nm were attributed to proteoglycan structures of extracellular matrix [A.Vazina et al.: NIM (2001) A470; NIM (2005) A543; NIM (2009) A603; Glass Phys. Chem. (2007) 33]. Thus, this periodicity is a nanostructural invariant of proteoglycan structures of epithelial tissues of animals, human, and insect also.

Poster Session

Deflection of THz vortex beam in non-polar liquids by means of acousto-optics

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Interaction of terahertz vortex beam and bulk acoustic wave in a number of non-polar liquids was investigated. It was found that hexane is the most promising liquid for acousto-optic applications. The diffraction efficiency for the vortex beam was of the same magnitude as for non-vortex beam.

Poster Session

The investigation of the aluminum nitride formation during the aluminum nanopowder combustion in air

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Synchrotron and Free electron laser Radiation: generation and... / Book of Abstracts

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The phase formation sequences, intermediate and final products of aluminum the nanopowder combustion are studied. It is found, that the main combustion product is aluminum nitride. In the combustion of aluminum nanopowder, aluminum \( \gamma \)-oxide is the first to form, and aluminum nitride is formed next. The formation of aluminum probably occurs by successive displacement of oxygen by nitrogen from the aluminum oxide. The use of sliding (incident at a small angle to the surface) synchrotron radiation made it possible to determine with high accuracy (in time) the sequence of stages of formation of crystalline products during combustion of the aluminum nanopowder.

Poster Session

In situ SR SAXS study of liquid intermediates arising during nucleation of gold sulfide nanoparticles in aqueous media

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Recently, using DLS, AFM (both, in situ and ex situ), STM and SAXS techniques, we found a series of fluid nanoscale intermediates preceding nucleation of gold sulfide in the reaction between aqueous HAuCl\(_4\) and sodium sulfide. In present contribution SR SAXS technique was applied to monitor the size evolution of scatterers which are presented initially by liquid clusters. The liquid clusters were found to have a small negative charge and coalescence into larger species exhibiting negative zeta-potential of about 30-40 mV and associating to submicrometer aggregates. The nucleation and following aggregation of gold sulfide nanoparticles proceed progressively rather than instantly. The processes were accelerated critically by the addition of 0.05M NaCl solution or at elevated temperatures. On the onset of reaction single fraction sticky spheres model fits the experimental SAXS data satisfactorily. However, starting from some instance, the second (smaller in size) fraction needs to be taken into account to successfully describe the SAXS data, which means that SAXS can be used to get insights into the prenucleation and nucleation processes. Similar results were taken for the process of hexachloroplatinic acid reduction by sodium sulfide.

Poster Session

SR XRF USED TO STUDY THE CONTENT OF CHEMICAL ELEMENTS IN THE LEAVES OF LONICERA CAERULEA (CAPRIFOLIACEAE), DEPENDING ON THE CHANGE IN SEISMIC ACTIVITY

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The active, deep faults of lithosphere are the channels, carrying the flows of substance and energy, that can affect natural environment, to the surface of the planet. The ascending migration of fluids and the emanation of gases in the zones of active, tectonic faults have a prominent effect on the distribution of chemical elements in soil, air, and plants. The impact of the active faults on biota is subjected to temporal variations due to seismic changes. The present work studies the content of chemical elements in the leaves of Lonicera caerulea L. (blue honeysuckle) over the region characterized by a high, seismic activity and the novel tectonic faults related to the central part of the Chuiskii earthquake of 2003. The studies were performed in 2004, 2008-2010 during a
gradual decrease in seismic activity in this zone. The total content of micro- and macroelements in soil and their accumulation in vegetable leaves were determined by the method of X-ray fluorescence analysis using synchrotron radiation (SR XRF) at the station of element analysis of the Siberian Center of Synchrotron and Terahertz Radiation, Budker Institute of Nuclear Physics (VEPP-3 storage ring). Fluorescence was excited by monochromatized synchrotron radiation with an energy of 23 and 38 keV, which allowed one to analyze the elements from K to Ba by the K-series, and from Pb by the L-one of the characteristic X-ray radiation. The concentration of an active form of the elements was measured in soil by atom-absorption method. The intensity of the biological absorption of the elements by the leaves of blue honeysuckle was estimated by the coefficient of biogeochemical activity, i.e., the ratio between the element content in the dry substance of plants and the concentration of the active element form in soil. The SR XRF method made it possible to study the relation between 26 elements in vegetable leaves and in soils with high sensitivity \((n \times 10^{-9} \text{ g/g})\) within a wide concentration range in small samples (10-30 mg). A comparative estimation of the element content in the leaves of L. caerulea subsp. altaica, collected in the zone of the active, tectonic fault in different years, indicated that during the period of high seismic activity (2004) the concentrations of K, Fe, Cu, Ti, V, Zr, and Nb in plants decrease and those of Ca, Ba, Br, and Sr increase. In this case, the relations between the basic elements (biophils) also changed. A reliable, positive connection was established between the gross content of Ca, Sr, and Ti in soil and leaves. The linear relations between the gross content and the content of the active form of biophilic elements Ca, Zn, and Sr in soils were also positive. In 2004, a more intense accumulation of secondary metabolites, i.e., chlorogenic acid and glycoside \([\text{U+FFFD]}\ \text{U+FFFD}]\ [\text{U+FFFD}, involved in the regulation of the mineral supply of plants, was recorded in the leaves, collected in the fault zone.

**Poster Session**

**STABILITY OF THE ELEMENT COMPOSITION OF VEGETABLE SAMPLES WITH LONG-TERM STORAGE**

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The content and composition of the elements of vegetable samples have been first analyzed in connection with storage periods by the method of X-ray fluorescence analysis, using synchrotron radiation (SR XRF). The objects of studying were the samples of Pentaphylloides fruticosa, cultivated in the gradient of radionucleotide contamination over the territory of the East-Ural radioactive trace (EURT), formed in 1957 due to the accident at the IM Mayak (the town of Ozersk, Chelyabinskaya oblast) and in the conditionally background region of the observation area. In 2005, the samples of the P. fruticosa leaves and stems, collected in four EURT regions, were analyzed and in 2015 the same samples were analyzed after a 10-year storage period. It has been established that the element composition of the samples was almost the same. In 2005, 18 elements were discovered, and in 2015 additional two elements (Cr and Nb) were determined, probably, because of the improving potentialities of the method. The content of K, Ca, Mn, Fe, Co, Cu, Zn and Sr in the samples of 2005 and 2015 remained practically the same. As a rule, the values varied by \(1-50\%\), depending on the organ and the environment. More considerable variations were observed in the content of Ti, V, Ni, As, Rh, Br, Y, Zr, Mo and Pb, which may be assigned to the increase in the relative standard deviation (%) as the measure of analysis reproducibility for these elements. In this case, as compared with 2005, in 2015 a noticeable decrease (2-4-fold) was recorded in the content of As and Br in the samples of the leaves except for those from the background region for which the difference was only 30-35%. Thus, the element composition of P. fruticosa, analyzed just after collection, and the element composition of that kept for 10 years, were similar. The most stable are K, Ca, Mn, Fe, Co, Cu, Zn and Sr. The tendencies to element accumulation by plants in the gradient of nucleotide pollution were preserved independent of storage period.

**Poster Session**
Exciton-like and defect-related luminescence in K3WO3F3 oxyfluoride crystals

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Complex metal oxyfluorides such as K3WO3F3 are attractive compounds for developing new noncentrosymmetric crystals having ferroelectric and ferroelastic properties. This is achieved due to the strong distortion of metal-(O,F) polyhedra in crystal lattice because of different ionicity of metal-O and metal-F bonds. The luminescence spectroscopy can be a sensitive method to study the character of lattice distortion of these compounds. Spectra of photoluminescence (PL) and X-ray excited luminescence, PL excitation spectra (322 eV), PL decay kinetics as well as thermoluminescence curves were measured for single crystals and ceramics K3WO3F3. Synchrotron radiation (HASYLAB, DESY, Hamburg) were used for low temperature PL experiments with time resolution. Crystals were grown and examined in Institute of Geology and Mineralogy SB RAS (Novosibirsk). The intrinsic luminescence of tungstates is usually ascribed to the radiative relaxation of exciton-like excitations localized on WO₆ octahedra or WO₄ tetrahedra. In K3WO3F3 there are anion sites with mixed oxygen/fluorine occupancy. Therefore, different octahedra form with different distortion. Two emission centers of exciton-like origin, with distinct relaxation time, different types of such octahedra were found. The time-resolved luminescence spectroscopy technique was applied to distinguish these centers, proving itself as a sensitive method to study the character of lattice distortion. The energy transfer mechanism between these PL centers is found and tentatively described by the diffusion of excitons. Apart from intrinsic luminescence, the PL of defect-related centers was found in samples irradiated by fast electrons from linear accelerator (E=10 MeV, D=120 Gy). The role of shallow charge carrier traps in the low-temperature luminescence was revealed.

Biomedical application of SR and THz radiation

Structural parameters of macroscopically flat lipid multilayers on a silica sol substrates

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A phospholipid bilayer on liquid substrate can be used as the simplest model of a cell membrane [1]. Previously, the method of preparation of macroscopically flat regular lipid multilayers with the use of colloidal silica hydrosols as substrates has been proposed [2]. Due to the specific boundary conditions at air/sol interface, polar lipid molecules exhibit a spontaneous ordering effect. In this report we present the investigations of influence of hydrosol compound (i.e. size of silica nanoparticles, pH level and concentration of alkali ions) on the structural parameters of phospholipid layers by the X-ray reflectometry method with the use of synchrotron radiation. Model phosphatidylethanolamine lipids DSPC, DPPC and SOPC have been used as samples. Experiments have been carried at X19C beamline (radiation energy E = 15 keV) of NSLS facility, Brookhaven. Depth-graded distributions of electron density have been extracted from the measured reflectivity curves by the model-independent reconstruction algorithm [3]. According to the obtained results, overall thickness of lipid film is consistent with the Debye length for the substrate sol and thus can be varied from the monolayer (d ~ 35 Å) to the stack of bilayers (d ~ 450 Å) at pH ≈ 11.5 and 9 respectively. Separate mono- and bilayers exhibit high degree of structural perfection, with calculated lipid area A = 45 ± 2 and 49 ± 3 Å² being in agreement with the theoretical values for 2-dimensional phospholipid crystal. Enrichment of the
substrate with Na\(^+\) and Cs\(^+\) leads to the absorption of alkali ions into the lipid membrane, with the surface concentration of ions up to \(2 \times 10^{19} \text{ m}^{-2}\).


X-ray Spectroscopy

XAFS spectroscopy - a useful tool for determining structure parameters and the electronic state of various nanosystems

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Some of the EXAFS spectra above Ga\(^{\text{K}}\)-and Ge\(^{\text{K}}\)-edges were measured at the VEPP-3 storage ring (beamline 8) of Siberian Synchrotron and Terahertz Radiation Center (SSTRC) of the Budker Institute of Nuclear Physics (BINP SB RAS, Novosibirsk, Russia). Other part of the spectra was measured at the ESRF (Grenoble, France). The high energy resolution fluorescence detected HERFD-XANES and EXAFS experiments have been performed at 12K at the beamline ID26 or ID20 equipped by 5-analyzer x-ray emission spectrometer.

1. **Electronic and spatial structure of “calibrated” gold clusters in the cavities of cucurbit[6, 7]uril molecules.**

   The Au\(^{\text{LIII}}\) X-ray absorption fine structure (XAFS) spectra have been measured for samples containing calibrated gold nanoparticles \(D \sim 1\) nm in cavities of cucurbituril molecules (CB[6, 7]). It has been found that gold clusters are characterized by smaller 0.03 A interatomic distances and a considerable reduction in the Au-Au coordination numbers \((N \sim 8)\) as compared with bulk gold. No visible shifts of Au\(^{\text{LIII}}\) absorption edges (0.02 eV) and differences in the Au charge state for Au\(^@\)(U+FFFD)[6, 7] samples containing small Au clusters were detected in comparison with bulk Au metal. A threefold and more increase in the Debye-Waller factor at 12K was found for the nanoparticles in comparison with bulk metal and correspondingly, a substantial increase in the structural disorder. It has been found that special chemical (catalytic) and physical (optical) properties of small gold particles are likely to be attributed to this structure changes and the size effect with the appearance of a band gap between the occupied and unoccupied electronic states, rather than changes in the charge state of gold clusters and presence of strongly pronounced vertices and edges in structure of nanoparticles.

2. **Microstructure of multilayered heterosystems GaNAlN.**

   A minimal drop (\(\sim 0.01\) A) in the interatomic GaGa distances \(R(Ga)\) relative to a thick solid film was obtained from the analysis of GaK EXAFS spectra for multilayered GaN/AlN samples with “thick” (550850 nm) superlattices, agreeing with the numerous dislocations found in them and the corresponding stress relaxation in the GaN layers. The interatomic GaGa distances \(R(Ga)\) for samples with fewer layers and “thin” (80150 nm) superlattices fell more substantially (by \(\sim 0.03\) A), corresponding to the more substantial deformations and stresses indicated by our earlier results for GaN quantum dots in a AlN host. The influence of the growth conditions and the thickness of the superlattices on mixing in the near boundary layers and the optical properties of the GaN/AlN superlattices were revealed. It was established that GaAl mixing occurs only in the layer nearest to the interface. Anomalously long GaAl distances (\(\sim 3.25\) A) were observed for samples with thick superlattices. This effect can be explained by a nonequilibrium transition from GaN growth to AlN growth and the more substantial stresses at the interface of such superlattices.
3. Structural characteristics of K-Bi citrate (De-Nol) and its clusters in aqueous solutions.
Bi\textsubscript{12}L\textsubscript{11} EXAFS spectra of an amorphous solid Bi complex with citrate (De-Nol) and its aqueous solutions in a wide concentration range are measured. For the solutions good agreement is revealed between their structural parameters and the averaged interatomic distances and coordination numbers of 12-nuclear Bi clusters. So, it is found that droplets of the colloidal solution have a structure close to the solid \( Bi_{12}O_{22} \) cluster structure. When the concentrated solution is diluted the cluster structure is somewhat modified, it remaining similar to the structure of the \( Bi_{12}O_{22} \) cluster and even at a tenfold dilution and the nearest (oxygen) spheres of the Bi environment changing insignificantly.
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X-ray Spectroscopy

Near-surface regions of chalcopyrite studied using XPS, HAXPES, XANES and DFT
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Chalcopyrite CuFeS\textsubscript{2}, is the most common mineral of copper; chalcopyrite and related compounds are of interest for thermoelectric, photovoltaic and other applications. We also examined valleriite (Fe,Cu)\textsubscript{2}S(Mg,Al)(OH)\textsubscript{2}, natural nanocomposite composed of chalcopyrite-like Fe,Cu sulfide layers and brucite-like oxide layers, which is important mineral of Norilsk ore deposits. Conventional XPS and synchrotron-based hard photoemission spectroscopy (HAXPES), surface-sensitive Cu L-, Fe L-, S L-edge TEY XANES and S K-, Fe K-edge TEY and PFY XANES spectroscopy were used along with some other experimental techniques and DFT+U calculations in order to characterize near-surface regions of the initial (in fact, oxidized in air) minerals and the ones chemically oxidized in Fe(III) solutions. The synchrotron irradiation experiments were performed at Russian-German laboratory and HIKE endstations at BESSY II facility (Helmholtz Zentrum Berlin). Both the element concentrations and the chemical state of atoms, e.g., mono-, di-, polysulfide and other S species, as a function of the probing depth and the reaction conditions, were studied. The near-surface regions of chalcopyrite are generally consist of (i) a thin, no more than 1-4 nm, strongly metal-depleted outer layer containing polysulfide species, (ii) a layer with smaller stoichiometry deviations and low, if any, concentrations of polysulfide, which composition and dimensions strongly depend on the chemical treatment conditions, and (iii) an about stoichiometric underlayer extended to at least several dozen of nanometers. The latter shows Fe K-edge TEY spectra altered probably due to a high content of defects, even if only oxidized in ambient air. In contrast to other spectra, Cu L-XANES patterns were surprisingly insensitive to the changes of the composition of the reacted near-surface layers. DFT+U analysis of the iron-deficient structures formed shows that polysulfide species are stable only near the surface, copper remains 4-fold coordinated to S in the layers with disulfide anions, the layer with iron deficiency transforms from antiferromagnetic and semiconducting state into paramagnetic metallic state. Moreover, the formation of these structures is generally energy-favorable under the oxidative conditions. The lengthy regions are expected to heavily affect the electronic characteristics and processes involving surface and interfacial charge transfer.

Poster Session

Multifunctional X-ray lithography station at VEPP-3
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The multifunctional station LIGA at synchrotron radiation source VEPP-3 is part of shared-usage “Siberian Center for Synchrotron and Terahertz Radiation” infrastructure. The apparatus used for X-ray lithography are usable for samples with size in range from 1 to 10 cm and resist layers thickness in range 1 µm to 7 mm. The development of processing methods and equipment of station is kept on. Present article deal with the capabilities of station LIGA and samples of produced microstructures.

X-ray structural analysis

Upgrade of the detector for imaging of explosions.

Methods of dynamic imaging of explosions at a synchrotron radiation (SR) beam and small-angle X-ray scattering experiments with exploding samples are being developed in the Siberian Synchrotron Radiation Center (SSRC) at the Budker Institute of Nuclear Physics for more than fifteen years. The detector for imaging of explosions (DIMEX) was developed for these purposes and successfully operating at the beam line 0 at the VEPP-3 storage ring and at the beam line 8 at the VEPP-4M storage ring. The DIMEX is based on gas technology and allow to measure SR flux as a function of position and time with spatial resolution of ∼200 µm (FWHM), maximum frame rate of 2 MHz and time resolution of ∼80 ns. Maximum value of the SR flux that can be measured by the present detector corresponds to ∼5000 photons/(channel*bunch) (20 keV average energy, channel area 0.1x0.5 mm², bunch revolution frequency 4 MHz). Maximum number of frames that can be stored in the present detector is 32 and the number of channels with 0.1 mm width is 512.

In order to significantly improve the precision of data obtained by the DIMEX an upgrade of the detector has been started. The electronics of the gaseous version of the detector has been changed such that the new detector is able to operate with frame rate of 8 MHz and store data in up to 100 frames. A new ASIC was developed for this purpose called DMXG64A that includes 64 channels with low noise integrator and 100 analogue memory cells in each channel. Input charge can be stored to and read out from analogue cells with maximum frequency of 10 MHz. The other parameters such as maximum measured SR flux, spatial and time resolutions are stay the same as in the old DIMEX. This new version of the detector is called the DIMEX-G and is planned to be used at the VEPP-3 storage ring and for SAXS studies at the VEPP-4M storage ring.

For imaging of explosions at the beam line 8 at the VEPP-4M storage ring, where SR flux is expected to be about 100 times higher than at the VEPP-3, a new detector based on Si micro-strip technology is being developed. Si micro-strip sensors with special design in order to be able to measure very high X-ray flux, are manufactured for us by Hamamatsu Photonics company. Each sensor contains 1024 30mm long strips with 50 µm pitch. The sensor thickness is 300 µm and it will be positioned at an angle of 1.7 degrees with respect to the SR beam plane in order to get effective Si thickness along the beam close to 10 mm. The new detector called DIMEX-Si will have spatial resolution of 50 µm (FWHM) and time resolution close to 10 ns. The new ASIC is under development for this project that will allow to operate with the frame rate of 50 MHz and record maximum charge that corresponds to the flux close to 10⁶ photons/(chan*bunch), i.e. about 100 times higher than with present detector.
X-ray apparatus

Performance and characterisation of CsI:Tl thin films for X-ray imaging application

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We developed method of thin scintillator films preparation based on thermal CsI(Tl) deposition on glass substrates. The influence of deposition conditions on the micro columnar structure and crystalline property of the films was studied by scattering electron microscopy. The element composition was investigated by X-ray fluorescence method. We measured light output and spacial resolution as a function of input photons energy (5-35 keV) and film thickness (2-20 mkm). It was observed the strong anti-correlations between the time of CsI(Tl) deposition, film light output and thallium concentration. The films can be used for charged particles beams monitoring as well as for X-ray imaging applications including micro tomography and topography.

Radiation sources and Centers

The MAX IV Accelerator Facility, concept, status and perspectives

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The MAX IV storage ring in Lund, Sweden, is the first Multi Bend Achromat (MBA) SR source going into operation. This report describes the MAX IV concept, technical solutions and commissioning status. Finally, an international overview of some of the MBA projects being constructed or planned is given.

Radiation sources and Centers

Status and Perspectives of Compton Sources

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Generation of high-energy photons via collision of relativistic electron and laser beams is known as inverse Compton scattering or laser Compton scattering. Compton sources, photon sources based on Compton scattering, have been developed in the world to realize high-flux/high-brightness X-ray/gamma-ray sources and exploit applications with energy-tunable and narrow-bandwidth photon beams from these sources. Recent progress of electron accelerator and laser technologies will open a new era in Compton sources. An electron beam of small emittance and high-average current contributes to improving spectral brightness of Compton scattered photons. Flux of generating photons is also increased by a high-power laser together with apparatus such as laser enhancement cavity. We overview the current status of Compton sources including an experiment carried out at the Compact ERL, which was the first demonstration of Compton scattering by combination of an energy-recovery linac and a laser enhancement cavity. Future prospect of Compton sources and their applications is also discussed.

FEL-based study and THz radiation application
Ultrafast Pump-probe Facility based on an RF Photogun

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Small-scale accelerator-based radiation sources have been used for developing advanced technologies and exploring new science with high convenience and low cost. Sometimes they are competitive comparing with giant facilities like X-ray free-electron lasers (X-FELs). We have developed a laboratory-scale ultrashort electron accelerator for investigating femtosecond dynamics of atoms or molecules with pump-probe experiments. This system has four beamlines. Two of them are for ultrafast electron diffraction (UED) experiments on solid and gas samples. The electron bunch duration at the UED beamlines are designed to be ~30 fs in rms. Our target value of the timing jitter between pumping laser pulse and probing electron bunch is 10 fs. The UED beamlines can perform single-shot measurement with a temporal accuracy less than 50 fs. As a pumping source, we have developed a high-intense terahertz pulse generator with the field strength of more than 0.5 MV/cm by using a femtosecond laser and a non-linear crystal. This small-scale facility can be used for investigating time-resolved diffraction experiments with samples of gas, liquid, solid, and surface. The application experiments of the UED beamlines will be performed by the collaboration with universities in Korea.

**Poster Session**

Dynamics of nanoparticles sizes during trinitrotoluene detonation

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In this work we carried out our experiments on measuring small angle x-ray scattering (SAXS) during trinitrotoluene detonation. Registration of SAXS signal at detonation of high explosives (HE) allows to measure fluctuation of density, which are connected with process of carbon condensation. Here we present dynamics of particles sizes during detonation of cast trinitrotoluene charge in diameter of 40 mm.

We also carried out SAXS calculation with real spectrum (which consist of the viggler radiation, TNT absorption and the DIMEX-3 detector absorption). Our calculation shown that average sizes of nanodiamonds behind the detonation front could be restored from measured data on pink SR beam SAXS distribution.

Our experiments with using synchrotron radiation were made on SYRAFEEMA (Synchrotron Radiation Facility for Exploring Energetic Materials) station at the accelerating complex VEPP-4M (Budker Institute of Nuclear Physics). This new station allowed to increase the mass of the studied charges up to 200 grams in comparison with similar station Extreme states of matter at the accelerating complex VEPP-3.
Poster Session

A study of deposition of nanoscale intermediary Au-S species on oxide supports from aqueous solution using XAFS and TEM

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Nanoscale products deposited on oxide supports at room temperature from 0.1 mM tetrachloroaurate aqueous solutions and colloidal solutions (sols) of Au0 and gold sulfide nanoparticles, which were prepared beforehand by the reduction of the Au(III) complexes with sodium sulfide, were studied using total electron yield X-ray absorption near edge structure spectroscopy (TEY XANES), extended X-ray absorption fine structure spectroscopy (EXAFS), X-ray photoelectron spectroscopy (XPS) and transmission electron microscopy (TEM). It was found, that intermediary species of the sulfide reduction (Na2S/HAuCl4 = 3) was deposited on metal oxides as disordered Au2S, the surface of which tends to decompose yielding Au0NPs with sulfur adlayer. This conclusion is supported by EXAFS, which shows that in case of gold deposited onto β-Fe2O3 in similar conditions, the average distance Au-S of the first coordination sphere was 2.32 Å, and a coordination number was equal to 1.1, i.e. gold was presented in a form disordered Au2S. According to TEY XANES and XPS data, the significant reduction or sulphidization of metal oxides surfaces are not occur, excepting CuO, onto which surface a Cu(I) sulfide was formed. For freshly prepared solution with the Na2S/HAuCl4 ratio of 3, a linear correlation between the quantity of gold deposited and isoelectric point of the oxide surface was found implying that intermediates bear a negative charge. The quantity of gold deposited from the intermediates solution of the citrate reduction of chloroaurate is lower, than from the final citrate sols and sulfidic sols, and high relative concentrations of oxidized forms of gold remain on substrates. Several metal oxides were decorated using the intermediates prepared with Na2S/HAuCl4 = 3. The TEM images show mainly Au0 NPs in the range 3-5 nm, excepting CuO where ?10 nm NPs were observed.

Radiation sources and Centers

Synchrotron radiation research and application at VEPP-4

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Presently at VEPP-4 storage ring facility varied experimental programs are carried out including HEP, nuclear physics, synchrotron radiation and e+e- beams (polarized and unpolarized).

Poster Session

ELEMENT COMPOSITION OF PENTAPHYLLOIDES FRUTICOSA OF THE RUSSIAN FAR EAST AND EAST SIBERIA

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The element composition of both bush cinquefoil (Pentaphylloides fruticosa), growing in the Russian Far East (Primorsky krai, Amurskaya oblast) and East Siberia (Buryatiya, Irkutskaya oblast), and the samples of soils has been studied by the method of X-ray analysis using synchrotron radiation (SR XRF) at the station of element analysis of the Shared-Use Center SSTRC Budker Institute of Nuclear Physics, SB RAS (VEPP-3 storage ring). The goal of the work was to investigate the interpopulation variability of the element composition of P.fruticosa and to reveal populations with a high content of macro- and microelements. No less than 21 elements were determined (K, Ca, Ti, V, Cr, Mn, Fe, Co, Ni, Cu, Zn, As, Se, Br, Rb, Sr, Y, Zr, Nb, Mo, Pb).

The maximal content of macroelements (K, Ca) was, as a rule, recorded in leaves and that of microelements was found in stems independent of natural habitat. The highest content of Fe, Ti, Zn, Ni, Pb, Nb, Co was determined in the stems of the plants of the Primorsky krai, that of Cr, Zr, Sr and Br was found in the plants of Buryatiya, and the highest content of Y and V was recorded in the plants growing in the Irkutskaya oblast. Mn was accumulated mainly in the leaves of the plants of the Amurskaya oblast, Rb was found in the plants of the Irkutskaya oblast and Cu ([U+FFFD] and Se in the plants of Buryatiya. The high content of Fe, Co, Ni, Cu, Rb, Zr, Pb in plants correlates with their content in the soils of sampling sites. Most stable is the content of Ca, K and Cu ([U+FFFD]= 8 20%) in the plants of various habitats. The variability of the rest elements of P. fruticosa is estimated as high and very high. In this case, the populations exhibit a small spread of element content (about an order of magnitude) which may be conditionally assigned to narrow samplings. The concentrations of Ni and Mo in the leaves of P. fruticosa vary by two orders of magnitude. The calculated mean geometric values of the element content in the leaves and stems of P. fruticosa of the populations studied are typical of the species, may be applied in a comparative analysis with other plants, may be used to standardize the vegetable staff and may be included in the data base.

The reported study was funded by RFBR according to the research project No.16-34-00699-.

Poster Session

Spatial microstructure of multilayered heterosystems, containing Ge quantum dots molecules in Si on the stages of their nucleation and growth by EXAFS spectroscopy

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The systems with interacting quantum dots (QDs) (in particular, quantum rings (QRs) or quantum dots molecules (QDMs) have attracted much attention both as ground for studying coupling and energy transfer processes between artificial atoms and as new systems, which substantially extend the range of possible applications of QDs. Some of the EXAFS spectra above GeK- edges were measured at the VEPP-3 storage ring (beamline 8) of Siberian Synchrotron and Terahertz Radiation Center (SSTRC) of the Budker Institute of Nuclear Physics (BINP SB RAS, Novosibirsk, Russia). Other part of the spectra was measured at the ESRF (Grenoble, France). The high energy resolution fluorescence detected
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HERFD-XANES and EXAFS experiments have been performed at 12K at the beamline ID26 or ID20 equipped by 5-analyzer x-ray emission spectrometer. Microstructural parameters (interatomic distances, coordination numbers, and Debye-Waller factors) were determined by means of EXAFS spectroscopy consistently on the stages of nucleation and growth multilayered heterosystems, containing GeSi quantum dots molecules (QDMs). The relationship between the variations in microstructural parameters and the morphology of superlattices and symmetric assemblies of QDs was established. In accordance with the results obtained previously for quantum dots SiGe it was found for QDMs, that distortion at the interfaces leads to a decrease of Ge-Ge interatomic distances on $\sim 0.03$ A. It was established for the samples with stoichiometric formula $Ge_xSi_{1-x}$, $(0.25 < x < 0.45)$, that average interatomic distances Ge-Ge and Ge-Si correspond to distances, defined for solid solutions and for GeSi quantum dots. The effect of heterosystems topology and temperatures at different stages of their growth on the interlayer diffusion was studied. It was found that the first stage of growth at the temperature of 700 C (growth of templates) provides the basis for QDMs and leads to the concentration of Ge atoms in the system about 38%. The further growth of the layers vertically aligned groups of quantum dots leads to the concentration of Ge atoms in layers about 43-47%, depending on the growth conditions. When QDs were formed without forming templates, Ge atoms concentration was $\sim 60$ – $70\%$ depending on the thickness and number of Ge layers. A comparative analysis of the different methods of EXAFS spectra measurement for reliable determination of structural parameters of SiGe heterosystems SiGe with various thicknesses was carried. This work was supported by the Russian Foundation for Basic Research (project no. 16-02-00175a)

Poster Session

Technogenic Fallout of Uranium and Thorium in Novosibirsk vicinity (Russia, West Siberia)

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The article is devoted to the problem of technogenic pollution of environment: the results of the mineralogical and geochemical investigation of suspension content of melted snow and soils samples of Novosibirsk vicinity are considered. Content of U, Th and heavy metals were determined by means of X-ray fluorescence elemental analysis with synchrotron radiation (SR-XRF) method. For the 235U and 238U isotope analysis is used mass-spectrometry with inductively coupled plasma (ICP-MS) method. Mineral formations of aerosol particles were studied with scanning electron microscopy. Previously shown that the V, Mn, I, Ga are the main geochemical indicators of emission of Novosibirsk heat and electric power plants (HEPP -2, -3, -5) [Artamonova, 2011]. But the Th and U is revealed to be the geochemical indicators of HEPP emission too. Brown coal of Kuzbas and Kansk-Achinsk coal basin are used as firewood of Novosibirsk TETs. Coal of these deposit basin is known to be enriched by heavy metals and natural radionuclides during their geological origin time. So, Th and U are significant in technogenic fallout of vicinity. This fact is confirmed by studies results: U and Th content in aerosols and soil of HEPP vicinity are a few times greater than in pure background cite. The second source of U fallout are the emissions of the Novosibirsk Chemical Concentrates Plant (NCCP) of Rosatom State Nuclear Energy Corporation. Namely the isotope 238U/235U ratio is used as the main indicator of NCCP aerosol pollution. The maximal distance of NCCP emission plume is revealed to arrive by 70 km, where 238U/235U ratio of aerosol is 132.1 ? 3.3. The 238U/235U increases up to about natural ratio 138.4 at 110 km far from NCCP emission northern east plume only. In the NCCP vicinity the particles of uranium oxides in technogenic aerosol were established by means of scanning electron microscopy. Thus the U, Th technogenic pollution of Novosibirsk vicinity is revealed. The ecological situation may be developed by heat and electric power plant modernization, by gaseous matter will stand the dominating firewood of Novosibirsk HEPP. The elemental SR-XRF analysis of snow precipitation and soil can be used to carry out a timely and reliable environmental assessment of an urban territory. The work was supported by the Russian Foundation for Basic Research under project No. 14-05-00289.
X-ray structural analysis

Investigation of influence NH4VO3+HOCH2CH2OH oxidation of ASD-4 powder

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To improve the characteristics of rocket propellants, high-energy metal powders are used. Aluminum powders are most widely employed metallic combustibles. However, the presence of an oxide film on the surface of particles is a grave disadvantage, which does not allow using their energy potential in full. This work presents results of a study of the oxidation process of Al powder modified ammonia metavanadate and ethylene glycol. The specific surface of Al powder was 0.4 m²/g; active metal - 98.7%; vanadium content - 0.5-0.8%. Carried out a comprehensive study of the modified aluminum powder oxidation by methods TG, DSC, X-ray diffraction using a synchrotron radiation source. The study was conducted in air at a heating rate of 10 deg./min. to 1250 °C. It has been found that the impregnation of micro-powder ASD-4 accelerates its oxidation under heat and shifts the process to the low temperature zone, compared with the initial powder Al. Showed the features of oxidation by the formation of intermediate phases of vanadium oxide at low temperatures. The two modifiers have been compared: NH4VO3 + HOCH2CH2OH and studied previously V2O5 nH2O. The latter has the advantage of the completeness of the final conversion, but for the more active powders is preferable to use ethylene glycol.

Invited Talks

FEL-pumped Silicon lasers based on hydrogen-like impurity centers

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Hydrogen-like impurity centers in monocrystalline Silicon can be utilized for terahertz(THz) lasers. Several types of such lasers have been demonstrated. The lasers operate at low lattice temperatures under optical pumping with a free electron laser (FEL). They emit light frequencies between 1.2 and 6.9 THz. Dipole-allowed optical transitions between particular excited states of substitutional impurities are utilized. Population inversion induced by optical pumping with the FEL is achieved due to specific electronphonon interactions inside the impurity atom. This results in long-living and short-living excited states of the impurity centers. Another type of THz laser utilizes stimulated resonant Raman-type scattering of photons by a Raman-active intracenter electronic transition. By varying the pump-laser frequency, the frequency of the Raman intracenter silicon laser can be continuously changed between at least 4.5 and 6.4 THz. In addition, fundamental aspects of the laser process provide new information about the peculiarities
of electronic capture by shallow impurity centers in silicon, lifetimes of nonequilibrium carriers in excited impurity states, and electron-phonon interaction.

**Poster Session**

**Power supply system for corrector magnets of the European X-Ray Free-Electron Laser**

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Total length of the European X-Ray Free-Electron Laser (European XFEL) tunnel is 3.4 km. There are used about 300 magnet correctors to justify position of electron beam. Each magnet is feed by separate power supply with high parameters. To have a stable beam the short time (up to 1 sec) deviation of output current should be less than $10^{-5}$, long term stability should be better than $10^{-4}$. For this application the BINP SB RAS developed 7 types of power supplies with output current up to 10 A and output voltage up to 70 V. To provide high reliability of power supply system there was developed hot replace system allowing remote replacement failed power supply by reserved one. Now the power supply system is introducing in operation. The paper is reviewing details of power supply system for European XFEL.


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According to microelectronic production leaders the lithography based on the free electron laser (FEL) could become the main technology for the elements mass production with scale to 5 nm in the nearest future. One of the main problem is the absence of the working FEL with required parameters. The feasibility study of those FEL based on superconducting energy-recovery linac (ERL) was made in Budker INP. The ERL average current is limited by longitudinal and transverse instabilities, caused by interaction between electron beam and its induced fields in the superconducting cavities. The estimations of the threshold currents and ERL parameters were made.

**X-ray apparatus**

**3D X-ray lithography**

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An increased depth of the transformed material, due to the low absorption of materials to X-rays, was one of the main advantages of the X-ray lithography for the photolithography and for the electron beam lithography. But, this property was an obstacle to its further development concerning 3D patterning. Practically, all the created by X-ray lithography so-called 3D structures [A.del Campo, C.Greiner. SU-8: a photoresist for high-aspect-ratio and 3D submicron lithography,
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J. Micromech. Microeng., v.17 (2007) R81-R95 are really 2D+ structures. In this work, a concept of manufacturing of real 3D structures is proposed. At the same time, the high spatial resolution of the method is kept. Using a high-contrast X-ray resist, 3D microstructures of various forms and sizes are created. The smallest microstructure achieve a size of about 0.3 microns.

Poster Session

Application of ELN-200 in deep X-ray lithography

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The negative tone electron beam resist ELN-200 has been developed in 80th years of the 20th century for thin film patterning. The sensitivity of the resist to X-rays has also been studied [E.S.Gluskin, A.A.Krasnoperova, G.N.Kulipanov, V.P.Nazmov, V.F.Pindurin, A.N.Skrinsky, V.V.Chesnokov, Experiments on X-ray lithography using synchrotron radiation from the VEPP-2M storage ring, Nuclear Instr. Meth. in Phys. Res., v.208 (1983) 393-398]. In the actual work, the X-ray lithographic properties of the resist have been studied after 33 years of storage. It was established that the contrast and the sensitivity of the resist to X-rays are not changed. The conditions of both formation of thick resist layers and manufacturing of high aspect ratio microstructures using hard X-rays are developed. The microstructures of up to 70 µm in height have been manufactured. The roughness of vertical sidewalls of the microstructures doesn't exceed 50 nanometers.

Poster Session

An inverse method of structured X-ray screen manufacturing

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It is shown that the spatial resolution of an X-ray screen can be increased by forming structures like channels which are filled with a phosphor [V.I.Kondratiev, M.V.Kuzin, N.A.Mezentsev, L.A.Mezentseva, V.P.Nazmov, Yu.T.Pavlyukhin, A.Krasnoperova, V.F.Pindyurin, A.A.Sidelnikov, B.P.Tolochko, Preliminary testing of microstructured imaging plates with improved spatial resolution. Nucl.Instr.Meth.Phys.Res.A448 (2000) 207-210]. It is established that the diameter of the channel is smaller, the higher spatial resolution can be achieved. However, the problem of filling of narrow channels is increasing. In the proposed method, the phosphor has been mixed with the structured material SU-8 - resist and the patterning is carried out. Thus, the need of filling of channels with a phosphor disappears. Due to the high penetration depth of hard X-rays into the materials during the patterning step, regardless heavy elements inside Gd2Os2 as a phosphor, the height of patterned microstructures can achieve tens or hundreds of micrometers. The optical isolation of the patterned phosphor pillars is carried out by electroplating of nickel between the pillars. The achieved spatial resolution and the details of the manufacturing process have been discussed.

Poster Session
The SR-XRFA usage in biogeochemical studies: element composition of larch tissues (Larix cajanderi Mayr.) of Kuranakh Gold Mining area (Russia, Yakutia)

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This article is devoted to the problem of biogeochemical peculiarities of plant of mining gold ore deposit zone. The tissues of larch (Larix cajanderi Mayr.) were the object of our studies using of X-ray fluorescence elemental analysis with synchrotron radiation (SR-XRF) method. Large possibilities of SR-XRFA, along with the simple preparation of biological samples, allowed us to carry out valuable biogeochemical investigations in Kuranakh Gold Mining area (Russia, Yakutia). New data on the accumulation of biophilic microelements Cu, Zn, Mo, chalcophilous Ni, Pb, Ag, As, Sb, rare lithophilous Rb, Sr, Zr, Y, Nb, scattered chalcophilous Ga, Ge, Se, Cd, Te, Tl in the tissues of larch (Larix cajanderi Mayr.) of gold ore deposit areas and of gold wastes tailings were obtained. The changes of elemental composition of plant under technogenic impact in area of tailings and in areas of natural geochemical anomalies of ore deposits is very significant to assessment ecological risk of technogenic impact in mining areas.

X-ray apparatus

Manufacturing of high resolution X-ray masks for LIGA technology in SSTRC

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One of main problems in deep X-ray lithography (DXRL) is X-ray masks fabrication. The primary way to get high-resolution X-ray masks is to use soft X-ray lithography with intermediate X-ray mask. The soft X-ray lithography is performed at LIGA station of VEPP-3 synchrotron radiation (SR) source in Siberian Synchrotron and Terahertz Radiation Centre (SSTRC). In addition, the technique of fabrication of intermediate X-ray mask based on titanium membrane is developed.

Poster Session

[U+FFFD] THOD FOR MANUFACTURING SELF-BEARING MICROSTRUCTURES OF THE PSEUDO-METALLIC TYPE

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We consider the peculiar features of the LIGA-technology methods elaborated in the Siberian Synchrotron and Terahertz Radiation Centre (SSTRC, BINP SB RAS) and applied to producing quasi-optical subterahertz filters and focusing elements based on high-aspect self-bearing microstructures of the pseudo-metallic type. The essence of the method consists in deep X-ray lithographic patterning of an organic glass (PMMA) substrate followed by covering its entire surface with a thin layer of metal (Ag or Al). The results of manufacturing the inductive microstructures with geometry of honeycomb arrays, including high-pass filters with the cut-off frequency of 0.28 THz and focusing elements operating at 0.65 THz, are presented. The details of spectral characterization of the developed structures using a BWO-spectroscopy technique are discussed. Good agreement between the results of measurements and numerical simulations is demonstrated.

**Poster Session**

**Hybrid magnet wiggler for SR research program at VEPP-4M**

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A compact hybrid magnet wiggler is under development for the VEPP-4 SR research program. The wiggler allows to increase the photon flux by two orders at the beam energy 4.5 GeV in the spectrum range of 60-100 keV. The magnet consist of conventional coils which generate magnetic field and permanent magnet insertions which concentrate the flux. The solution increases field at the beam orbit up to 2.05 T. Here we present design, magnetic calculations and analysis of the beam dynamics under hybrid wiggler influence.

**Poster Session**

**MANUFACTURING LIGA-MASKS WITH LASER MICRO-MACHINING**

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In the LIGA-technology, the first stage in forming a micro-relief is the deep X-ray lithography which is implemented, as a rule, by using synchrotron radiation (SR) of the wavelength spectral range $\lambda \approx 1-3 \mu m$. Realization of this technology implies availability of a high-contrast X-ray mask (LIGA-mask) containing X-ray opaque topological drawing which is normally formed via galvanic deposition of heavy metals on the surface of a X-ray transparent bearing membrane. It is worth noting that a number of products made with the LIGA-technology, e.g. quasi-optical terahertz filters and focusing elements are characterized by self-bearing topology. It opens up a possibility of the structures production by means of the alternative but significantly simpler technology laser cutting of a metal foil that allows eliminating the bearing membrane and excludes technological problems related with electroplating. In this work, we describe the results of experiments with two different laser systems based on solid-state pulsed lasers ($\lambda_1 = 1064 \text{ nm}$, $\lambda_2 = 532 \text{ nm}$, pulse duration $\approx 10 \text{ ns}$) which were utilized for producing LIGA-masks via patterning brass and lead foils 50 um thick. The best results from the viewpoint of the cut edge roughness were obtained.
with a brass foil patterned with the 1064-nm-laser which provided the average roughness size ~ 273 um. The technological constraints imposed by the proposed laser technique on the structural geometrical parameters of LIGA-masks, as well as the ways for their mitigation are discussed.

Poster Session

Obtaining Soft X-ray Emission and Absorption Spectra using SR from the VEPP-4 storage ring on the metrology station "Cosmos"

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X-ray spectroscopy is powerful tool for investigation of the electronic structure of functional materials. X-ray emission spectra (XES) corresponding transition from valence orbitals to core levels allows to obtain information about atomic partial densities of occupied states of the elements in complex chemical compounds. X-ray absorption near edge structure (XANES) allows to study atomic partial densities of unoccupied states. Usage of synchrotron radiation for obtaining XES and XANES spectra is to essentially increase quality of experimental data. The experimental equipment of the “Cosmos”- synchrotron station was adjusted for obtaining soft XES-spectra of reference samples (quartz glass, aluminum and copper foils). In the present work were used two different optic schemes: flat (Bragg-scheme) and cylindrically bent crystals (Iogans scheme). Using the cylindrically bent crystal geometry has shown best result in obtaining soft XES-spectra. Also it was a special interest to obtain soft X-ray absorption spectra. For obtaining such spectra the double crystal scheme was used. Thus fine structure of soft X-ray absorption spectra was recorded for reference samples (aluminum, copper foils). Experimental XES and XANES spectra were compared with the spectra obtained on a laboratory X-ray spectrometer and the results of the quantum-chemical calculations. The authors are grateful to prof. L.N. Mazalov for useful discussions of experimental data.

Poster Session

Structure and properties of ZnSxSe1-x alloy nanostructures embedded in anodic alumina membrane

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ZnSxSe1-x alloys, as an important member of IIIVI ternary semiconductors, have attracted significant interest until now due to their variable band gap, spanning from 2.7 eV to 3.67 eV, which makes them appropriate for developing short wave length LEDs and laser diodes operating entirely in the blueviolet region. Recently, the ZnSxSe1-x alloy nanostructures were synthesized by a variety of methods such as chemical vapor deposition (CVD) and metal organic chemical vapor deposition
The electronic structure and band gap of ZnS\textsubscript{x}Se\textsubscript{1-x} were also discussed [5]. The design of Zn\textsubscript{y}Se\textsubscript{1-y}/GaAs (001) heterostructures and their dynamical X-ray diffraction were reported [6,7]. It is worthy to note that the 1D ZnS\textsubscript{x}Se\textsubscript{1-x}-based ZnO/ZnS\textsubscript{x}Se\textsubscript{1-x} core/shell nanowire arrays, ZnO/ZnS\textsubscript{x}Se\textsubscript{1-x}/ZnSe double-shelled heterostructure and ZnS/ZnS\textsubscript{x}Se\textsubscript{1-x} nano-heterostructures have also been synthesized by chemical vapor deposition method or magnetic force assistant growth technique, which exhibit efficient visible light absorption, enhanced photoelectrochemical performance and integrating emission property, respectively [8-10]. However, the data on the optical absorption and photoluminescence spectra yielded different results for the ternary compounds produced by different methods, and understanding of the physical mechanism leading to this controversy is still not achieved. The semiconductor nanostructures in dielectric matrices are of current interest due to absorption and luminescent properties improved in comparison to thin films of materials [11, 12]. In nanocrystals, the surface-to-volume ratio is considerably large, therefore there is a high contribution of surface in the luminescence properties. The unsaturated bonds on the surface create band-gap states that can easily capture the excited electrons and holes and relax the energy in non-radiative ways [13]. In addition, matrix isolation allows to protect nanostructures from external influences. In this work we for the first time have successfully synthesized alloyed ZnS\textsubscript{x}Se\textsubscript{1-x} nanowires embedded in anodic alumina membrane via thermal evaporation of the mixture of ZnS and ZnSe powders. Various compositions can be easily obtained by changing the mole ratio of ZnS to ZnSe in a source material. The X-ray diffraction patterns show that the lattice structure of these nanostructures is cubic ZnSe-like, as S atoms replace Se and nanostructures compositions correlate with their initial S/Se ratio. Chemical composition was controlled by XPS method. Optical absorption spectra show that band gaps of the alloy cover the entire range from 2.7 eV to 3.67 eV by changing the component ratio, in agreement with literature. The study of the local atomic structure was carried out via EXAFS method. The set of structural parameters, namely the interatomic distances and corresponded coordination numbers, were established. All possible structural models were discussed in detail. Additionally ZnS\textsubscript{x}Se\textsubscript{1-x} samples were characterized by the TEM, EDX and XPS methods. The data obtained by all the methods are in a good agreement.


Poster Session

Synergistic influence of HF welding on nanostructural orderliness of epithelial tissues of gastrointestinal tract

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The results of study of influence of high-frequency electrosurgical welding (HF welding) on molecular and nanostructural orderliness of thin intestine tissues of a pig - mucosa, submucosa, muscle layers, serosa, and also the purified mucin preparations from pigs gastrointestinal tract are presented. Structural study of biological tissues by X-ray diffraction methods with synchrotron radiation usage were carried out on constructed by us small-angle station diffraction cinema DICSI at the NRC Kurchatov institute, Moscow. Considerable distinctions on X-ray patterns of thin intestine tissues are registered before and after HF welding: the X-ray patterns of native tissue display a small-angle diffuse scattering; diffraction lines practically do not reveal. After application of HF welding, the small-angle X-ray patterns of welded sutures in intestinal tissue are literally filled with sharp diffraction rings: a series of sharp Debye rings with main spacing of 4.65 nm was recorded within the nanoscale range of 10 to 0.4 nm. We earlier attributed the nanostructural invariant of 4.65(0.15) nm as periodicity caused by regular attachment of oligosaccharide chains to the protein core of giant proteoglycan molecule of mucus and extracellular matrix of tissues [A. Vazina et al., Bullet. RAS, 2013, 77(2)]. Considerable changes in X-ray patterns of gastrointestinal tissues under synergistic influence of various parameters of welding (temperature, toolkit geometry, pressure, modulation of an electromagnetic field, etc.) can be interpreted by increase in concentration of scattering components due to cold steam formation at acoustic excitation of a site of a tissue between electrodes. Thus, under the influence of welding in tissues it is formed high ordered proteoglycan scaffold providing the functional arrangement of cells during reparation. Proteoglycan systems can be not only a marker of the physiological status of a tissue, but also a regulator providing adequate adaptation of biological systems to change of external parameters of environment. The research was supported by RFBR-Moscow region Grant #14-44-03667.

Poster Session

Phase content of interfaces Ti / Al3Ti in metal-intermetallic laminate studied by x-ray and synchrotron diffraction

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In the TiAl3Ti composites produced by reaction sintering of titanium and aluminum foils under pressure, any oxides that accumulate at the front of an intermetallic reaction form a thin extended region in the forming intermetallic Al3Ti layer. This region can be a weak point in the microstructure of the laminate composite material. Sintering route was optimized to minimize the quantity of oxides particles (basically Al2O3 particles) in order to improve some mechanical properties. The phase content of micron composite layers was studied by x-ray and synchrotron diffraction. X-ray diffraction (Bragg-Brentano geometry with a flat-plate sample)
showed that composite layers basically contain only Ti and Al3Ti phases. Synchrotron diffraction (a transmission mode to pass through the 390 micron thickness sample layer) allowed to reveal the presence of residuals of aluminum phase. Also, the frame capture mode, using of synchrotron beam with the size 0.4x0.1 mm, allowed to prove the absence of Al2O3 phase particles along the Ti / Al3Ti interfaces.

Diffraction experiments were performed at the 4-th SI-channel of VEPP-3 beam station. The research was carried out within the state assignment of FASO of Russia (theme “Deformation” No. 01201463327).

Poster Session

Application of the ?-const method on VEPP-3 in small-angle diffractometry of biological objects

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The researches of test samples (collagen fibres and alive muscles in various functional conditions) were carried out by method of power diffraction (?-const) with use of synchrotron radiations of VEPP-3 storage ring (INP SD RAS, Novosibirsk) with a continuous spectral interval from 5 to 20 keV (7 BeV, I = 2 - 10 mA). Use the semi-conductor Si/Li detector with high energy resolution has allowed to register the informative diffraction pictures during exposition times, essentially smaller, than at traditional methods of registration. X-ray patterns of collagen show about 16 diffraction peaks in the range of 1100 - 310 nm, distance between peaks to 800 eV, and X-ray patterns of muscle display up to 7 peaks in the range of 1430 - 530 nm. Results of the work have shown that high efficiency of registration and the energy resolution of the detector give possibilities for development of a method of high-speed diffractometry of biological objects. Thus, the big intensity of SR, the wide spectral interval surpassing the intensity of a monochromatic beam on several degrees in integrated intensity, and modern high-sensitive detectors open possibilities for the decision of a new class of the problems connected with dynamics of structural transformations.

Poster Session

Status of the experimental station on the fourth beamline of VEPP-3 storage ring

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Present status of the experimental station on the fourth beamline of VEPP-3 storage ring (Siberian Center for Synchrotron and Terahertz Radiation, INP SB RAS, Novosibirsk, Russian Federation) and some recently obtained results are presented.

Biomedical application of SR and THz radiation
Analysis of K-lines X-ray fluorescence of Rare-Earth and High-Z elements on storage ring of the VEPP-4M

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X-ray fluorescence analysis with high-energy photons (40-100 keV) is described. Measurement of K-lines from high-Z elements has attractive advantages, which are not available by L-line analysis. The experimental data on the minimum detection limits (MDLs) of rare-earth elements from La to Lu in standards of igneous rocks are described. The first results were obtained at the minimum detection limits of heavy platinoids (Os, Ir, Pt, Au) based on the lines of the K series of the characteristic radiation excited by photons with energy 100 keV. The main parameters of the beamline, layout, monochromatization system and detection system of the X-ray fluorescent are described.

X-ray apparatus

DIAMOND REFRACTIVE LENSES FOR DIFFRACTION-LIMITED X-RAY SOURCES

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An intensive development of X-ray refractive optics instrumentation and tools has given birth to X-ray refractive lenses [1] which are now standard elements at third-generation synchrotron radiation sources. In view of the global switch to the fourth generation of synchrotron sources and X-ray Free Electron Lasers, there is a growing need for x-ray optical elements fabricated from materials that can withstand extreme heat and radiation loads while still providing effective focusing and imaging. Diamond can satisfy all the requirements provided that a suitable lens manufacturing technology is available.

In our research [2], for the first time single crystal diamond planar refractive lenses were fabricated by laser micromachining in up to 1.2 mm thick diamond plates which were grown by CVD and HPHT. Various linear lenses with apertures up to 1mm and radii of the parabola apex up to 500m were manufactured and investigated with SEM, AFM, Raman spectroscopy and, of course, X-ray tests at the ESRF ID06 beamline. X-ray focusing showed the high quality of the lens side walls and profile allowing to focus the X-radiation in accordance with the lens demagnification factor. Planar lenses were followed by 2D parabolic X-ray refractive half lenses, which were also manufactured by laser micro-machining of single-crystal diamond. A single 2D lens had an aperture of 1 mm and parabola apex radii of 200 m. Forming a compound refractive lens with 24 single lenses within, it has been successfully tested in the focusing and imaging modes both at the APS source [3] and at the laboratory setups using Cu Kα X-radiation from the rotating anode generator and microfocus MetalJet X-ray tube with a liquid-gallium jet as the anode using Ga Ka line. The lens has successfully reproduced the triangular object with the theoretical demagnification while the focusing of the 20 m source was performed with the small deviation from the theoretical value.

The present study demonstrated that laser micro-fabrication technology provides a straightforward method for the fabrication of single-crystal diamond refractive lenses with large acceptance and...
high shape and surface (peak-to-valley roughness ~ 1m) quality. Unique optical properties of diamond single-crystal lenses (the refractive index decrement, , in diamond is double that of beryllium) coupled with its excellent thermal qualities (high thermal conductivity and shock resistance; low thermal expansion coefficient; high temperature stability) allow them to be applied as focusing, imaging and beam-conditioning elements at high-heat flux beams of today and future X-ray sources.

References


Poster Session

APPLICATION OF SHORT-WAVE DIFFRACTION OF SYNCHROTRON RADIATION FOR IN-SITU INVESTIGATION OF ZIRCONIUM HYDRIDES FORMATION AT GAS-PHASE HYDROGENATION

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Zirconium alloys are widely used as construction materials in an active zone of nuclear reactors. During operation of such alloys in contact with a hydrogen-containing environment, hydrogen penetrates into them. This significantly influences on the physical, chemical and mechanical properties of the material [1,2]. To investigate the effect of hydrogen accumulation on the zirconium alloys properties is necessary to prepare the experimental samples with different hydrogen concentrations and different distribution. However, the hydrogenation process can be hampered by the presence of a thin oxide film on the samples surface, which is greatly effect on the hydrogen distribution by material volume. The application of the nickel coating increases the hydrogen sorption rate. Also nickel has a high permeability to hydrogen. Moreover, the nickel oxidation is less active than zirconium, which contributes to hydrogen absorption.

In this paper zirconium alloy (Zr1%Nb) samples with a micron-order nickel layer were investigated. Hydrogenation was carried out by gas phase method at temperatures of 350, 450 and 550 °C. Hydrogen pressure in chamber was 1 atm. Hydrogen sorption curves were obtained on the automated complex Gas Reaction Controller. In situ diffraction measurements were carried out in the process of hydrogen saturation of samples at the station "Precision diffractometry II" of the Institute of Catalysis of the Siberian Branch of the Russian Academy of Science at the 6 synchrotron radiation channel of the VEPP-3 electron storage ring. Comparison of the data obtained in the analysis of hydrogen sorption curves with data of the phase transitions in the zirconium-hydrogen system at a gas phase hydrogenation allowed describing the processes occurring in zirconium by hydrogenation at different temperatures.


Invited Talks

Semiconductor spectroscopy with infrared and terahertz free-electron lasers
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This talk reviews some recent spectroscopic studies on semiconductor structures carried out using the mid-infrared and terahertz (THz) free-electron laser (FEL) facility FELBE in Dresden, Germany. Its intense, nearly transform-limited picosecond pulses, which can also be combined with synchronous pico- or femtosecond pulses from near-infrared tabletop lasers, provide unique research opportunities to advance our knowledge on the interaction of intense mid-infrared and THz fields with materials and devices.

X-ray structural analysis

Status of dynamic diagnostics of plasma material interaction based on synchrotron radiation scattering at the VEPP-4 beamline 8

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The residual mechanical deformation and stress were measured in the preliminary experiments carried out at synchrotron radiation (SR) scattering stations at VEPP-3 in the Siberian Center of Synchrotron and Terahertz Radiation (Precision diffractometry and anomalous scattering, Diffractometry in hard X-rays). The deformation and stress were calculated on the basis of the dependence of the scattering angle on the inclination of the irradiated tungsten sample. Significant changes in the SR diffraction are found as the result of material recrystallization or irradiation of the material by plasma. It implies that the SR scattering diagnostics may be an informative instrument for in-situ observations of the state of the plasma facing components.

The next stage of the SR scattering diagnostics development at the VEPP-4 beamline 8 is the dynamic measurements during pulsed heat loads. Currently a 1J Nd:YAG laser is used for the
0.2ms heat load simulation and a 100J laser is under development. The parameters of the heating are sufficient for simulation of the expected pulsed heat load in ITER. The destructive effect of pulsed heat loads is caused by mechanical stresses that occurs in highly non-uniformly heated materials. The main aim of the current development of diagnostics based on SR scattering is the dynamic measurements of the mechanical deformation and stress dependences on the depth below the surface. It looks like the rotation of crystallographic planes due to mechanical deformations is the dominant effect in the SR scattering. So the deformation and stress distributions may be calculated using measurements of the diffraction peak parameters of SR passed through the sample. The set of requirements (the pass through material, dynamical measurements of pulsed processes) determines restrictions on SR brightness and energy. The SR from VEPP-4 with energy 69keV will be used for experiments with tungsten. Also a single crystal samples are necessary for increasing of the diffraction peak brightness. Currently, the one-dimensional gas X-ray detector DIMEX is used for measurements. The development of the silicon detector for increasing of the sensitivity is in progress.

The first dynamical measurements of the diffraction peak of SR passed through the 250m thick single crystal tungsten were carried using the one-dimensional detector. The inclination of the sample to the initial SR beam was about 45 in order to result in the diffraction peak shift. The change of the SR scattering angle about 0.1 during the laser irradiation and the subsequent fast return during the comparable time were found out. The behavior could be explained by the bending of the initially plane sample. The bending moment is proportional to the difference of the temperature at the heated and opposite sides. The moment disappears while the temperature distribution flattens across the thin sample. Conversely the temperature propagation along the sample surface takes more time. Thus the expected effect of the crystal planes rotation could be estimated as the diffraction peak shift after the temperature flattening across the sample. The measured shift was about 0.03. The measurements of deformation and stress distributions across the sample requires the analysis of the diffraction peak shape during the laser irradiation.

**Poster Session**

**On the dynamical theory of the X-ray DuMond-type spectrometer**

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Dynamical Laue-diffraction of X-rays on the bent crystal is presented. The phase of diffracted wave takes into account cubic terms. The DuMond-type spectrometer is considered for the X-ray source, situated on the Rowland-circle. Dynamical theory of the DuMond-type spectrometer is developed for ideal and mosaic curved crystals. Dependence of the spectral resolution on the source dimensions is analyzed.

**Poster Session**

**Spatial-temporal diffraction of thermal neutrons by ideal deformed crystals**

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Dynamical theory of thermal neutrons diffraction by ideal deformed crystals is presented. Deformations of different kinds (mechanical bending, temperature gradient, ultrasound oscillations) are considered. The diffraction phenomenon of spatial-temporal focusing of neutrons is analyzed.
Synchrotron-based experimental study and theoretical simulation of hydrogen desorption for solid-state hydrogen storage material Mn(BH4)2

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The material under study is manganese borohydride Mn(BH4)2. The unique thermodynamics and kinetic properties in combination with high volumetric and gravimetric hydrogen densities make this compound a very promising candidate to solid state hydrogen storage. Local atomic structure of Mn(BH4)2 upon heating was completely studied by in-situ temperature-dependent measurements of X-ray powder diffraction patterns and Mn K-edge XANES (X-ray Absorption Near Edge Structure) at SNBL-01b beamline of ESRF. Diffraction patterns indicate amorphisation during heating the sample above 110 oC. XANES Mn K-edge spectra also undergo significant changes at the temperature from 120 oC to 160o C. TGA analysis reveals drastic weight reduction (up to 10 mas %) of Mn(BH4)2 at the same temperature range. It was concluded that temperature induced phase transition which is accompanied by sample amorphisation process as well as abundant hydrogen release were observed. We have performed ab-initio structure prediction using pseudopotential approaches within DFT approximation (VASP 5.3 code). Structural models with different contents of hydrogen atoms per unit cell were considered. Cell shape, cell volume and atomic position were relaxed. The simulation indicates collapse of porous structure and decrease in the interatomic distances Mn-B and Mn-Mn provided by significant reduction of cell volume. To find possible stable structures of dense Mn-B phase we have applied evolutionary algorithms as implemented in the USPEX code. Several low-energy candidates were selected for further analysis. Owing to amorphisation process we expect that the sample after heating has unhomogeneous structure with a nanodomain features corresponding to various crystalline atomic ordering. XANES Mn K-edge spectra were calculated for the lowest energy structural models, predicted by means of simulations. Numerical analysis of the discrepancy between experimental and theoretical XANES spectra was performed in order to verify the MnxBy local structure observed in the experiment.

Poster Session

Scanning X-ray fluorescence analysis of biological samples

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The objective of the work is examination of plant standards for estimation of the minimum limits of detection of trace elements in biological tissues and samples, as well as visualization of the distribution profile of some chemical elements in a clinical sample. The work considers the advantages of using synchrotron radiation (SR). The examination of prepared samples was conducted by a standard technique, a reference to which is given in the work. The analysis enables determination calculation of the minimum limits of detection of elements. A conclusion was made that analysis enables detection of heavy elements with a content of about 1 ppm in a biological tissue. The linear profile of platinum distribution in a clinical sample and a two-dimensional map
of the distribution of elements has been obtained; a correlation between the relative content of sulfur and platinum in the material of a bulb of the brain has been revealed. The corresponding figures and graphs are presented.

Poster Session

On the structural features of mechanically alloyed Cu-Ag and Au-Co by severe cold and cryogenic plastic deformation

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Systems of positive enthalpy of mixing Cu-Ag and Au-Co in a condition of powder mixtures were processed by high pressure torsion at 293 K (cold deformation) and 80 K (cryodeformation). Original concentration of the elements for Cu-Ag was 80:20 at.% respectively and for Au-Co were 80:20 and 50:50 at.% respectively. The samples were studied by an X-ray diffraction analysis by synchrotron radiation at a wavelength of 0.03685 nm. It was found that the substitution solid solutions on the base of Cu for Cu-Ag and on the base of Au for Au-Co are formed. Besides that another phases as a result of decay or incomplete dissolution was also recorded. It was revealed that for Cu-Ag the cryogenic temperature effect during strain at constant pressure doesn’t contribute to increase the content of Ag in solid solution. But for Au-Co this effect is reversal. More than that each of the following factors promote to increase the Co content in solid solution: increasing of pressure, reducing the temperature to cryogenic one, increasing of the applied strain and, of course, increasing of Co content in original mixing.

Poster Session

XRF with SR micro beams in the study of geological samples.

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Micro beams of synchrotron radiation at the experimental station of elemental analysis in the storage ring VEPP-3 obtained using polycapillary focusing optics. The minimum sizes of the exciting radiation spot with an energy of 8-25 keV on the sample surface is 10x15 square microns. This allows us to make detailed studies of the elemental composition of various geological samples: mono crystals, inclusions, annual layers in the bottom sediments and other. A set of analyzed elements depends on the excitation energy. For the energy of 25 keV it is possible to analyze about 30 rock-forming and trace elements: K, Ca, Ti, V, Cr, Mn, Fe, Co, Ni, Cu, Zn, Ga, As, Se, Br, Rb, Sr, Y, Zr, Nb, Mo, Ru, Rh, Pd, Ag using K-series, and Ta, W, Au, Pb, Th, U using L-series. The detection limits for light elements (K, Ca) are estimated to be 500-300 ppm, for Pd, Ag in optimal conditions, the detection limit is about 50-100 ppb. Local microanalysis or two-dimensional scanning is possible using one focusing lens. One can make a three-dimensional study of micro inclusions by using confocal scheme of experimental station (two lenses scheme). Also instrumental functions of polycapillary lenses and confocal x-ray microscope were calculated to vary spot size on the sample (in range from 10 to 100 microns) and confocal volume size.
Small-Angle X-Ray Scattering Study of Principles of Self-Organization of Amphiphilic Block Copolymers Based on Polylactide and Poly(ethylene oxide)

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Amphiphilic block copolymers are able to self-organization in water with a formation of various supramolecular structures such as spherical micelles, rod-like micelles, filomicelles (also called worm-like micelles), polymerosomes, etc. It is possible to control evolution of specific morphologies of the copolymers in water solutions by a variation of the polymer composition, architecture, molecular weight and monomer chemistry. Possibility to produce micelles based on amphiphilic block-copolymers with a defined morphology makes them attractive for applications as drug delivery systems.

The objects of this study were non-toxic, biocompatible, biodegradable amphiphilic diblock (PLLAx-b-PEOy) and triblock (PLLAx/2-b-PEOy-PLLAx/2) copolymers with various hydrophobic/hydrophilic blocks' lengths ratio. The aim of this study was to investigate influence of a composition of the initial block copolymers on the construction and properties of the micellar structures by small-angle x-ray scattering (SAXS). The research work was performed using synchrotron radiation at the P12 beamline at the Petra III storage ring (DESY, Hamburg).

We have revealed that the investigated block copolymers in water solution formed complicated two-level systems consisting of individual micelles and ordered supramicellar structures, properties and parameters of which strongly depend on the architecture and composition of the initial block copolymers. In particular, considerable increasing of the length of the hydrophobic lactide block PLLA from 64 up to 418 units (the length of the hydrophilic block poly(ethylene oxide) was a constant) in the diblock copolymers resulted in a paradoxical decreasing of the periodicity of the ordered motifs, that can be explained by a formation of a folded structure of the long PLLA block. Increasing of the length of the hydrophobic lactide block PLLA from 30 up to 120 units in the triblock copolymers did not lead to a change of the system spacing. All block copolymers demonstrated a high degree of the polydispersity. For the investigated block copolymers based on PLLA and PEO micelles the main relatively narrow fraction was presented by micelles with diameter of about 20 nm, but there were also larger scattering objects with the sizes up to 150 nm in the solution.

As a result, on the base of SAXS data, dynamic light scattering, atomic force and cryo-transmission microscopy data a theoretical model of the system was suggested.

This work was supported in part by Russian Foundation for Basic Researches, project 16-03-00375.
Due to the presence of a ripple on the output of various power sources, and mechanical vibrations the VEPP-3 wiggler beam is subject to certain perturbations (oscillations). Such disturbances can distort the results of precise experiments. At the same time the existing stabilization system of the SR beam works with a large averaging times and almost not sensitive to these disturbances. The differential high-speed detector based on pin-photodiodes and electronic processing circuits (that provide filtering and correlated double sampling of the signals) was developed for observation of the SR beam vertical oscillations. Detector is supplemented by electromechanical system for automatically adjustment of the sensor in beam center and allows to record slow displacement with a resolution of up to 0.2 microns.

The measurements showed the presence of perturbations on the harmonics (50, 100, and 300 Hz) and subharmonic (25 Hz) of power supply. In addition, the frequencies of 20 or 21.5 kHz were observed too, that is possibly related to the sources of the inverter type. It is expected that in the future this system can be connected to fast correctors of VEPP-3 storage ring for feedback suppression of the beam oscillations.

**Poster Session**

**Experimental modeling of the impulse diffraction system with a "white" SR beam.**

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Some matrix detectors (CCD and CIS) have direct X-ray sensitivity and can operate as dispersionless spectrometers with a very large number of elements (10E6 and more). In the case of the impulse diffraction such detector can simultaneously record a significant number of independent events. In the reading the position and energy of the absorbed X-ray photon may be restored for each event. The mathematical processing of the received data with respect to the Bragg condition can theoretically lead to the coordinates of the events of one fixed energy and to form an analogue of powder diffraction patterns. In this case the registration can be performed on the “white” SR beam for a short time (up to 1 ns or less).

The possibility of using of the 2000-element linear CCD ILX511 (Sony) as X-ray dispersionless spectrometer allowed to conduct simulation experiments to obtain diffraction patterns of graphite, boron nitride and TNT-hexogen solid detonation products. SR beam was modulated by a mechanical chopper and statistics were provided by a multiple recording with on-line processing.

**GLOBAL RESEARCH INFRASTRUCTURES (GRI) ON NEW ESFRI ROADMAP EUROPEAN PERSPECTIVE**

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The term research infrastructures (RI) refers to facilities, resources and related services used by the scientific community to conduct top-level activities in all fields of science. This definition covers major scientific equipment or sets of instruments; knowledge-based resources such as collections, archives or data banks; e-Infrastructures (networks, computing resources, software). Some research facilities, particularly in physics or astronomy (called global research infrastructures - GRI) are so large, complex or expensive that they require international cooperation for construction and operation and it is being impossible for one country or region alone to build and operate these facilities. The examples of GRIs are CERN: the European Organization for Nuclear Research; JINR: the Joint Institute for Nuclear Research; ESRF: European Synchrotron Radiation Facility; SKA: Square Kilometer Array, the next generation radio telescope. The efficient planning, design, construction and operation of the global research infrastructures requires not only a sharing of costs, but also the sharing of knowledge. A Group of Senior Officials (GSO) on GRIs established in 2008 strives to reach a common understanding on matters such as governance, funding and management of global research infrastructures. Russia is the member of the GSO group. According to the last GSO Progress report 2015, the current list of the national based research infrastructures of global interest covers 39 RIs from 14 countries; among them there are six Russian Mega science projects. A key area of Russia-EU S&T cooperation involves the joint development of GRIs. Russia is a partner of a number of research infrastructure initiatives located in Europe: the EU X-ray Free-Electron Laser (XFEL); the Facility for Antiproton and Ion Research (FAIR); the International Thermoneutron Experimental Reactor (ITER); the Large Hadron Collider (LHC, CERN); the European Synchrotron Radiation Facility (ESRF). All these projects have a global dimension and participants and are located on the roadmap of ESFRI, the European Strategy Forum on Research Infrastructures. Since its foundation in 2002, ESFRI has played a leading role in coordination of research infrastructures across Europe and development of integrated and coherent approach for policy making in this field. The new ESFRI roadmap 2016 consists of 21 ESFRI Projects with a high degree of maturity and 29 ESFRI Landmarks - RIs that reached the implementation phase by the end of 2015. The Landmarks in the area of physical sciences and engineering cover 11 internationally well-known facilities: E-ELT European Extremely Large Telescope; ELI- European Light infrastructure, EMFL- European magnetic field laboratory; ESRF UPGRADES: European spallation source; European XFEL, FAIR, HL-HLC; ILL20/20, SKA; SPIRAL 2. The impact of many of the ESFRI research infrastructures is global and this has prompted a reflection in two main fora: the Global Science Forum of OECD and the GSO group tasked with defining a strategy for GRIs. Developing excellent RIs for physical sciences and engineering is one of the top European priorities. Bringing together scientists and users of the Europes major projects with their counterparts all over the world has an intention to generate new synergies that will stimulate a truly global integration of existing infrastructures. The key of this integration process will be the efficient access to and the open sharing of data and information produced by the RIs. This work was supported under the grant No. 14.572.21.0004 of the Federal Targeted Program for Research and Development in Priority Areas of Development of the Russian Scientific and Technological Complex for 2014-2020.

Biomedical application of SR and THz radiation

**Synchrotron Small-Angle X-Ray Scattering as Universal Instrument of Structural Analysis of Bio and Nanosystems**

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Small-angle X-ray scattering is a universal diffraction method for studying the supra-atomic structure of matter. The potential of this technique has greatly increased in recent years due to the development of bright synchrotron radiation sources. The extensive use of these sources, in combination with new techniques for interpretation of scattering data and structural modeling, made small-angle scattering one of the most effective analytical methods to study nanoscale structures. In the present work, after a brief outline of the basic principles of small-angle scattering by isotropic dispersed nanosystems, two areas of nanodiagnostics, in which the progress in the small-angle experiment and the latest techniques for interpreting scattering data has...
become pronounced in recent years, will be demonstrated. These areas - the analysis of the structure of biological macromolecules in a solution and structural studies of metal nanoparticles synthesized in polymer and aqueous media - are illustrated by examples of practical biological and nanotechnological applications.

This work was supported in part by Russian Foundation for Basic Researches (projects 15-54-74002 EMBL, 16-03-00375, 16-03-00379 and 16-29-11765).

Poster Session

SAXS study of Escherichia coli Dihydrolipoamide Dehydrogenase: structural characteristics and molecular docking

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Dihydrolipoamide dehydrogenase from Escherichia coli (LpD) is a bacterial enzyme that is involved in three different multi-enzyme complexes that catalyze similar decarboxylation reactions of 2-oxoacids. All of these complexes comprise three enzymes known as [U+FFFD], [U+FFFD], and [U+FFFD], where LpD is the [U+FFFD] component and the E2 subunit is used by LpD as the lipoamide-containing protein substrate. The [U+FFFD] and [U+FFFD] subunits have different structures in different complexes, whereas the [U+FFFD] protein is essentially the same in all of the complexes [14]. The pyruvate dehydrogenase complex from Gram-negative bacteria (for example, from E. coli) is composed of 24 E1 subunits and 24 E2 subunits, whereas the multiplicity of E3 remains unknown. According to different estimates, there are 12 or 24 E3 subunits; i.e., E3 may consist of six dimers or six tetramers. It was shown that E. coli LpD exists as a dimer in the crystalline state [5]. However, the solution structure of this protein was unknown. The aim of the present study is to investigate the behavior of LpD in solution, i.e., under near-physiological conditions, by small-angle X-ray scattering (SAXS) and complementary methods. Using modern techniques for the interpretation of SAXS data and analytical ultracentrifugation we determined that in solution LpD exists as an equilibrium mixture of a dimer and a tetramer. The tetramer structure was determined by modeling SAXS data and molecular docking. The results obtained by these two methods correlate well with each other. It was shown that there is the relationship between the oligomerization of the protein in solution and its functional properties. In particular, the possible flexibility of the tetramer follows from the stoichiometric and functional demands of the multienzyme complexes containing LpD as a component.

This work was supported in part by Russian Foundation for Basic Researches (projects 15-54-74002 EMBL, 15-04-01406, 15-04-00563).


Poster Session

Small-Angle X-Ray Scattering Study of Carbosilane Dendrimers in Hexane Solution

Author(s): Dr. SHTYKOVA, Eleonora

Small-Angle X-Ray Scattering Study of Carbosilane Dendrimers in Hexane Solution

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Small-Angle X-Ray Scattering Study of Carbosilane Dendrimers in Hexane Solution

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Small-Angle X-Ray Scattering Study of Carbosilane Dendrimers in Hexane Solution

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Dendrimers are three-dimensional, highly branched, monodisperse macromolecules with a well-defined regular structure. They possess unique properties due to its size, composition and controlled functionalization. Among the variety of dendrimers, carbosilane dendrimeric macromolecules attracted great attention for their reactive functional groups, chemical inertness of the molecular skeleton and possibility of medical and biological applications [1, 2]. In the present work three-dimensional structure of monodisperse carbosilane dendrimers of zero, third and sixth generations were studied by small-angle X-ray scattering (SAXS) in hexane solution. Using modern methods of SAXS data interpretation, including procedure of ab initio modeling, spherical shape and internal architecture of the dendrimers were revealed depending on the generation number and on the number of cyclosiloxane end groups constituting a shell of the macromolecules. With the help of a computer simulation a possibility of shape reconstruction of the dendrimer molecules in clusters has been considered, and the analysis of the intermolecular interaction of the dendrimers in solution was performed applying the latest developments of the method of molecular tectonics.


Poster Session

Ellipsometric measurement of the complex refractive index of liquids in the terahertz range

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Complex refractive index of various liquids has been measured in the THz range using the ellipsometric measurement system with an internal reflection silicon-prism with the Novosibirsk free electron laser being a source of monochromatic radiation. The measurement technique has been optimized both theoretically and experimentally for water solutions. Precision of the measurement of the absolute values of the real and imaginary parts of the refractive index (n, k) equal to 0.01 is achieved experimentally.

Poster Session

Unusual Properties of Structural Characteristics of Potato Virus A Coat Protein in Solution Revealed by SAXS

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Structural analysis of the Potato Virus A Coat Protein (PVA CP) in solution was performed by small angle X-ray scattering (SAXS). SAXS experiments were carried out using synchrotron radiation at the P12 beamline at the Petra III storage ring (DESY, Hamburg). Modern techniques for interpretation of scattering data and structural modeling, including ab initio protocol, have been applied in the present work [1]. Obtained values of radii of gyration ($R_g = 8.9 \pm 0.1$ nm for PVA CP at pH 7.8 and $R_g = 8.5 \pm 0.1$ nm for this protein at pH 10.5) are too large to be a characteristic of the isolated protein macromolecule with molecular mass of 30.26 kDa calculated from the amino acid sequence of PVA CP. The average molecular masses (MMs) of the solutes were obtained by us (i) from the extrapolated I(0) and (ii) from the excluded (Porod) volumes $V_p$ of the species. MMs calculated by these 2 independent methods clearly indicate formation of large aggregates containing dozens (30-60) of individual PVA CP macromolecules. Thus, the overall parameters ($R_g$, excluded Porod volume, MMs) point to the existence in the solution of large particles consisting of many protein macromolecules. On the other hand, Porod plots as well as Kratky plots revealed rather compact particles with definite shapes though less expressed for PVA CP at higher pH. It means that possibly we observed not aggregates, which as a rule do not possess a certain shape, but we can see a formation of some kind of ordered structures, most possible short virus-like particles (VLPs). The structure and overall characteristics of the VLPs depend strongly on such solution condition as pH: while a scattering curve from the sample at pH 7.8 demonstrates intensity pattern characteristic for a specific compact scattering object, SAXS curve from PVA CP changes dramatically at pH 10.5 and corresponds to a partly disordered body without clearly defined boundaries: the CP associates at pH 7.8 are more compact then those at pH 10.5. The shape of PVA CP at lower pH obtained by ab initio method is closed to a cylinder, thereby reproducing the shape of the virus, i.e. this self-assembling in solution to short VLP is an intrinsic biological property of the PVA CP allowing it to create an envelope to protect genetic material of the virus. Increasing pH leads to a partial distortion of this virus-like ensemble: the whole shape of the PVA CP associates at pH 10.5 becomes loose and splits into separate spherical parts. Thus, according to McDonald and Bancroft [2, 3], PVY CP at pH 10.5 disintegrates into monomers, destroying the structure of the VLP and pointing to the way of the virus deactivation. This work was supported in part by Russian Foundation for Basic Researches (projects 15-54-74002 EMBL, 15-04-01406, 16-03-00375 and 16-04-00563). References [1] Blanchet, C.E. and Svergun, D.I. (2013), Annu. Rev. Phys. Chem. 64, 37-54 [2] Mc Donald JG, Beveridge TJ, Bancroft JB.(1976), Virology, 69 (1), 327-331. [3] Mc Donald JG and Bancroft JB. (1977), J. Gen.Virol., 35, 261-263.

Poster Session

Characterization and transformation of Terahertz Bessel beams with angular orbital momentum

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Recently, using silicon binary axicons with spiral zones, we transformed a NovoFEL Gaussian beam into Bessel beams with orbital angular momenta $l = 1$ and $l = 2$. In this paper we study the propagation of beams through random media and investigate optical systems which allow extending the length of non-diffractive beam propagation.
Poster Session

The role of cholesterol recognising amino acid consensuses on amphipathic structures of matrix proteins in raft membrane organisation of some enveloped viruses

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We have recently revealed the amphipathicity of six CRAC containing alpha-helices of influenza virus (IFV) M1 protein, and suggest that the phenomenon may play a role in supporting both raft structure of the membrane and organising regular structure the M1 protein shell in the virion [1]. Computer modelling and comparative analysing 3D structures of the matrix proteins was carried out for three taxonomically different enveloped viruses with raft-type membranes, IFV, Newcastle disease virus (NDV), and Human immunodeficiency virus (HIV), to disclose amphipathic CRAC containing 3D configurations in their structures, and to identify putative motifs, which may be specifically involved in their interactions with raft membranes. Common structural feature of all three proteins was found to be the amphipathic structure of their CRAC motifs, and localization of the outer CRAC regions on the opposite sides of their globules. Notably, in contrast to IFV M1, one CRAC motif on NDV M appeared on the amphipathic beta-sheet structure. To verify our approach we also comparatively tested the M protein structure of the non-raft vesicular stomatitis virus: the disclosed surface CRAC motif appeared no amphipathic configuration. Small-angle X-ray structural analysis (SAXS) of IFV and NDV matrix proteins in solution gave further evidence in support of our conclusions. M1 protein exists in solution as a monomer with a compact NM-fragment and an extended and partially flexible C-terminal domain. SAXS analysis demonstrates that CRAC motifs of the M1 protein are located on the opposite sides of the NM-fragment perpendicular to the C-domain. NDV M protein dimerizes in solution, and its surface CRAC motifs have anti-parallel location on the opposite sides of the dimer. Thus, such localization of CRAC motifs of two different proteins with two different organizations in solution allows these matrix proteins to interact both with the membrane and with the internal components of the virus. Moreover, our results suggest that oppositely situated CRAC motifs in pairs of both monomeric IFV M1 and dimeric NDV M in the virions may serve as anchors between two neighbouring raft platforms consolidating and fastening the envelopes of the viruses. CRAC motifs on the surface of these proteins may be involved in arranging their regular oligomeric structures both in the virions and in solution. This work was supported in part by Russian Foundation for Basic Researches (projects 15-54-74002 EMBL and 16-04-00563) References 1. T. Tsfasman, V. Kost, S. Markushin, V. Lotte, I. Koptiaeva, E. Bogacheva, L. Baratova, V. Radyukhin. Amphipathic alpha-helices and putative cholesterol binding domains of the influenza virus matrix M1 protein are crucial for virion structure organization, Virus Research, 2015, v. 210, pp. 114118.

References

FEL-based study and THz radiation application

Pump-probe setup for far-infrared subnanosecond time-resolved spectroscopy at the Novosibirsk free electron laser

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A one-color pump-probe system has been commissioned at the Novosibirsk free electron laser facility. Results of current experiments on the study of dynamical absorption of semiconductors will be described.

**Poster Session**

**New Insights into Nature of Self-Assembly of Influenza Virus Matrix Protein M1 at Different Conditions: SAXS & AFM Study, and Modeling**

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Influenza A viruses are important pathogens that still rank among the major global health problems. Matrix protein M1 in the virus particles is one of the most important and abundant proteins broadly involved in essential processes of the viral life cycle. It makes the structural investigation of the M1 protein particularly important. We employed synchrotron small-angle X-ray scattering (SAXS) and atomic force microscopy (AFM) to study the structure of M1 at different conditions. The low-resolution structural models built from the SAXS data reveal a structurally anisotropic M1 molecule consisting of a compact NM-fragment and an extended and partially flexible C-terminal domain [1]. We demonstrated also that even at low pH the M1 monomers co-exist in solution with a small fraction of large clusters possessing a layered architecture similar to that observed in the authentic influenza virions. AFM analysis on a lipid-like negatively charged surface reveals that M1 forms ordered stripes correlating well with the clusters observed by SAXS [1]. Low pH condition occurs at the very beginning of cell infection leading to an acid-triggered fusion of the viral membrane. Moreover, change of pH was found to serve as a impuls allowing M1 to carry out its multiple functions in the uncoating, nuclear transport, and assembly of the viral ribonucleocapsid [2]. Revealed by us helix-like shapes could be treated as pre-matrix protein superstructures, whose formation is an intrinsic biological property of the M1 protein. It can be assumed, however, that the oligomerization of M1 should strongly depend on pH and on the protein charge. That is why it was important to analyse the structure and self-assembly of M1 at gradually changing pH (up to the neutral pH condition) in solution and on the bare mica surface using SAXS and AFM, correspondingly. We found that the oligomerization processes occur in a similar way in the solution and on the substrate, and quantitatively described these processes. Moreover, pH 6.0 was found to be the condition at which binding between M1 molecules starts to break. Our results provide new insights into the mechanism of M1 to form matrix and virus-like particles alone without partners and give a basis for a further analysis of the hierarchy of M1 in the virus life cycle.

This work was supported in part by Russian Foundation for Basic Researches (projects 15-54-7402 EMBL and 16-04-00563)


**Poster Session**
THE USE OF LABORATORY-BASED X-RAY COMPUTED TOMOGRAPHY FOR THE DIAGNOSTICS OF X-RAY REFRACTIVE OPTICS

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A promising direction of development of modern X-ray optics is an optical system, based on the compound refractive lenses (CRLs). Main applications of CRLs are microfocusing and X-ray microscopy. In order to improve a technology of X-ray optics manufacturing it is urgent to develop an effective approach of precise nondestructive CRL metrology. At this work, we report the assessment of the efficiency of non-destructive inspection - a high-resolution x-ray computed tomography (HRXCT). This technique allows to realize non-destructive testing irregularities, the presence of voids, inclusions in lenses material and at the same time to analyze the shape and geometric parameters: the coaxiality, the distance between the refracting surfaces and shape of refractive surfaces. HRXCT was performed using the X-ray inspection system YXLON designed for generating high-quality X-ray images with the 1 m resolution. We present the CT imaging results of lenses made of high-purity aluminum with the parabola apex radii of 50 m. Comparing the obtained images with specified requirements of the geometrical characteristics of the lenses, the quality of the refractive surface and internal size of defects allows us to estimate the lens manufacturing quality.

Poster Session

Development of laboratory Metrology for X-ray refractive lenses

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The focusing of X-ray synchrotron radiation is one of the main problems of X-ray optics. Development of compound refractive lenses (CRL), proposed twenty years ago [1], should be considered as one of the promising solution to this problem. Despite the successful development of refractive optics for X-ray nano-focusing [2] and high-resolution X-ray microscopy [3] the diffraction resolution limit for CRL has not been achieved. As already noted [4], imaging properties of the CRL depend on the refractive properties of the used materials, and on the accuracy of the manufacturing technique of the lens optical surface (figure errors). To improve the surface quality, dedicated metrological methods to measure geometric characteristics of the CRL parabolic profile are required.

In this work aluminum and beryllium refractive lenses with a parabolic profile (small radii of curvature R = 50 ? 1000 ?m) made by pressing technique was examined. A comparison of different metrological methods to study lenses profile is presented. Proposed comprehensive laboratory metrological approaches allow to control the quality of the lens profile and to make the input quality control of press tools.


Radiation sources and Centers

Novosibirsk Free Electron Laser unique source of the terahertz and infrared coherent radiation

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A powerful free electron lasers (FEL) has been created at Budker INP. Its wavelength can be tuned over a wide range in terahertz and infrared spectrum regions. As a source of electron bunches this FEL uses multi-turn energy recovery linac which has five straight sections. Three sections are used for three FELs which operate in different wavelength ranges (the first one - 90-240 microns, the second - 37-80 microns and the third - 5-20 microns). The first and the second FELs were commissioned in 2003 and 2009 respectively. They operate for users now. The third FEL is installed on forth accelerator track which is the last one and electron energy is maximal here. It comprises three undulator sections and 40 m optical cavity. The first lasing of this FEL was obtained in summer, 2015. The radiation wavelength was 9 microns and average power was about 100 watts. The designed power is 1 kilowatt at repetition rate 3.75 MHz. Radiation of third FEL has been delivered to user stations recently. The third FEL commissioning results as well as current status of the first and second FELs and future development prospects are presented.

Poster Session

Source-Based Calibration of the Soft X-ray Detectors at the SSTRC

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The development of the procedure of absolute calibration of the soft X-ray detectors is presented. In our measurements, the detector is positioned in a calculable synchrotron radiation flux behind filters of well-known transmittance. The spectral responsivity of the detector is restored on the basis of measurement data using a set of integral equations. Previously, this procedure was used for calibration of the silicon photodiodes SPPD11, which had a relatively low sensitivity and large dark signal. Currently, the calibration procedure is developed for the calibration of a diamond type detectors. The report comprises description of the program for the data processing and solution of the system of integral equations. The preliminary results of the calibration of the absolute sensitivity of fast coaxial-type diamond detectors is presented too.

Invited Talks

20 years of X-ray refractive optics: Status and New opportunities for diffraction limited X-ray sources.

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After the first successful experimental demonstration 20 years ago [1], the use of X-ray refractive optics has rapidly expanded and they are now in common use at 15 synchrotrons in 10 countries. This development has intensified after the successful implementation of transfocators - tunable devices based on refractive lenses [2]. In addition to traditional micro-focusing applications, the transfocators can provide the following beam conditioning functions in the energy range from 3 to 100 (200) keV: - condensers with a tunable beam size, - micro-radian collimators, - low-band pass filters - monochromator [2] - high harmonics rejecters [3] New advanced parameters of the beam provided by the diffraction limited sources XFELs and new synchrotrons with the reduced horizontal emittance will open up a unique opportunity to build up a new concept for the loss-free beam transport and conditioning systems based on in-line refractive optics. Taking an advantage of the substantially reduced horizontal source size and the beam divergence these new systems integrated into the front-end can transfer the photon beam almost without losses from the front-end to any further secondary optical systems (mirrors, crystals, lenses etc.) or directly to the end-stations. Evidently, beamlines will benefit from the possibility to include active moveable lens systems in the front-ends. In this regard, development of diamond refractive optics is crucial [4,5]. The implementation of the lens-based beam transport concept will significantly simplify the layout of majority of the new beamlines [6]. It will also allow a smooth beamlines transition from the present beam parameters to the upgraded ones, avoiding major optics modifications [7]. The field of applications of refractive optics is not limited to beam conditioning, but can be extended into the area of Fourier optics, as well as coherent diffraction and imaging techniques [8-12]. Using the intrinsic property of the refractive lens as a Fourier transformer, the coherent diffraction microscopy and high resolution diffraction methods have been proposed to study 3-D structures of semiconductor crystals and mesoscopic materials [1214]. Another promising direction of refractive optics development is in-line X-ray interferometry. Recently proposed bi- and multi-lens interferometers can generate an interference field with a variable period ranging from tens of nanometers to tens of micrometers [15,16]. This simple way to create an X-ray standing wave in paraxial geometry opens up the opportunity to develop new X-ray interferometry techniques to study natural and advanced man-made nano-scale materials, such as self-organized bio-systems, photonic and colloidal crystals, and nano-electronics materials. As a classical interferometer it can be used for phase contrast imaging and radiography. Finally it can be useful for the coherence characterization of the X-rays sources and free electron lasers.

Poster Session

THE CRL’S OPTICAL PROPERTIES OF BERYLLIUM GRADES IS-50M AND O-30-H

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X-ray refractive lenses [1-3] are widely used as beam transport and beam conditioning optics at the new generation synchrotron radiation sources. In order to extend their use for microscopy applications the significant improvements of the optical properties in terms of shape quality and bulk homogeneity are needed. It turned out that beryllium, as a low-Z element, is the most suitable for lens manufacturing. However, beryllium being a sintered material has an internal grain structure with a relatively high content of the beryllium oxide, which create a strong small- and ultra-small angular scattering. In this work we report results of study of different types of beryllium (Materion Brush and Russian ROSATOM grades) using small angle scattering and phase contrast imaging techniques. It was shown that Be with a reduced oxygen content is preferable for microbeam applications. As for the full-field microscopy, Be with small grain sizes is required. In this view a so-called nano-beryllium with grain sizes smaller than 50 nm is very promising for lens manufacturing.

References


Poster Session

Ferrum fluorides as nanostructured conversion cathodes: in situ XAFS and XRD study using synchrotron radiation.

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Transitional metals fluorides have been known from 1960s in the field of rechargeable batteries. They belong to the conversion materials, and by reaction with lithium can be reduced to the mixture of zero valent metal and lithium fluoride, resulting the so-called more-than-one electron reaction. However, possibly due to the lack of nanostructuration of commercial compounds, fluorides gave the way for more immediately interesting and promising materials.

Since 1960s various technologies for producing of nanocompounds have been developed, so fluorides became one of the leading research directions for Li-ion industry. Most efforts are being put into iron trifluoride for several reasons: its more environmentally friendly than others, cheaper, requires reasonable synthesis conditions, has relatively low polarization, etc.

In our study we pursue the goal to determine structural changes which take place inside the full cell and to look at those processes in situ during cycle. Experiment was carried on the B station of BM01 (SNBL) beamline at ESRF, Grenoble, as a mixed XAFS/XRD experiment. Samples were prepared with synthesis of iron fluoride nanoparticles inside reduced graphene oxide sheets which increases conductivity. The material offers a stable discharge energy of 600-700 Wh/kg over 100 cycles, which is higher than the widely applied cathode materials (300-500 Wh/kg). Each sample we cycled with 20 mAh/g in the 1.2-4.2 V range, while measuring Fe K-edge XAFS spectra in transmission mode and XRD patterns with 15 minutes interval. For measurements we used self-made test cells with glassy carbon windows connected to Gamry potentiostats responsible for cycling and data acquisition.

The Li intercalation in the first discharge is different from subsequent cycles. Up to 1.8 V, a maximum of 0.66 Li will be inserted into the channels of the framework structure of initial FeF₃ ? 0.33H₂O (from synthesis conditions). Then peaks in the XRD patterns disappear and nanocrystalline LiF and Fe phases will form below 1.8 V. On charge, the ReO₃-type FeF₃ phase with higher density will form instead of the open-framework structure. On second and subsequent discharge reactions, we will form LiFe₂F₆ instead, which then also converts to LiF/Fe. This is of theoretical nature, because of the nanocrystalline structure of the involved phases, which is too small to detect in XRD, and also similar Fe-F6 environments, which make the FeF₃ phases very hard to distinguish in XAS.

Results of the x-ray studies were associated with cycling data to obtain structure-charge state dependency. HTB structure of the as-prepared material has open intercalation channels as a result, full electrochemical reaction can be separated into initial intercalation of one Li-anion per formula unit and following conversion reaction involving two more Li-, which gives us 3LiF/Fe mixture and a complete three electron transition. To prove this we performed principal component analysis (PCA) on the series of XAFS experimental spectra. We have used FitIt software to mathematically decompose the series of the Fe K-edge spectra at different voltages into independent sub-spectra. It was found that all spectra for discharge process can be reproduced as a combination of three components. First component corresponds to HTB structure, second to the intercalated structure with Fe²⁺ charge state and the third one corresponds to metallic Fe. We have observed that pure Fe forms after HTB conversion to intercalated phase. We also performed a set of ab initio calculations and DFT modeling for different concentrations of Li in cathode material. Theoretical simulations for the Fe K-edge XANES are in progress now to figure out if we can distinguish intercalated HTB structure from the LiFe₂F₆ phase in the XANES data.

Biomedical application of SR and THz radiation

Biomedical Applications of Terahertz Radiation

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Early experiments by pioneers of THz imaging demonstrated that the technique could have a variety of applications, including medicine and dentistry - terahertz images of biological tissues showed contrast between lean meat and fat; changes in refractive index of teeth at THz frequencies allow for the detection of early decay.

Since then there have been many studies of tissues, from wax embedded tissue block, histopathological samples, excised tissues and even some in vivo imaging. Some of this most significant work revealed contrast between regions of healthy skin and basal cell carcinoma, the most common form of skin cancer, in vitro and in vivo. Further, other work on excised breast carcinoma has shown good contrast between tumours and normal tissues. This has led to the development of a prototype handheld (intra-operative THz) imaging probe for use during breast surgery.

The high absorption of THz by water in this range (23 mm⁻¹ at 1 THz) makes transmission imaging through a body impossible, but it is the difference in absorption due to water content which explains the contrast seen between muscle and adipose tissue and between tumour and normal tissue. Terahertz spectroscopic measurements of carcinoma show significant differences in the frequency dependent refractive index and absorption coefficient. Such contrast on the surface is often obvious at optical frequencies but it is the ability of THz to penetrate below the surface that provides the potential for medical imaging, in particular, of epithelial tissues (cancer with origins in tissue surfaces both external and internal), which includes skin, breast and colon cancer, accounts for 85% of all cancers.

It is the ability of THz to penetrate just below the surface that provides the, as yet not fully realised, potential for tissue imaging, visualisation of the subsurface spread of tumours and other applications. Full understanding of the contrast mechanisms in tissues and their physiological significance will enable THz to fulfil its potential for medical applications.

To date in vivo measurements have been limited due to the restricted nature of imaging systems. Compact, mobile THz imaging systems are now being developed which allows for TPI measurements in a clinical setting.

Invited Talks

Novosibirsk free electron laser as a user facility

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Nowadays, all three laser systems of the Novosibirsk free electron laser facility are in operation. First laser system generates tunable monochromatic radiation in the terahertz spectral range from 90 to 240 micrometers, whereas the spectral range of the second laser spans a region from 40 to 80 micrometers. Both these systems are available to users. The third laser has been commissioned, and its radiation (5-20 micrometers) was transmitted to the user stations recently. In this report we describe the instrumentation available to researchers at the user stations, and provide examples of experiments carried out in the two years that have passed since the previous conference.
X-ray Spectroscopy

XAFS investigation of charge state and local atomic geometry of Pt in the series of synthetic minerals.

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The aim of the present project is to characterize the charge state, electronic and local atomic structures of Pt in the key chemical systems, critically important for ore geology, geochemistry, and for material sciences. This project will benefit from our experience in the synthesis and crystal growth of chalcogenides. Using the novel synthesis methods developed by members of our team we have synthesized all possible crystalline compounds in the systems: Pt-S, Pt-Se, Pt-As, Pt-Sb, Pt-Bi, Pt-In, Pt-Ga, Pt-Sn and some other compounds. Most of these compounds are synthetic analogues of natural minerals and characterized by powder X-ray diffraction. In the current project we measured X-ray absorption spectra at Ll, LII and LIII edges for all 30 samples. We used EXAFS spectra for detail investigation of local atomic structure. XANES spectra were used to get information concerning electronic structure of Pt atom and its chemical bonding. Systematic comparison of the position and intensities of spectral features in XANES, calculated by DFT charge properties and valance states were carried out.

FEL-based study and THz radiation application

Carrier dynamics in doped Ge measured at the free electron laser facility FELBE

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Cooled germanium (Ge) photoconductive detectors are one of the most sensitive detectors at terahertz (THz) frequencies. They are widely used in laboratory spectroscopy and imaging experiments. The speed of a Ge photoconductive detector is set by technical limitations such as the bias circuit, the geometry of the detector crystal and the electric field applied to the detector. The recovery speed of the detector material is, however, fundamentally limited only by the lifetimes of the intraband relaxation of the free charge carriers within the valence or conduction band and by band-to-impurity relaxation (capture) down to the impurity ground state. Therefore, capture and intraband relaxation processes have been measured for different dopants in uncompensated and compensated n- and p-type Ge by a pump-probe technique at the free electron laser facility FELBE.
Duration of coherent synchrotron radiation pulses accessed via time-resolving and correlation techniques

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Coherent synchrotron radiation (CSR) is emitted from bunches of relativistic electrons in a storage ring. The CSR spectrum depends on the shape of the electron bunch from which the CSR radiation is emitted. Instantaneous monitoring of the form and duration of the field transients in CSR pulses is important for applications in pump-probe studies of carrier dynamics in semiconductors and superconductors, where the required excitation must be simultaneously unipolar and much shorter than the expected relaxation time. Here we use two types of ultra-fast detectors, Schottky diodes and superconducting microbridges, to evaluate the duration of transient field-oscillations in CSR pulses under stable average intensity of the CSR source by means of time-resolving and field-correlation techniques. We show that both methods deliver close results.

X-ray apparatus

Coherent hard X-ray microscopy for the characterization of mesoscopic materials

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We present a coherent high energy X-ray microscope to study the wide range of natural and artificial mesoscopic materials that are structured on scales of the order of a few to a few hundred nanometers. The concept of the proposed microscope is based on employing compound refractive lenses allowing to retrieve high resolution diffraction pattern and real-space images in the same experimental setup [1-4]. This idea, well-known for the studies of crystals by high resolution transmission electron microscopy, is the key ingredient of our approach. The microscope operates under a coherent illumination where a diffraction pattern of the specimen is formed in the back focal plane of the condenser and an inverted two-dimensional image of the object is formed by objective lens in the image plane [5]. The diffraction mode is used to investigate the structure over the macroscopic distances and to orient the crystals parallel to the low index direction to perform high-resolution imaging on the local scale. The image formation relies on phase contrast due to the interference of several diffracted beams [6]. A high spatial coherence is needed in the imaging mode to ensure a reasonable contrast. The coherence in terms of the angular source size determines the lens angular resolution (< 1?rad) to get high resolution diffraction patterns. Functioning at 10 - 30 keV, the microscope is one of the branches of the multimodal instrument which is under the development at the ID06 ESRF beamline. It consists of the condenser, the objective lens and two X-ray CCD cameras large area detector for diffraction and high resolution CCD for imaging. Condenser and objective assemblies are comprised of Be parabolic refractive lenses. Switching from the diffraction mode to the imaging is achieved by placing the objective lens into the beam, and the chosen detector. The tunable objective lens offers full-field imaging with variable resolution and field of view. It allows for the identification of features of interest in a
coarse resolution overview before increasing magnification to study these features with maximum resolution. At present, at the maximum magnification a resolution of 100 nm is achieved, but it should be noted that the studies on its improvement are cured out and in the near future we can expect resolution about 30 nm. The microscope was applied for study of natural and synthetic opals, metal inverted photonic crystals and colloidal suspensions [5,7]. The combination of the direct-space imaging and high resolution diffraction provide a wealth of information on their local structure and the long range periodic order. The concept of the hard x-ray microscope emerged concomitantly with the realization that the ESRF source upgrade would, through the greatly enhanced brilliance and fraction of coherent light, open entirely new frontiers in materials imaging [8]. Short acquisition times with modern area detectors allow to extend the microscope to time-resolved studies of the crystallization dynamics, response of the mesoscopic structures to external stimuli such as mechanical strain, temperature jump or temperature gradient as well as external fields.

References

Poster Session

The new control for magnet system of KCSR.

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The running cycle of Kurchatov Synchrotron Radiation Source (KSRS) includes the injection of electrons with energy 80 MeV from the linear accelerator in the booster storage ring Siberia-1, the accumulation of a electron current up to 400 mA and, then, electron energy ramping up to 450 MeV with the subsequent extraction of electrons in the main ring, storage ring Siberia-2, and accumulation there up to 300 mA, and at last the energy ramping up to 2.5 GeV. Several years ago, a modernization of the current system of automated control systems (ACS) has started. This article presents one of the most important parts - the new control subsystem of the magnet system.

Poster Session

SR micro-XRF installation on VEPP-3 storage ring. An approach and difficulties in increasing the spatial resolution.

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X-ray fluorescence microanalysis on synchrotron radiation beams (SR micro-XRF) is a method to explore the elemental composition of samples and objects of various nature with a typical spatial resolution of 15 to 25 μm. Such resolution, available in qualitative micro-XRF (mapping
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of fluorescent signal distribution or exploration of area of interest without detailed consideration of effects caused by absorption by a sample) is defined by the transverse size of the focal spot of x-ray optics used. As a rule, up-to-date (monolithic) polycapillary lenses are applied. This qualitative elemental mapping in some cases can provide useful information. It can be in demand, for example, in reconstruction of paleoclimate (layered samples of bottom sediments, so called varves) and conditions of rock formation in deposit occurrences, as well as in examination of biological objects (hair and large cells), particles of earth and meteorite dust and some other objects [1-2].

The SR XRF experimental station on the VEPP-3 storage ring (the SCSTR) [3] is equipped with the installation “SR micro-XRF”, which is intensely used by a number of users in most of these areas of research. The installation was engaged in an extensive series of experiments [3] using qualitative SR micro-XRF; unique results were obtained in several cases. Two directions of improving the method and installation were selected so far. The first one is development of certified techniques of (semi-) quantitative XRF, and the second one is 1.5-2 time improvement of the spatial resolution due to application of complicated algorithms to process experimental data (so called regularization), including embedded indirect accuracy control.

Regularization is a method of approximate solution to unstable inverse problems. It is based on certain controlled introduction of systematic error into the result. This error is introduced so that its contribution optimally balances [4, p. 52-55] the influence of random noise, because of which the problem cannot be accurately solved by traditional methods. Development of any regularization algorithms implies analysis of feasibility of the above rule (in other words, the convergence of the approximate solution) over the entire range of all input parameters. The algorithm user is assumed to know the rules for use of one or another regularizing procedure (that may expect, for example, normal distribution of noise, absence of systematic errors, special prior constraints to the solution, and others) and adhere to them. Unfortunately, this is not always the case, and application of regularization of algorithms is often blind and inefficient, especially when third-party programs are used.

This work presents an attempt of non-strict but effective practical analysis of the influence of various factors “hindering” the regularization (inaccuracy in the determination of the instrumental function, discretization errors, Gibbs boundary effects etc.) on the process of solving inverse deconvolution problems arising in SR micro-XRF using the Tikhonov algorithm [4, p. 52]. The analysis performed resulted in practical recommendations on the optimal regimes of experiment with improved spatial resolution and highlighted the role of numerical simulation in the control of solving real inverse deconvolution problems.

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References

X-ray apparatus

Development of compact SR light sources in JAI and in UK
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In this overview talk we will describe development of compact light sources of various types, ranging from plasma acceleration betatron light sources to compact SC ERL sources, in the John Adams Institute and in UK. We will describe the accelerator science challenges and progress in
 developments of such sources, in particular in increasing the average flux and repetition rate of such sources and will also illustrate the prospects of practical use of such sources by examples of their application for medical studies.

X-ray Spectroscopy

Probing magnetic sublattices in multiferroic Nd0.5Ho0.5Fe3(BO3)4 single crystal via hard x-ray magnetic circular dichroism

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We present an element-specific magnetization study of the multiferroic Nd0.5Ho0.5Fe3(BO3)4 single crystal. Our experimental results confirm the coexistence of three magnetic subsystems (Fe, Ho, and Nd). It was found that the Ho magnetic moment undergoes the pronounced spin-reorientation transition. At the magnetic field (less than 1 T) the Ho and Nd magnetization curves demonstrate different of the slopes indicating different strength of f-d exchange interactions with iron subsystem. It was shown that the Fe magnetic moment behave like a weak ferromagnet, but not antiferromagnetically. These magnetic findings are relevant to understand magnetism of Nd0.5Ho0.5Fe3(BO3)4 single crystal and other multiferroic ferrobates.

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Radiation sources and Centers

Present Status and Perspectives of Long Wavelength Free Electron Lasers at Kyoto University

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A mid-infrared FEL named as KU-FEL has been developed for energy related sciences at Institute of Advanced Energy, Kyoto University. The wavelength range of KU-FEL is 5-20 micro-m and routinely operated for various user experiments. A compact THz-FEL, which consists of a photocathode RF gun, a bunch compressor and an undulator, is now under development. Present status and future plan of the FEL accelerators at Kyoto University will be presented in the conference.

Biomedical application of SR and THz radiation

The possibility of direct analysis of biological tissues of a few milligrams by SR XRF method

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Development and improvement of analytical methods to explore the element and microelement composition of biological tissues enables obtaining additional information on functioning of systems of organs and tissues. It is the information on the multi-element composition of biopsy material of living organisms that is relevant. Since this material always has very low weight, 4 mg to 0.5 mg of dry weight, such amount of sample material cannot be directly explored by the conventional non-destructive methods of analysis. The works performed use the unique properties of synchrotron radiation, which enable analysis of small-weight samples (a few milligrams), which is virtually impossible with the conventional methods. The SR XRF method enables analysis of small-weight samples and selection of optimal conditions for analysis of the elements. For nine years, the experimental station for X-ray fluorescence analysis (VEPP-3, BINP) has been involved in joint systematic exploration with Meshalkin Research Institute of Blood Circulation Pathology. About 1000 samples of the vascular system in patients with different pathologies have been analyzed: congenital heart disease, coronary heart disease, dilated cardiomyopathy (heart transplant), aortic aneurysm, etc. A new sample-preparation method for direct analysis of biopsy material weighing to 0.5 mg was developed. A technique of analysis of fragments of biopsy and surgical samples by K-lines of S, Cl, K, Ca, Sc, Ti, V, Cr, Mn, Fe, Co, Ni, Cu, Zn, As, Se, Br, Kr, Rb, Sr, and Mo was developed. The results of the determination of the elemental composition of fragments of biopsy and autopsy samples of cardiovascular disease by the SR XRF method gave a method of evaluating the functional state of the heart. It can be used in clinical practice for pre-testing in pathology of heart transplant and in aortic dissection.

Poster Session

SR XRF in identification of trace elements in trace amounts of humic acids

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Humic acids (humic and fulvic acids) are natural substances that perform a number of functions in the biosphere to maintain its stability. This is due to the ability of humic acids to accumulate, deposit, and store carbon and a wide range of trace elements for long, geologically-comparable periods, as well as inhibiting chemical compounds toxic to living organisms. The content of humic acids in soils and natural and man-made deposits is ambiguous. The amount of humic acids can often be deciles (or even less) of percent. This means handling of very large masses, up to a kilogram or more, of soil samples to study. This, in turn, limits the possibility of quantitative research to identify the ecological state of the environment. Since the extracted preparations of humic acids have a weight of milligrams, there arises a problem of non-destructive methods for their investigation and creation of base of preparations of humic acids for their further study. Testing of different methods of analysis (NAA, two-jet arc plasma AES etc.) for determination of the trace element content in humic acids resulted in a conclusion that X-ray fluorescence analysis using synchrotron radiation (SR XRF), which a non-destructive and multi-element method, fits the best to a wide range of problems concerning the condition of the natural environment. It is particularly important to identify elements such as As, Se, Br, Rb, Sr, Y, Zr, Mo, I, Pb, Th, and U, determination of which in humic acids by other methods may be difficult or unreliable. Thus, the use of SR XRF in identifying a wide range of trace elements in humic acids enables, first, using weights of a few milligrams, secondly, extension of the possible range of elements to identify, and, finally, storage of preparations of this natural component, which is very significant in the functioning of the biosphere, for further research.

Poster Session

Mercury in ancient organic materials (Noin-Ula, Mongolia): EDXRF, SRXRF and micro-SRXRF analysis

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Recently the anomalously high content of copper in ancient hairs in royal burials of Xiongnu (I B.C. I A.D., Northern Mongolia) was discovered. Detailed investigation of all types of organic findings from the burials was performed by X-ray fluorescent technique with synchrotron radiation (SRXRF), X-ray microtomography, X-ray absorption spectroscopy (XANES and EXAFS) and electron microscopy. The results revealed the exogenous source of copper in ancient hairs and allow to exclude copper poisoning in lifetime [1]. There were 40 hair plaits in ancient burial site. Mercury was detected in all hair samples (up to 1200 ppm). High content of Hg may be caused by the burial environment as well as the accumulation in lifetime. Information, which can elucidate the probable cause of such a high Hg content in hairs, can be helpful in reconstruction of culture, life and technologies of ancient people. The unique archaeological material demands nondestructive analytical methods, as SRXRF analysis and X-ray microanalysis (SRXRF). SRXRF methodology for Hg determination in archaeological hair samples, mercury concentration in metallic objects from the burial, and information on cross-section distribution of Hg in hair strand (SRXRF) allow to determine probable source of mercury in ancient hairs.


Poster Session

Influence of a LIGA-raster on the spatial distribution of the radiation from a flash X-ray generator

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The development of essentially new methods of manufacturing X-ray optics in the energy range 20-200 keV is a challenge. Namely, the development of manufacturing methods of X-ray rasters with high aspect ratio and, on substrates of any forms, using LIGA technique. A spherical X-ray wave can be formed, placing a foil with microchannels onto a spherical surface either near the X-ray focus or away from the focus. In case of a plane raster, the quasi-plane wave with a divergence determined by the ratio of the diameter to the lengths of channels will be formed. At the same time, for X-ray tubes of all the types, including flash generators applied at the Institute of Hydrodynamics SB RAS for the diagnostic of high-speed and explosive processes, a three-dimensional microraster can be used as a collimator. That allows a decreasing of the visible source size, resulting in an increasing of the spatial coherence of the X-ray beam. In the experiment, a flat nickel raster of 1250 microns thick, whose channels are 80 microns in diameter, and manufactured using LIGA technique, has been applied. For a flash X-ray device with an explosive electron emission and a bias of 150 kV, the angular divergence was reduced from 180 degrees to 3 degrees (FWHM), i.e. 60 times. The quasi-parallel X-ray beam along the axis passing from the center of the focus and perpendicular to the raster was formed. The beam diameter is similar to the focus size in the near field and divergent (with an angular divergence of 3 degrees) in the far field. Because of non-zero raster transparency, a background of scattered radiation has been observed. The intensity of the background is characterized by the sixth order of symmetry that corresponds to the raster symmetry. Thus, the opportunity of the use of microraster manufactured by means of LIGA for the increase of spatial coherence of X-rays is experimentally demonstrated.
Poster Session

SR micro-XRF installation on VEPP-3 storage ring. Possibilities in increasing the spatial resolution.

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To investigate the possibility of increasing the spatial resolution of the installation “SR micro-XRF” [1], we conducted earlier an experiment on the elemental mapping of one-dimensional test sample (a fragment of an RF bipolar transistor chip, containing a number of gold stripes), as well as processing of some of the experimental results (one mapping line) [2-3] using the classical Tikhonov regularization algorithm [4, p. 73]. In this work we present the results of the processing of all data of this experiment (by two variational realizations [4, p. 119] of the Tikhonov algorithm) and a detailed analysis of the results. For example, we performed a numerical simulation of the direct and inverse deconvolution problems. We did that for cases with different resolutions, to study the plausibility of the results of solving the deconvolution problem. We also analyzed the influence of certain effects that cause systematic errors in the calculations and the resulting solution.

As a result of this work, we found out that it was very difficult to practically increase the spatial resolution 3 times (which corresponds to the distance between the gold stripes of the sample and their width of 5 ?m at an FWHM of the lens instrumental function of 15 ?m), while it was theoretically possible, on the “SR micro-XRF” installation with the available intensities of fluorescent signals and noise levels. A quantitative analysis of the reasons for this is discussed in this work and illustrated in detail in a number of model cases. At the same time, in certain experimental mapping regimes, the achievable resolution can be increased 1.5-2 times (provided there are no significant systematic errors in the calculations).

The results of the research will help to plan subsequent mapping experiments (they showed that the data of the mapping conducted earlier were ill-suited to processing because of non-optimal experimental regime, which led to a substantial systematic error with the desired 3-fold resolution increase, because of the edge effects and not too good signal-to-noise ratio).

This work was supported by the RFBR Grants 14-02-00631, 16-32-00705. The work by D.S. Sorokoletov was supported by a scholarship of the President of the Russian Federation (SP-2761.2016.2).

References

Titanium and zirconium alloys are widely used in medicine, chemical and oil industries, aircraft industry, in nuclear power industry. Hydrogen penetration and accumulation in titanium and zirconium products lead to its properties and can lead to the delayed fracture due to the hydrogen embrittlement [1-4].

Degree of hydrogen impact on the titanium and zirconium properties depends on its concentration and condition in metal. Hydrogen saluted in lattice has a low impact on properties of the metal. However, hydrogenation under operation of titanium and zirconium alloys often occurs with hydrides formation. As a consequence, investigation of hydrides formation and dissociation in titanium and zirconium has both fundamental and practical interest.

Samples of commercially pure titanium alloy and Zr1%Nb zirconium alloy were instigated in this work. Hydrogenation was done by gas-phase method at temperature 600 C and hydrogen pressure 1 atm. Spectrum of hydrogen thermo-stimulated desorption from samples after hydrogenation were obtained with the help of automated complex Gas Reaction Controller LPB with mass-spectrometer RGA100.

In situ diffraction measurements were carried out in the process of samples heating at the station “Precision diffractometry II of the Institute of Catalysis of the Siberian Branch of the Russian Academy of Science at the 6 synchrotron radiation channel of the VEPP-3 electron storage ring. Comparison of the data obtained in the analysis of hydrogen desorption curves with data of the phase transitions in the zirconium-hydrogen and titanium-hydrogen systems at the heating allowed describing the processes occurring in zirconium and titanium hydrides under thermal treatment.


FEL-based study and THz radiation application

FEL-based study of intervalley elastic scattering of donor excited states in multivalley semiconductors

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FEL-based optical pump-probe technique is usually applied for study of highly non-equilibrium electrons in semiconductors and in particularly for the relaxation of donor or acceptor bound excited states. Here we are focused on intervalley elastic scattering that is typical for donors in silicon crystal. As shown such process results in an additional feature in a pump-probe response that has been ignored so far in the interpretation of data obtained using FELIX and FELBE free electron lasers. On the other hand the knowledge on the elastic intervalley scattering is also important from physical point of view.

Poster Session

New RF gun for Novosibirsk ERL FEL

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In BINP SB RAS the new rf gun making an intensive high-quality electron beam for injecting in Novosibirsk microtron recuperator (ERL) and driving Free Electron Laser (FEL) is made. Bench tests of rf gun confirmed good results in strict accordance with the calculations predicting average current of a bunch of 100 [U+FFFD] energy of particles of 400 [U+FFFD] and an emittance ? 15 microns. The rf gun stand testing showed reliable work, unpretentious for vacuum conditions and stable in long-term operation. The bunch injection system built-in to the existing system of the microtron injector with the static gun and keeping high quality of bunches is developed and designed.

Poster Session

Properties of quarter wavelength coaxial cavity for triode-type thermionic RF gun

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Some infrared FEL facilities apply thermionic RF guns due to their advantage of compactness and cost effectiveness as compared to RF photocathode electron guns. Other remarkable advantages are the long lifetime and low vacuum condition requirements. On the other hand thermionic RF guns are suffering from back-bombardment phenomena, which limits the bunch charge and macro-pulse duration. In order to overcome this disadvantage a novel configuration named as
triode-type thermionic RF gun, which has an additional small pre-bunching cavity around the cathode, was developed at Institute of Advanced Energy (IAE), Kyoto University. The triode concept has already been proved by numerical simulation. For a proof of principle experiment, a quarter wave pre-bunching cavity was fabricated. The cold test of the quarter wave pre-bunching cavity at high and low RF power has been completed. In this work we report results of hot test of the cavity.

X-ray structural analysis

Novel technique for spatially resolved imaging of molecular bond orientations using X-ray birefringence: applications and developments

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Birefringence has been observed in anisotropic materials transmitting linearly polarized X-ray beams tuned close to an absorption edge of a specific element in the material. Synchrotron bending magnets provide X-ray beams of sufficiently high brightness and cross section for spatially resolved measurements of birefringence. The recently developed X-ray Birefringence Imaging technique has been successfully applied for the first time [1] at the Diamond Light Source versatile Test Beamline B16. The orientational order of CBr bonds of brominated guest molecules within crystalline host tunnel structures (thiourea or urea inclusion compounds) has been studied using linearly polarized incident X-rays close to the Br K-edge. Imaging of domain structures [1], changes in CBr bond orientations associated with order-disorder phase transitions [1], and the effects of dynamic averaging of CBr bond orientations [2] have been demonstrated. The setup uses a vertically deflecting high-resolution double-crystal monochromator upstream from the sample and a horizontally deflecting single-crystal polarization analyser downstream with a Bragg angle as close as possible to 45. In this way, the rotation angle of the polarization of the beam transmitted through the sample is measured as in polarizing optical microscopy. The theoretical instrumental background is calculated and compared with experimental observations. The summary of recent results and some perspective developments are presented and discussed.

References:

Poster Session

The End-Station "NanoPES" at the Kurchatov synchrotron radiation source: Present status and prospects

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The End-station “NanoPES”, currently under construction, is intended for the implementation of techniques for angle-resolved photoemission spectroscopy (ARPES), NEXAFS, LEED, SPM microscopy and spectroscopy. The station was projected for fundamental researches in solid state physics, surface science and for processing technological operations in the course of creation devices for micro- and nanoelectronics. The station is located at the bending magnet beamline 6.5 and covers the region of excitation energies between 25 eV and 1500 eV due to a plane grating.
monochromator. The electron energy analyzer used, is a hemispherical analyser PHOIBOS 225 with an energy resolution of 1 meV and an angular resolution of 0.1. The station is provided with a complete set of accessories for in-situ sample preparation, sputtering and deposition of monolayer and submonolayer thin films, as well as AFM and STM microscope for in-situ topography and electronic structure studies of samples. The report shows the optical design, the specifications, the first spectra and discussed prospects of development of the station.

X-ray structural analysis

The study of the phase composition of ceramic materials obtained by SHS from previously mechanically activated reaction mixtures Ti-Cr-B

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In this work, ceramic materials based on borides of transition metals were prepared by SHS compaction from the pre-mechanically activated (MA) of the powder mixtures. The use of [U+FFFD] enables SHS process in the low exothermic systems. The role of MA appeared in a simultaneous increase in heat evolution and heat evolution rate of the combustion reaction, a positive influence on the thermodynamics and kinetics of the process. Kinetics of the SHS process, stages of chemical transformations and structure formation in the Ti-Cr-B system were investigated. It is demonstrated that the interaction of the reagents on the solid phase mechanism takes place at a temperature of the combustion. MA was found to markedly affect the combustion temperature and burning velocity of TiCrB blends. SHS reactions in TiCrB blends with Cr contents above 10% proceeded with involvement of gas transport agents. The role of gas transport was found to grow with increasing MA duration, due to an increase in the amount of boron oxides. The mechanisms of combustion and structure formation in Ti-Cr-B were well characterized using different method such as stop combustion front, SEM, EDS, X-ray photoelectron spectroscopy, XRD including the use of synchrotron radiation. Results, the main reaction is the formation of the titanium borides. Then, the complex borides are formed in the post combustion zone and the secondary structure formation zone. Ternary phases Cr₄Ti₉B and Ti₂CrB₂ were detected. [U+FFFD]₄Ti₉B phase is formed at a chromium content of up to 20%, second boride (Ti₂CrB₂) is formed with increasing chromium concentrations up to 30%. It is found that they are chemical compounds - borides hexagonal lattice (structural types hP2₈ / 5 and tP10 / 2). The technology of compact samples of pre-activated mixture Ti-Cr-B and their properties were studied.

Biomedical application of SR and THz radiation

The study of the nonthermal effects of terahertz radiation on living systems.

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In connection with the increasingly widespread use of terahertz radiation in the practice and the lack of consensus on the biological effects of terahertz radiation, it seems urgent to study in detail the effects of such exposure as model organisms, including bacteria and human cells. We studied the impact of terahertz radiation on E. coli biosensor cells containing plasmids with promoters of stress-sensitive genes controlling the expression of GFP. GFP level was measured by fluorometry. The impact of terahertz radiation was non-thermal, i.e. special care was taken to keep specimen temperature in the 35±FFD range during irradiation so that heat shock genes would not be induced. GFP level was measured by fluorometry. We found that terahertz radiation activates genes associated with oxidative stress response. Results of Ames test and SOS-chromotest indicate that terahertz radiation doses tested no direct action on DNA mutagenic and does not genotoxic. Proteomic analysis, it was found that exposure to E. coli cells terahertz radiation leads to increased expression of 14 genes of rapid response. Among these genes discovered glutamine synthetase gene (glnA). Based glnA gene promoter designed biosensor sensitive to the effects of terahertz radiation. Human embryonic stem cells (hESCs) are extremely sensitive to environmental stimuli, and we therefore utilised this cell model to investigate the non-thermal effects of THz irradiation. We studied DNA damage and transcriptome responses in hESCs exposed to narrow-band THz radiation (2.3 THz) under strict temperature control.

**X-ray structural analysis**

**Nanodomain states of strontium ferrites and their structural transformations**

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Depending on substitution degree and external conditions strongly-nonstoichiometric strontium ferrites have certain features of the local structure [1-3]. It is related to extremely high amount of oxygen vacancies, laying beyond the homogeneity region of perovskite or any of vacancy-ordered phases. That vacancy concentration tend to be adopted by the structure through formation of local ordering of the vacancies and phase separation with forming nano-heterogeneous structures inside the perovskite matrix. Ion-transport functional properties of nano-heterogeneous materials are primarily related to their local structure, which differ from the structure averaged over the entire crystal. In situ XRD experiments show the transformation of the structure under low oxygen partial pressure at different temperatures (up to 900°C). Phase composition and structure parameters are in strong correlation to equilibrium oxygen content at different environment conditions. X-ray diffraction experiments were carried out with involvement of equipment belonging to the shared research center SSTRC, BINP SB RAS.


**Poster Session**

**EXAFS, XANES and XRD investigations of doped ZnS nanostructures**

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In the current state of materials science, considerable attention has been devoted to the study of new materials for light emitting devices. Light-emitting electroluminescent panels based on powders and thin films have found widespread application in electroluminescent devices, character displays, and backlight panels of advertising constructions. One major drawback of such devices is the use of high frequency and high voltage AC power sources. Traditional materials for electroluminescent light sources are powders and films of zinc sulfide doped with copper, chlorine, manganese, and other elements. It was established that the wavelength and intensity of light depend on the concentration and type of dopants, i.e., there are circumstances under which it is possible to provide a source of white light emission. However, along with the advantages of electroluminescent light sources, there are significant disadvantages: high energy consumption, the need for specific power supplies, and short operation time. This is due to degradation of the active layer under specific principles of operation of such devices in extremely high electric fields (before breakdown luminescence).

In this work, we use templating approaches based on the formation of doped ZnS particles in porous alumina membranes with highly ordered channels with controlled diameter. It is necessary to identify the influence of synthesis conditions on the structure and electrical properties (including light emitting) of the material. All this defines the scope of application of SR methods. EXAFS, XANES, and XRD, as powerful probe instruments, are used in investigations of doped ZnS nanostructures for the creation of a new class of fluorescent materials to form the basis of their layers for high-performance and high-brightness light-emitting electroluminescent panels. Some changes in the phase compositions and local structure arrangements of the studied ZnS doped samples (different compositions and preparation ways) were characterized in detail. The interatomic distances and corresponded coordination numbers were revealed. All possible structural models were discussed. Some correlations between their properties, local structural distortions, and state of doped components were established. This work is supported by Russian Scientific Foundation (Project 15-19-10002), SB RAS comprehensive program II.2P (Project 0305-2015-0018).

**Structural Session**

**Structural investigations of Ni deposited on porous anodic alumina matrices**

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Iron, nickel, cobalt, platinum, and other 3d and platinum group metals, their alloys and intermetallic compounds are traditionally used as cathodes in electrolytic hydrogen evolution reaction (HER) in aqueous acid and alkaline solutions [1]. Nickel stands out among them because of the fact that, having high corrosion resistance in acidic and alkaline media compared with platinum group...
metals, it has a low cost and so is widely used [2,3]. It should be noted that the efficiency of HER depends on the area of contact of the cathode material with the electrolyte, so there is an increased interest in literature to the nickel based materials having a developed surface due to the formation of metal nanoparticles with different morphology [4]. This is due to the large contribution of surface electronic states of the metal in HER. It is also strongly affected by the structural-phase state and local atomic and electronic structure causing nickel chemical activity. The aim of this work is to develop methods of magnetron sputtering nickel on high surface area membranes of a porous anodic alumina morphology and to study the structural-phase state, local atomic and electronic structures of the samples, depending on the parameters of the Al2O3 porous structure (the pore diameter, the distance between the individual pores). As a result of a complex study (XAFS, XRD, SEM), the state and local structure of samples obtained by few ways were revealed. The geometrical characteristics of Ni nanostructures, their crystalline and local atomic structure were studied. The local atomic structure parameters were compared to those revealed in investigations of the nickel film obtained on smooth Al2O3 surface. All possible structural models were discussed in detail. This work is supported by program Umnik (Grant 0020390), SB RAS comprehensive program II.2P (Project 0305-2015-0018).


Poster Session

Structural study of novel lipid-dependent dimerization of human GLTP induced by point mutation

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Protein-protein interactions are common in cell and molecular biology events, and essential for cellular function. Both homodimers and heterodimers are commonly involved in catalysis, regulation and structural assembly. However, the role of dimerization in controlling the action of amphitropic peripheral proteins that can exist in water-soluble and lipid-bilayer-bound states is not well studied. Human Glycolipid Transfer Protein (GLTP) carries out the important function of non-vesicular transport of glycosphingolipids (GSLs) between membranes [1] but details of the all alpha-helical GLTP-fold mechanism of action remain unclear. Previously, reversible lipid-dependent dimerization was discovered for holo-GLTP [2]. Structural studies using synchrotron radiation indicated a homodimer, reproducibly revealed in different crystal forms of GLTP bound with various GSLs. The homodimer is characterized by a 70-80 degree angle between wild-type monomers complexed with sulfatide, but the inter-monomer angle narrows to 63-66 degrees upon D48V mutation [2]. The inter-monomer contacts were found to mainly involve helix6-helix6 (H6-H6), as well as helix2-helix2 (H2-H2) at their C-termini. The X-ray structure of another mutant, K87Q, complexed with 18:1-glucosylceramide, reveals a novel homodimer with a different dimerization contact region that includes the mutation site, which was not involved in the original
dimerization contact region. Fluorescence spectroscopy assays involving intrinsic Trp emission changes show that K87Q-GLTP retains the original binding capacities for such GSLs, as sulfatide, glucosylceramide and galactosylceramide. Thus, GLTP dimer design could provide a way to dissect certain steps of the glycolipid transfer process. The influence of dimer type on steps of lipid transport by GLTP needs further investigation. This work was supported in part by Russian Foundation for Basic Research project 14-04-01671 and 15-04-07415, NIH NIGMS GM45928 and NCI121493, and CICbioGUNE research funds. 


Poster Session

Structure investigations of radiation-modified polymers

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Polymethylmethacrylate and polyethylene were irradiated with intense beam of high-energy electrons or with ‘white’ beam of synchrotron radiation with different doses. Results of changes in structure are presented: using synchrotron radiation X-Ray diffraction and thermal analysis. X-ray diffraction experiments were carried out with involvement of equipment belonging to the shared research center SSTRC, BINP SB RAS.

Poster Session

OBSERVATION OF GRAIN-BOUNDARY STRUCTURE FEATURES IN ALUMINIUM ALLOYS BY MEANS OF XAS AND X-RAY TOMOGRAPHY BY SR

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Aluminum and its alloys are considered to be the promising agents for hydrogen production in reaction with water to use them in compact fuel cells. The only problem is to increase their reactivity. One of the methods of activation is the treatment of aluminum by gallium-based liquid metal eutectics. Previously, we noted that the process of activating the aluminum alloys was associated with the grain boundary diffusion of the eutectic components. To determine the nature of high reactivity of the activated material in reaction with water, a number of commercial aluminum alloys were studied with the help of the methods of XAS and X-Ray tomography by SR after activation. It was shown that the highly active state was associated with the formation of the nanostructured material, wherein the components of Ga-In eutectic distributed throughout the grain boundaries in the all the mass of the original sample. To confirm this observation, the aluminum alloys with different grain boundary structures have been investigated including those having high reactivity as well as the samples which lost the activity during prolonged storage under atmospheric conditions. Using X-ray tomography by SR, it was shown that in all the highly reactive samples, the components of Ga-In eutectic were distributed in the grain boundaries of the entire material. The activated samples, in which the components were not evenly distributed, did not completely dissolve in water. By XAS method, it has been found that in the highly active
material, the eutectic components were in metallic state. Deactivation during prolonged storage under atmospheric conditions was associated with oxidation of the components of the eutectic alloy. It is shown that the distribution of the eutectic component in the sample is determined by the grain boundary structure of the initial material.

Poster Session

NEXAFS study of molecular arrangement in polyaniline films prepared by electrochemical deposition

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Nanoscale films based on conductive polymers and polyaniline/polypyrrole multilayers are of great interest and may be used in various applications such as protective and antielectrostatic coatings, membranes, sensors, capacitors, etc. Physical-chemical properties of polyaniline films depend on their molecular arrangement. Up to date, the effect of the electrochemical synthesis conditions on the chemical structure and molecular orientation in the films is far from being comprehensively studied. In the work, the polarization-dependent NEXAFS spectra were used to obtain the data on the molecular arrangement in a relatively thick (~1 micron and thicker) polyaniline films. The influence of different parameters of electrosynthesis was investigated such as polarization potentials; polarization modes (potentiostatic, cyclic); growth from still/stirring electrolyte solution. XPS and NEXAFS spectra were measured at the dipole Russian-German beamline at the BESSY II, HZB Berlin.

In most films, the polarization-dependent NEXAFS spectra have shown a preferential orientation of the macromolecular chains of polyaniline in the surface layer of the films. The films are arranged differently depending on the preparation conditions. For example, at the initial stages of the deposition under potentiostatic conditions in stirring electrolyte, most of the aromatic rings of the polymer molecules are arranged along with the film surface. With increasing deposition time, a preferential molecular orientation disappears. For the film prepared under the same conditions, but in still electrolyte, there is significant polarization effect, indicating that the aromatic rings are arranged perpendicular to the sample surface. However, the polymer chains themselves are oriented along with the sample surface. Such an orientation of the molecules can be assigned to the capture of macromolecular fragments and oligomers from the near-electrode layer, in which these fragments are accumulated.

The work was supported by the Russian Foundation for Basic Research (grant 16-43-180228) and the Russian-German Laboratory at BESSY II, HZB Berlin.

Poster Session

Propagation of beams with orbital angular momentum through 1D and 2D periodic grating (an analogue of the Talbot effect)

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Talbot effect (self-imaging of periodic gratings) attracted attention for 170 years that have passed since its discovery. The invention of lasers generating coherent beams caused a new impetus to these studies. Commissioning of the Novosibirsk free electron laser facility, which generates powerful coherent monochromatic radiation in the terahertz range, enabled us to demonstrate recently the Talbot effect in this region of the spectrum. Large wavelength of the terahertz radiation enables performing experiments with gratings, fabricated using LIGA technology at synchrotron radiation source VEPP-4, the structural elements of which are close to the wavelength. In this paper we describe first observation of diffraction patterns in the free space region behind the periodic structures illuminated with beams carrying orbital angular momenta with topological charges equal to plus-minus 1 or 2. The images observed did not reproduce images of the gratings exactly, but manifested some periodicity inherent to the Talbot effect with distorted spots related to individual grating openings. The numerical simulation of the electromagnetic field behind the gratings, carried out in a frame of the Fresnel-Kirchhoff model, is compared with the experimental data.

Poster Session

Cooling of the superconducting magnet with nitrogen-filled heat pipes.

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The paper describes usage of nitrogen-filled heat pipes for pre-cooling of the superconducting wiggler with “dry” cryostat (ANKA/CLIC Superconducting multipole wiggler 2015). In our design the first stages of cryo-coolers are connected to the magnet yoke by two heat pipes, which provide high heat transmission rate (up to 100 W per tube) in the temperature range from 300K down to 70K. When the nitrogen reaches its freezing point, an automatic thermal contact gap occurs. The heat pipes allowed us to exclude usage of liquid nitrogen from the process of pre-cooling of the magnet.

Invited Talks

The nanosecond time resolved X-ray diffractometry with synchrotron radiation for exploration of fast processes in solids

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The technique of X-ray diffraction with time resolution has shown tremendous progress recent years. This is due to the development of the accelerator technology, methods of generation of synchrotron radiation (SR), and fast X-ray detectors. The report reviews the main principles of time-resolved X-ray diffractometry and requirements to the object, equipment and detectors. Considered are the basic parameters of the diffraction installations at BINP, SLAC, and EuXFEL. To obtain the best experimental parameters it is necessary to minimize the duration of the SR flash and the divergence of the primary beam, as well as increasing its intensity and monochromaticity. Unfortunately, currently it is impossible to improve all the parameters simultaneously, so the experimenters have to compromise. For example, they increase the flux of photons at the expense of deterioration of the monochromaticity (BINP and APS/ANL) and carry out experiments in the “pink” spectrum. Or they increase the exposure time at the expense of summation of photons from a few bunches. This mode was applied to investigation into the dynamics of nucleation and growth of nanodiamonds in a shock-wave impact on hydrocarbons. Possible options of development of time-resolved X-ray diffractometry installations at BINP are considered. Now we are preparing an experiment to study the behavior of the crystal lattice of the material of the fusion reactor first wall in a plasma discharge on the diverter. A fast one-coordinate X-ray detector was developed for this experiment. The detector enables fast recording of 100 diffraction frames with an exposure time of 73 ps and a periodicity of 100 ns. Thus, we can record X-ray “movies” with high time resolution, which store information about the dynamics of plasma interaction with the structure of the crystal surface in a plasma discharge (100 ?s in the ITER). To solve this problem we are developing an installation to work on beams of synchrotron radiation of VEPP4(BINP SB RAS). The installation will enable obtaining information about what is happening to the crystal lattice when the plasma of the ITER reactor interacts with the wall for a short period of time. The plasma discharge parameters in the ITER are as follows: an energy of 100 J for 100 ?s on an area of 1 mm2. We conducted first successful test experiments, having recorded changes in the crystal lattice W, using a laser with a power of 1 J and a pulse width of 100 ?m. Now diffraction patterns with a time resolution of 73 ps can be recorded. The same method will be used for investigation into the behavior of the crystal lattice of space materials under the impact of shock waves and meteorites, flying with speeds of up to 11 km/sec. For this experiment, a gun launching small pellets with such speeds has been designed.

Invited Talks

X-ray Dark-Field Imaging (XDFI): Recent Developments and Clinical Applications

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This paper describes a novel x-ray phase contrast imaging technique called X-ray dark-field imaging (XDFI) that has been under development for the past 10 years. We describe the theory
behind XDFI, the x-ray optics required for implementing it in practice, and algorithms used for 2D, 2.5D, and 3D image reconstruction. The XDFI optical chain consists of an asymmetrically cut, Bragg-type monochromator-collimator that provides a planar monochromatic x-ray beam, a positioning stage for the specimens, a Laue-case angle analyzer, and one or two cameras to capture the dark and bright field images. We demonstrate soft-tissue discrimination capabilities of XDFI by reconstructing images with absorption and phase contrast. By using a variety of specimens such as breast tissue with cancer, joints with articular cartilage, ex-vivo human eye specimen, and others, we show that refraction-based contrast derived from XDFI is more effective in characterizing normal anatomy, articular pathology, and neoplastic disease than the convectional absorption-based images. For example, XDFI of breast tissue can discriminate between the normal and diseased terminal duct lobular unit, and between invasive and in-situ cancer. The final section of this paper is devoted to potential future developments to enable clinical and histo-pathological applications of this technique.

References


Biomedical application of SR and THz radiation

Investigation of the principles of detection of small amounts of nanoparticles of drugs and their conglomerates during the synthesis and transport in vivo using methods of X-ray synchrotron radiation

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The basic idea of the investigation is using the unique properties of the synchrotron radiation can receive information about the drug are in the gastrointestinal tract (GIT) of a living body at low concentrations. In particular: the phase state, i.e, tablet dissolved in the stomach or not; the charge state, i.e, the drug is active or passive; of the time (and place) of sedimentation of the drug; about the localization of the drug on the damaged wall of the digestive tract. In this project, the first steps was made towards the realization of the idea with using a model system. The experiment was conducted at the experimental station of synchrotron radiation (SR) 4 VEPP
-3. Medicament (in one experiment in the crystal, and in the other - in liquid form) was poured into the stomach of a turkey, and irradiated with a narrow beam with an energy 33.7 keV. With area detector mar-350 diffraction pattern of the drug was reserved, imposed on the scattering from the tissue of the stomach. The main objective was to allocate the sum of the diffraction pattern information about the structure of the drug. The main result of the experiment - using SR can receive information about the crystal lattice of drugs in real biological. Thus, using the an unique properties of synchrotron radiation in this study managed to obtain structural information about drag, are in conditions close to real ones (in terms of interaction with X-rays) - virtually the same as in the stomach of a living organism. The drug was in solid or in dissolved form. As an object of research we selected anti-ulcer drug substance based on bismuth tripotassium dicitrate (BTD). BTD is a drug with bactericidal activity against Helicobacter pylori has anti-inflammatory and astringent. Work was carried out on behalf of the Ministry of Health RF for the development of domestic analogue drugs De Nol (Netherlands). The project succeeded in synthesizing a complete analogue of imported medicines De-Nol (Netherlands) and to explore its metabolism in the chemical environment similar to the environment in different parts of the gastrointestinal tract. By simulating the behavior of BTD in the gastrointestinal tract by small-angle x-ray scattering discovered that bismuth forms a stable complex of a colloidal particle. The particle has size in order of 22 Å. If the solution is diluted with water particle size changes. The structure of the colloidal particles was investigated by X-ray diffraction on a synchrotron radiation beam. It was found that the structure is sensitive to pH

X-ray structural analysis

Hydrostatic and shock-wave compression of a molecular crystals

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The report presents analysis of experimental data on hydrostatic and shock-wave compression of energy-saturated material. The semi-empirical Mie-Gruneisen equation of state was used to describe thermodynamic properties of metastable molecular crystals without considering phase transitions. The equation of state describes experimental data on isothermal compression of a molecular crystal, and this data is obtained using the powder diffraction method. The Hugoniot curve expression plausibly describes shock-compression data on the studied material having various initial porosities.

Poster Session

The experimental station "DETONATION" at beamline 8 from 7 pole 1.3 T wiggler installed at e+e- collider VEPP-4

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The new experimental station “Detonation” was put into operation in test mode on the 8-th synchrotron radiation beamline from collider VEhPP-4. The station was designed for the study of detonation and shock-wave processes in condensed media in conditions approaching parameters (geometrical dimensions, weight), used in practical, industrial applications. Station “Detonation”, uses radiation of 7-pole wigglers (five main poles with the field of 1.3 T and the two side, with half the field [1])/ The new station has a significantly higher parameters as compared with the previous station at VEPP-3, experimental values of the basic parameters such as the intensity and spectrum stiffness, and volume of the explosion chamber permissible increase weight of explosion samples. This makes it possible to significantly reduce the influence of confounding factors, specific to the explosive sample is small and connected with the influence of the near surface, the curvature of the front, and so on. The main detector at the new station will position sensitive detector DIMEX [2], the use of which the station “Explosion” VEhPP-3 drive has shown great potential methods associated with the use of synchrotron radiation to study fast processes [3].


X-ray Spectroscopy

Current status of EXAFS station of SSTRC. Application of XAFS spectroscopy for the study of promising functional nanomaterials.

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Nowadays the method of XAFS spectroscopy in a different methodical options using of SR sources is a recognized powerful tool to study the state of the elements and the local structure for the various nanosystems having different aggregation: alloys, high dispersed material, semiconductors, solutions, glasses and others. In this report, by the example of works performed at the EXAFS station of SSTRC, for a variety of nanosized systems used in catalysis, materials science, biology, demonstrated the possibility of XAFS spectroscopy, as an independent method and in combination with other physical methods of research - HRTEM, XRD, XPS, SEM. The prospects of an integrated approach to study of nanostructured systems: dilute encapsulated ordered semiconductor nanostructures, nanocomposite catalysts having complex composition, biological nanomaterials and others 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. This work was supported by Russian Academy of Sciences and Federal Agency of Scientific Organizations, SB RAS comprehensive program II.2P (Project 0305-2015-0018), RFBR (140301066, 160301139).

Biomedical application of SR and THz radiation

Radiation therapy of human glioma tumors experiments in SSTRC
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In modern society, brain diseases belong to most frequent causes of death and reduction of the working age. For prevention and treatment of neuropathology, a variety of therapeutic and surgical approaches are being developed and improved, including radiation therapy methods. These methods have got a significant impetus to improvement through the creation of high-energy radiation sources and development of tools providing accurate targeting and strict time dosing in radiotherapy. Microbeam synchrotron radiation is one of the most promising trends in the modern radiotherapy. This technology is based on the use of synchrotron flux split into separate microbeams. Such spatial arrangement allows one to significantly amplify the dose on targets, e.g. tumors, without causing significant damage to healthy tissue. In our work we try to offer new possibilities for improving therapeutic technologies based microbeam radiation which is consists in combined use of radiotherapy and applying nanoparticles. In our experiments we used of combination of the effect of radiotherapy with saturation of tumor cell with nanoparticles of metal oxides, including particles with high catalytic activity. This combination may provide a synergistic effect for cytolysis of glioma cells based on nanoparticles enhance oxidative effect of reactive oxygen. In present time we search optimal combination of complex approach to the radio and chemo therapy of human glioma tumors.

Poster Session

XAFS study of model flotation system - xanthate/zinc sulphate: stuctural determination of interaction forms existing in aqueous solution and deposition.

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Presently our country takes the first place in terms of reserves and production of mineral resources, but the quality of many ore deposits is low. Ores most involved in the development of new deposits can be classified hard-to-concentrate, with a low content of useful components. Existing technologies of enrichment in some cases do not correspond to the quality of modern ores. So Russia, having large mineral reserves, faces difficulties in providing new technologies that can cost-effectively and efficiently process those materials. The decision of the problem of increasing the profitability of processing minerals and improving comprehensiveness of their enrichment are of paramount importance. One of the possible technological solutions is the developing approach of selective flotation separation of minerals with optimization of the proportion and properties of chemical and physical forms of sorption by combining different types of flotation reagents. One of the most popular reagents is butyl xanthate potassium, which is widely used for the extraction of valuable components by flotation in aqueous solution. The main aim of this work is a study of model flotation system - xanthate/zinc sulphate by the method of XAFS spectroscopy. All EXAFS and XANES spectra (Zn-K edge) of the samples studied (both in solution and deposition) were recorded at EXAFS station of Siberian Synchrotron Terahertz Radiation Center (SSTRC). Some differences of local structure of initial solutions of zinc sulfate (having different concentrations...
from $10^{-4}$ up to $10^{-1}$ mol/l) between each other and great ones compared to the bulk samples were found. The presence of different Zn-O forms were detected. For the model flotation system - xanthate/zinc sulphate in aqueous solution, Zn-S forms were predominantly found. Structural determination of all interaction forms existing in aqueous solution and deposition were carried out in detail. All possible structural models were discussed. Additionally powder samples of initial reagents and final depositions were characterized by XRD. The atomic structures of initial bulk sample were established. 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. This work is supported by Russian Scientific Foundation (Project No 15-17-10017).

X-ray Spectroscopy

**XAFS study of catalytic nanosystems promising for environmental catalysis**

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XAFS study of catalytic nanosystems promising for environmental catalysis E.P. Yakimchuk, V.V. Kriventsov Boreskov Institute of Catalysis of SB RAS, Novosibirsk, Russia

**X-ray Spectroscopy**

**Application of SR methods for the study of nanocomposite materials for Hydrogen Energy.**

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Application of SR methods for the study of nanocomposite materials for Hydrogen Energy. S.N. Pavlova, V.V. Kriventsov, Z.S. Vinokurov, N.F. Eremeev, V.A. Sadykov Boreskov Institute of Catalysis of SB RAS, Novosibirsk, Russia

In the emerging field of Hydrogen Energy, tailor-made design of the active components of monolithic catalysts for selective oxidation/autothermal reforming of hydrocarbons into syngas at short contact times, water gas shift reaction and preferential oxidation of CO in the hydrogen ex-cess is also based upon the concept of the bifunctional reaction mechanism, in which oxygen mobility in complex oxide fluorite-like support (doped ceria or ceria-zirconia) plays an important role. Complex oxide nanocomposites with perovskite-like and fluorite-like structures are known as good ionic or mixed ionic-electronic conductors. This ensures their broad application in the rapidly de-veloping technologies based upon solid state ionic devices including fuel cells (as electrolytes, components of cathodes and anodes), catalytic membrane reactors for ultra-pure oxygen and syn-gas generation. Defect structure of these complex oxide systems is considered as an important factor in ensuring their high performance. However, this restricts ability of traditional structural techniques to elucidate atomic-scale details of their real structure responsible for the transport properties of these systems. This is the reason why Synchrotron Radiation studies (XANES, EXAFS, XRD) of the real/defect structure of complex oxide nanocomposites become tremendously important. This presentation summarizes results of these SR studies for such nanocomposite systems as ceria doped by Me; ceria-zirconia doped by Ln and PrNi0.5Co0.5O3 ? (PNC), Ce0.9Y0.1O2 ? (YDC), Ce0.65Pr0.25Y0.1O2 ? (YPDC), CeO2 and Pr6O11 powders were
Synchrotron and Free electron laser Radiation: generation and... / Book of Abstracts

synthesized by modified Pechini route. Complimentary methods such as HRTEM, Raman, UV-Vis, XPS, magnetic measurements etc are applied as well to verify different hypothesis on the type of the real structure. As follows from analysis data, Ce cations charge was generally 4+ for PNC YDC, while Pr charge was commonly 3+ for PNC and both 3+ and 4+ for its nanocomposite. Since Pr charge varies differing from Ce, it can be Pr which is generally responsible for oxygen vacancies formation observed. This is in agreement with data on oxygen mobility and surface reactivity studies being carried out previously. Pr and Ce coordination numbers obtained from radial distribution functions are close to the ones in slightly distorted P and F structures. Ce LIII edge spectra for nanocomposite is similar to YPDC with difference explained by local Pr:Ce ratio variation and structure distortion. Thus, generally Pr3+ migration from P to F phase and its charge variation may cause additional va-cancies formation. This agrees with data obtained in the current work and previous studies. For nanocrystalline doped ceria-zirconia system, the most important factor controlling the lattice oxygen mobility appears to be length of Ce-O (Zr-O) bond and distortion of respective coordination spheres, while free anion vacancies are less important if present at all. Instead, the fast oxygen diffu-sion pathways could be associated with disordered domain boundaries including those between do-mains of different chemical compositions. In this case, the trend in variation of the real structure pa-rameters with the content and size of a doping cation is much more complex due to coexisting in the host fluorite-like lattice of small (Zr4+) and big (Ce4+) cations with different modes of the first co-dination sphere distortion. Moreover, this affects even the mode of the cations spatial distribution in the lattice or in the surface layer. Thus, doping of ceria-zirconia (1:1) solid solution by Ca or Gd re-sults in depletion of the surface layer by Zr. For these systems, more symmetric coordination envi-ronment around Zr cations in doped samples suggests some ordering due to incorporation of a big doping cation, which results in decreasing the lattice oxygen mobility. 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. This work was sup-ported by BIOGO FP7 Project, Russian Academy of Sciences, and Federal Agency of Scientific Or-ganizations (project V.44.1.17).

Poster Session

The synchrotron radiation beamline "Plasma" at collider VEPP-4

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Now we are preparing an experiment to study the behavior of the crystal lattice of the material of the fusion reactor first wall in a plasma discharge on the diverter. A fast one-coordinate X-ray detector was developed for this experiment. The detector enables fast recording of 100 diffraction frames with an exposure time of 73 ps and a periodicity of 100 ns. Thus, we can record X-ray “movies” with high time resolution, which store information about the dynamics of plasma interaction with the structure of the crystal surface in a plasma discharge (100 ?s in the ITER). To solve this problem we are developing an installation to work on beams of synchrotron radiation of VEPP-4 (BINP SB RAS). The installation will enable obtaining information about what is happening to the crystal lattice when the plasma of the ITER reactor interacts with the wall for a short period of time. The plasma discharge parameters in the ITER are as follows: an energy of 100 J for 100 ?s on an area of 1 mm2. We conducted first successful test experiments,
having recorded changes in the crystal lattice W, using a laser with a power of 1 J and a pulse width of 100 ?m. Now diffraction patterns with a time resolution of 73 ps can be recorded.

Poster Session

Experimental realization of X-ray diffraction and small-angle scattering using polychromatic synchrotron radiation in the range 20-30 keV

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The aim of this work is to develop a method to carry out X-ray analysis using a broad spectral of synchrotron radiation. This will give an opportunity to increase the number of photons in the primary beam on the order of 2-3 and thus reduce the exposure time for diffraction experiment to nanosecond at VEPP-3 and picosecond at VEPP-4.

Poster Session

New X-ray method for monitoring of polymers radiation crosslinking

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In this work the effect of radiation of relativistic electrons on the crystal structure of polyethylene after radiation crosslinking studied. The main attention was paid to changes in the temperature of the Debye. The structure factor equation given earlier yields the diffracted intensities as though all atoms were stationary. As the temperature rises the atoms execute increased excursions from their average position. Such excursions can involve complex coupled motions of several atoms. However if one restricts consideration to just the independent motions of each atom there is a relatively simple trick that can be used which is to (artificially) modify the atomic scattering factors, f, of each atom to represent the net broadening of their electron clouds resulting from atomic motion. For example a given atom will have its “stationary” f changed to $f_T$ according to: $f_T = f \exp(-B \sin^2 \theta / \lambda^2)$ where T is the temperature and B = 8?2n2 (units of A2 ), ?2 being the root mean square displacement of the atom from its average position. The B values are often referred to as B factors (Debye-Waller is another, similar, term often used) and increase with temperature (typical values are 0.2 to 0.8 A2 ). So clearly temperature will modify the diffraction intensities by changing the various values of f to $f_T$ in the structure factor equation. To determine the Debye powder X-ray series was filmed at different temperatures of the polyethylene samples irradiated by relativistic electrons. The method allows to monitor the changes in the structure upon irradiation.
The formation of silver nanoparticles by synchrotron radiation and relativistic electrons irradiating of water solution of silver salts

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It has been found that irradiation of aqueous solutions of silver salts by synchrotron radiation start formation of silver nanoparticles. Similar processes occur upon irradiation by relativistic electrons. In aqueous solution, the silver particles are agglomerated into large particles of micron sizes. It was found surfactant, preventing agglomeration. The result is a stable colloid solution in which no precipitation of particles within a few months.

Poster Session

SIZE AND SHAPE DEPENDANCY OF CO ADSORPTION ON Pd14 AND Pd55 NANOCLUSTERS: DFT AND FTIR STUDIES

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Palladium is a very effective catalyst for the oxidation of CO. This reaction is very important for reducing automotive and industrial emissions and considered to be a good model for fundamental studies of transition metal catalysts. It is known that different types of facets as well as size and shape of a metal nanoparticle have an effect on catalyst properties. The scope of our work is to investigate how the reactivity of Pd nanoparticles towards CO oxidation depends on their size and shape. The keys to the solution are ab-initio DFT calculations in combination with in-situ FTIR spectroscopy. For theoretical part, we have chosen the set of Pd clusters of different size and respective shape. After optimization of their geometry, the adsorbed CO molecules were relaxed at the top-on positions of cluster surface. That allowed us to generate IR spectra to be compared with experimental ones of CO adsorbed on Pd nanoparticles. In order to be closer to the industrial-type processes, a special setup allowing in-situ FTIR measurements was designed as well. In our theoretical studies we used both molecular orbital (ADF-2014) and periodic band structure (VASP 5.2) DFT calculation schemes to model small metallic Pd clusters. The basis in calculations was chosen as Triple Zeta with one polarization function and frozen core. We used standard GGA approximation within PBE scheme. Occupations steepest descend method was used for better geometry convergence. All the parameters mentioned above with Scalable SCF gave good convergence in all criteria. The cutoff energy for periodic plane wave pseudopotential simulation was set to 400eV. The cell around Palladium clusters was constructed as a cubic one with 30 nm edge. A single K-point was used in simulation. In this work we constructed and optimized geometry of Pd14 and Pd55 clusters with octahedral symmetry. The relaxed interatomic distances (Angstrom) of Palladium 55 cluster with CO molecules placed on top hollow sites of Pd obtained by both ADF and VASP DFT calculation schemes are presented in Table 1.

Table 1. The relaxed interatomic distances (Å) of Pd-55 cluster with CO

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<thead>
<tr>
<th></th>
<th>ADF</th>
<th>VASP 5.2</th>
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<tr>
<td>Pd-C</td>
<td>2.052</td>
<td>2.042</td>
</tr>
<tr>
<td>C-O</td>
<td>1.198</td>
<td>1.200</td>
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IR spectra for different configurations were obtained during the calculation. For example main peaks are at 2107 and 503 cm⁻¹. Both CO adsorption and oxidation on Pd nanoparticles have been studied extensively with different experimental techniques [R1]. It is assumed that reaction of CO oxidation follows a Langmuir-Hinshelwood mechanism [R2], where both reactants, CO and O₂, adsorb on the catalyst surface prior to reaction between the adsorbed species to form CO₂. There were also found some differences in the kinetics among the (111), (110), and (100) planes in studies on single crystals of Pd [R3]. Significant number of experimental studies of CO adsorption on surface of Pd have been performed by using Diffuse Reflectance Infrared Fourier Transform Spectroscopy (DRIFTS). DRIFTS is a useful technique for probing catalysts at catalytically relevant temperatures and pressures without intense sample preparation [R4]. Our experimental setup for in-situ DRIFT studies of CO adsorption on Pd nanoparticles as main components includes Vertex 70 FTIR spectrometer, Praying Mantis Diffuse Reflection Accessory equipped with Low and High Temperature Reaction Chambers and a two-channel gas mixing system assembled by using the Swagelok tube fittings.

References

Poster Session

Cell for X-Ray investigation of new materials for Li-ion batteries

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Li-ion batteries are commonly used in our everyday life. However, we face several problems with their applications such as aging of the material inside, necessity of specific charge and discharge cycles, low capacity and small temperature range. In order to overcome these difficulties we have to understand what is happening with the material inside the battery. The solution to the problem is constructing a special experimental cell for X-Ray experiments, which would allow us to measure in-situ battery characteristic.

Our experimental setup consists of two parts. First part is electronics, which allows measuring desired characteristics such as battery capacity, volt-ampere characteristic by charging and discharging the battery repetitively. Control unit of the electronic part is based on AVR microcontroller ATmega 2560 with 10-bit analog to digital converter. It allows having up to 120 measurements per minute with 2 mV and 1 mA accuracy. Control of the power circuit is realized via 8-bit external multichannel digital to analog converter. In addition, there is on-board USB-COM Bridge for communication with specially designed computer software. This software provides control of work regimes. Main functions are charge or discharge of the battery, measurement of capacity, measurement of the level of charge, cycling the battery with specific current in needed range of voltage and charge or discharge to specific capacity.

The second part is a special cylindrical cell consisting of two parts with thin replaceable conducting and X-Ray transparent windows. One part is made from aluminum for cathode material and the other one is made from copper for anode material. They are separated with PTFE gasket. The design of cell allows to conduct XAS, XRD and other types of synchrotron and free electron laser experiments.

Invited Talks

High-intense femtosecond radiation with gas medium and gas cluster beams
Interactions of laser radiation with gas cluster targets have been investigated in the past two decades. Cluster jet, obtained by the adiabatic condensation of gas flow, has pronounced nonlinear optical properties and combines the advantages of solid-state and gas target. Cluster inherits high local density and this implies high value of nonlinear response. At the same time cluster beams are not exposed to ablation and renew their properties before each act of interaction with the laser pulse. There were observed and predicted various nonlinear effects during interaction of intense laser pulses with cluster jets: generation of X-ray radiation, generation of optical harmonics, self-focusing, Stimulated Raman Scattering. It was shown theoretically and experimentally that absorption of laser radiation in cluster beam can reach high value (up to 95%) which is related to linear (Mie) and nonlinear resonance interactions. Resonance absorption of pulse energy results in efficient production of X-Ray and fast charged particles. In this way, further studies of interaction of intense laser pulses with clusters seems reasonable due to the possibility of usage in various practical applications and solving fundamental problems of behavior of matter under intense laser fields.

In some recent publications, intense THz generation in cluster beam excited by ultrashort laser pulses was reported. It was observed more than two orders of magnitude enhancement of THz pulse intensity in Ar cluster jet compared to that in gaseous Ar with equal average atomic density. With increasing of excitant pulse energy up to its maximum value of 70 mJ (corresponding to the vacuum intensity $\sim 10^{17}$ W/cm$^2$) THz pulse energy increased by the square law without saturation. There was reported in that the directivity pattern of terahertz radiation from clustered plasma has four-lobed structure.

At the present moment there is no clear theoretical interpretation of the experimental results and complete understanding of the mechanism of low-frequency emission in cluster plasma. It seems reasonable to carry out further study of terahertz generation in a clustered plasma, and examine this process in both ways: as a fundamental issue of laser-matter interaction on the way to solving a problem of the dynamics of laser-cluster interaction, and as a practical goal of obtaining an effective source of pulsed THz radiation. In addition, there is possible to apply two-color excitation scheme, which has been successfully used in the past to increase the efficiency of the optical to terahertz conversion in laser-induced plasma of gas media. In this scheme, the fundamental laser frequency at $\omega$ is mixed with its second harmonic at $2\omega$. Two-color scheme allows to increase THz yield by 2...3 orders of magnitude and nowadays this scheme is widely used for THz generation in gaseous media. Nevertheless, optical to terahertz conversion efficiency in laser-plasma generation method is still low. In addition, there was observed a saturation of THz yield in a two-color scheme at high excitant pulse energy that originates from THz absorption in dense plasma. Cluster target seems to be attractive to solve these problems.

In this paper we present the results of experimental and theoretical studies of generation of terahertz emission in the laser-induced clustered plasma. We have performed experiments using both single-color and two-color excitation schemes. Simultaneously with the control of terahertz emission of clustered plasma we measured the power of accompanying X-ray radiation, which is an important source of information about the processes that occur in a cluster plasma. In the theoretical section we made an attempt to explain the experimental results.

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**Radiation sources and Centers**

**Possibilities for future SR and FEL development in the UK**

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