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BOOK OF ABSTRACTS

September 27th - October 1st 2021

ESRF ID10 beamline from the synchrotron source down to sample

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The growing over last decades interest to the study of the surfaces and interfaces using grazing incidence geometry techniques leads to the consistent development of both the theory and the experimental instrumentation. The recent modernization of the storage ring according to a revolutionary concept has made it possible to build a new generation synchrotron, ESRF-EBS (Extremely Brilliant Source), with a hundredfold increase in the brightness and coherence of X-ray radiation. Station ID10, one of nearly four dozen specialized experimental installations, was conceived, developed and built for research on surfaces and interfaces of soft condensed matter. These substances include systems such as Langmuir films, two-dimensional self-assembly of molecules, nanoparticles, or protein complexes on the surface of a liquid. A unique feature of the ID10 station is a device that deflects the synchrotron beam in a large angular range to the surface of the liquid for X-ray reflectivity (XRR) and diffraction measurements (Grazing Incidence Wide Angle Scattering (GIWAXS), the Grazing Incidence Small Angle Scattering (GISAXS) and the Grazing Incidence X-ray Fluorescence (GIXF)). The main part of the presentation will be devoted to the details of the ID10 beamline layout.

Coherent X-ray imaging and scattering for materials science applications

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Coherence of X-ray beams is a key property of 3rd and 4th generation synchrotron sources (X-ray free electron lasers). In combination with high brilliance and repetition rates it opens opportunities for unprecedented science: enhancing imaging capabilities for objects with low density contrast down to the nanoscale, using pump-probe methods to characterize ultrafast 'one-off' processes (without strobing), and collecting very large volumes of data to underpin advanced analysis techniques that include Bayesian and AI approaches. A crucial role in enabling these experiments is played by the development and adoption of specially optimized optics that preserve coherence and allow maximum information to be extracted. In this talk I will present examples of research studies conducted with our collaborators by my research team at MBLEM lab at Oxford that engage with different aspects of materials science, from lithium-ion batteries to biological tissues.

Ambient Pressure Photoelectron Spectroscopy at MAX IV – Studying Catalytic Systems with Milli- and Microsecond Time Resolution

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Chemical transformations occurring at the interfaces between gases and solids are the driving forces responsible for multiple industry- and research-relevant processes such as heterogeneous catalysis, gas sensing, and thin film growth. These processes are dynamic by nature with various steps occurring at different time scales. To obtain complete picture about surface reaction one has to study their time evolution using time-sensitive experimental techniques. We present recent developments at MAX IV that allow such time-resolved APXPS measurement using gas pressure/composition as perturbation that drives the system away from its equilibrium. One of such approaches makes use of a fast valve that creates gas pulses with an internal pressure in the mbar range and a rising edge of few hundred of microseconds. A gated detector based on a fast camera is synchronized with the valve operation to measure X-ray photoemission spectra with up to 20 μs time resolution. We will present several experiments characterising the setup's performance including the CO oxidation reaction over Pt (111) to demonstrate the capability of the setup to correlate the gas phase composition with that of the surface during transient supply of CO gas into an O_2 stream.

Coherence diffraction microscopy – seeing the invisible

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Coherence X-ray diffractive imaging (CXDI) is a novel scattering technique [1,2] that exploits the unprecedented degree of coherence of modern synchrotron sources. It has potential for high resolution imaging of isolated microscopic objects beyond the values achieved with X-ray lenses and represents an interesting tool to bridge the gap between high resolution electron and visible light microscopy. The image is obtained by applying an iterative phase retrieval algorithm to the diffraction pattern measured with sufficient oversampling. Because of the high penetration power of the X-ray the imaging of thick object (<10 mm) without sectioning is possible in 3D [3-7].

The objective of this talk is to provide the basic concepts of the coherent diffraction microscopy, in particular the notion of plane wave propagation, oversampling criterion, iterative phase retrieval algorithm. Several examples will be shown to demonstrate the capability of the technique for material characterization with nanometer resolution.

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Ion beam figuring for making diffraction quality X-ray optics

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The paper reports the success of IPM RAS research in the field of precision surface treatment of optical materials by accelerated ion beams. The characteristics and capabilities of the equipment (Figure 1b) are given, as well as the tasks solved with it. Conventionally, the operation modes can be divided into three directions: correction of local shape errors (etching by small size ion beam, Figure 1a), ion polishing and aspherization (treatment by wide ion beam, Figure 1c). For each of these tasks, the approaches to solving and the results achieved are described. In particular, for fused silica the record parameters of the effective surface roughness (σ_{eff}=0.14 nm) in the whole range of spatial frequencies ($q \in [4.9 \cdot 10^{-2}\text{-}6.3 \cdot 10^{1}$ μm^{-1}]) are given.

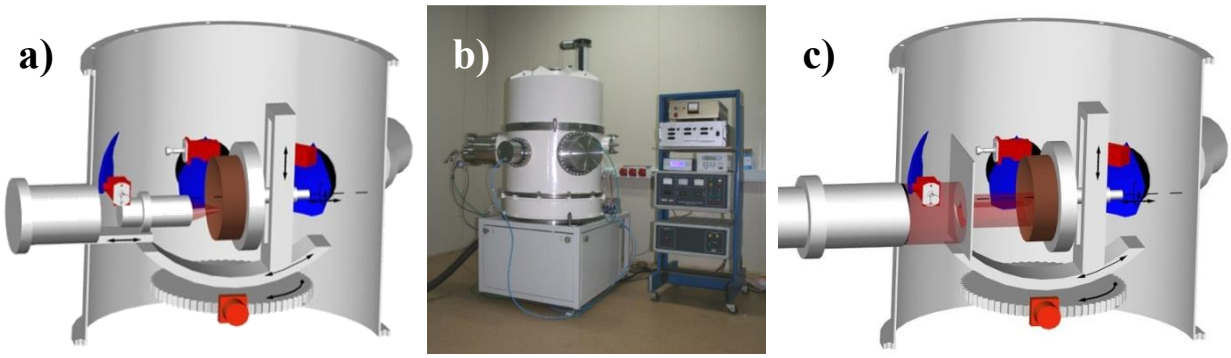

Figure 1. Ion-beam etching machine. a) the local shape errors correction by a small-size ion beam mode; b) photo of the machine; c) the axisymmetric aspherization through the forming diaphragm mode.

Considerable attention is paid to the study of the influence of ion etching parameters on the roughness and morphology of the surface of monocrystalline silicon of various orientations. Samples with crystallographic cuts <100>, <110> and <111> were experimentally studied. As a result of the work, the parameters providing etching to a depth of more than 2 micrometers with surface roughness preservation on the super-smooth level σ_{eff}<0.3 nm (Figure 2) for all the studied orientations were found and a method for manufacturing precision X-ray optical elements for high intensity synchrotrons based on single-crystal silicon was proposed.

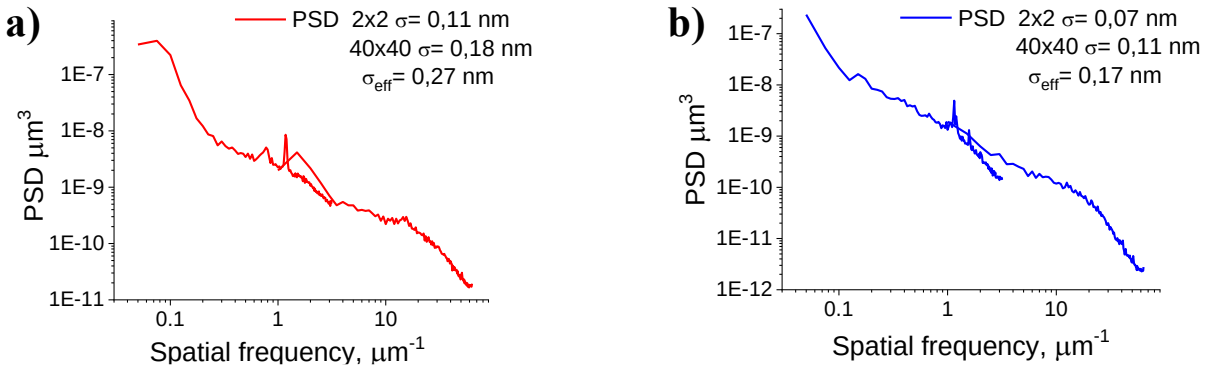

Figure 2. PSD roughness of functions for Si <110> before (a) and after (b) ion polishing.

The work supported by the Russian Science Foundation, (project No. 21-72-30029).

Are there multilayer Laue lenses for focusing hard X-rays?

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Elements of multilayer X-ray optics are widely used in synchrotron radiation installations for transporting X-ray beams, in EUV lithography, in astronomy and for focusing radiation.
Currently, there are various methods for focusing synchrotron radiation, among which the use of multilayer Laue lenses, according to [1], have great prospects in X-ray optics. Multilayer Laue lenses are created mainly by magnetron sputtering in accordance with the configuration of the Fresnel zones. On the other hand, despite their similarity to Fresnel zone plates, multilayer Laue lenses should exhibit another focusing properties. It is still believed that the focusing of hard X-ray radiation by multilayer Laue lenses is directly related to the structure of Fresnel zone plates. Is this opinion true? Are there any prospects for focusing with multilayer Laue lenses up to a focal spot size of several nanometers, which has been declared in many articles? The results of [2] to some extent contain answers to these questions. It is shown that the size of the effective aperture of the diffracted beam emerging from the Laue lens depends on the mismatch of the period of the layers of the multilayer Laue lens and is not related to the configuration of the Fresnel zone plate. Since this conclusion is still ignored in a number of works, the first step was to study the Laue diffraction of synchrotron radiation by sectioned multilayers with a constant period [3].

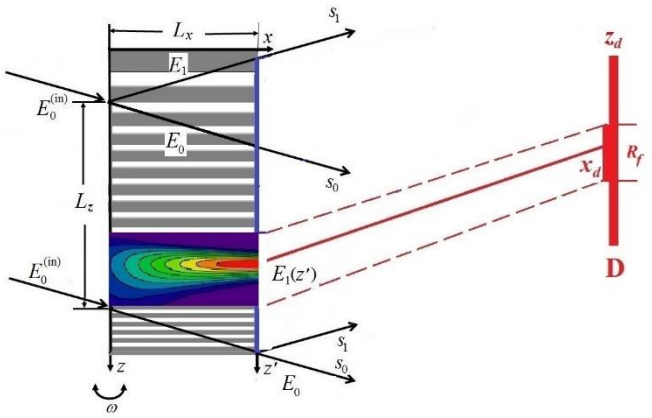

Figure 1. Schematic representation of X-ray diffraction from a multilayer Laue lens and the intensity distribution of the reflected wave inside the multilayer structure

The report discusses the formation of X-ray fields inside a multilayer Laue lens. Figure 1 shows the intensity distribution of the reflected wave inside the multilayer structure. This distribution of the X-ray field is due to the pendulum Laue effect of diffraction in the graded structure. The calculated focal spot sizes differ significantly from the values given in other works. The physical nature of collimation (focusing) of radiation by a multilayer Laue lens is revealed, which is determined by Bragg diffraction and is not related to the configuration of the Fresnel zone plate.

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Development and Fabrication of X-ray silicon optical elements and test structures

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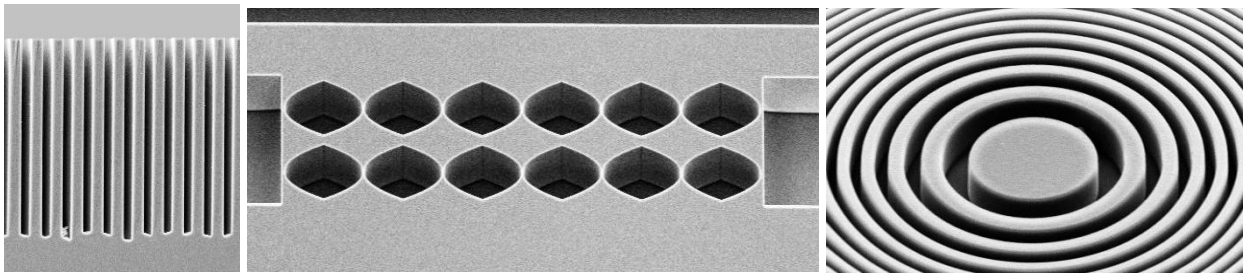
Silicon wafers and technologies developed for microelectronics and MEMS applications are in use for X-ray diffractive and refractive optics fabrication [1, 2]. Silicon is very attractive material because it has perfect crystal structure, high radiation and temperature stability. Another advantage of silicon is the presence of a large number of technologies for its processing.

Anisotropic wet etching of silicon wafers (100), (110) and (111) allows one to fabricate structures for a wide range of applications. Dry reactive ion etching is the main process for crystal orientation independent precise microstructuring of silicon wafers.

Deep silicon etching based on Bosch or cryogenic processes allowed us to fabricate a variety of planar compound refractive x-ray lenses (CRLs) [1]. Planar compound parabolic ones are the most attractive for applications at new X-ray sources, such as third-and fourth-generation synchrotrons and free-electron lasers [3].

However, all available parabolic lenses with physical aperture of 50, 30, and 10 μm are not perfect in terms of focusing parameters, shape and quality of their refractive surfaces since the applied silicon etching processes have some limitations related to processing inaccuracies [4]. The main limitations that effect X-ray optics performance are reviewed.

To overcome the limitations in order to fabricate nanofocusing (< 50 nm) parabolic lenses with refractive shapes and surfaces close to ideal ones we proposed some new approaches and technologies in lens design, lens manufacturing and lens shape metrology [5].



(a) (b) (c)

Figure 1. SEM images of: (a) U-grooves in Si (110), (b) bilens interferometer based on two CRLs, (c) circular Fresnel lens.

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Actinide Chemistry by Cutting Edge X-ray Methods

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Understanding the mechanisms of different chemical reactions with actinides at the atomic level is a key step towards safe disposal of nuclear wastes and towards the identification of physical-chemical processes of radionuclides in the environment. This contribution will provide an overview of the recently performed studies on Uranium[1–4], Thorium[5,6] and Plutonium[7,8] contained materials at the Rossendorf Beamline (ROBL) of the European Synchrotron (ESRF) in Grenoble (France). This innovative and world-wide unique experimental station was used to study actinide systems by several experimental methods: X-ray absorption spectroscopy in high energy resolution fluorescence detection (HERFD) mode, Resonant Inelastic X-ray Scattering (RIXS) at the An L_3 and $M_{4,5}$ edge and X-ray diffraction (XRD). We will show how the detail information about local and electronic structure of actinide materials can be obtained, including information on the electron-electron interactions, hybridization between molecular orbitals, the occupation and the degree of the *f*-electron localization. The experimental spectral features have been analysed using a number of theoretical methods based on density functional theory and atomic multiplet theory. It might be of interest for fundamental research in chemistry and physics of actinide systems as well as for the applied science.

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Ultrafast dynamics of spatial magnetic fluctuations in Co/Pt multilayers studied at European XFEL

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Optical manipulation of magnetization on sub-picosecond timescales has been a field of intense research since the discovery of ultrafast demagnetization in 1996 [1-3]. While the global behavior of demagnetization was intensively investigated over the last decades, information on the ultrafast spatial evolution of magnetization is scarce. The first evidence that lateral superdiffusive currents contribute to the demagnetization of a domain network was given by Pfau and Iacocca et al. [4,5]. Described herein are the results of the recent pump-probe XFEL experiment, employing time resolved resonant magnetic small-angle X-ray scattering in which for the first time an optically induced transient magnetic scattering was detected in a Co/Pt film on a picosecond timescale in the wave-vector region above 0.2 nm^{-1}. In contrast to Iacocca *et al.* [5] the transient signal appeared only in the collective region of the magnetic phase diagram far below the ferromagnetic-paramagnetic phase transition.

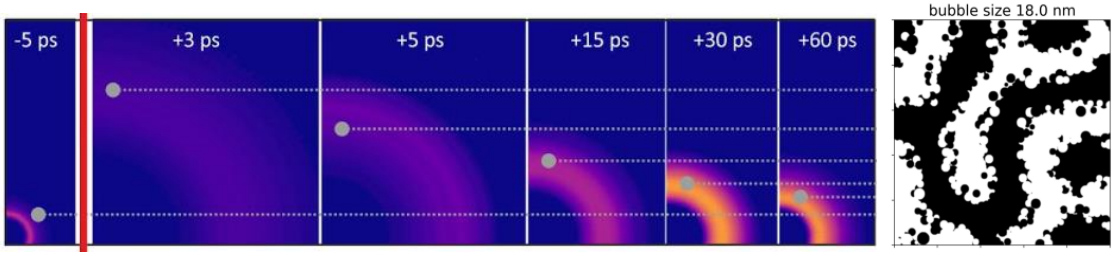

Figure 1. Time resolved transient magnetic scattering (left) and related domain network model (right).

Interestingly, the size of detected magnetic fluctuations (7-30 nm) was significantly smaller than the 190 nm domain periodicity. The short-range magnetic fluctuations were assumed to appear at the domain boundaries once the system is "shaken" by the IR pump. A phenomenological model of magnetic bubbles appearing at domain boundaries upon optical excitation has been built. Besides, a micromagnetic two-temperature model describing the transient magnetic fluctuations in the Co/Pt system has been proposed. The value of the presented study is related to the unraveling of the ultrafast magnetic response upon optical pumping. The obtained knowledge is supposed indispensable for the development of potential spintronic applications and from the point of the fundamental physics involved.

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Введение в статистическую рентгеновскую оптику

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Лекция представляет собой краткое введение в основные понятия статистической волновой оптики с приведением ряда примеров из области оптики рентгеновского диапазона длин волн. Предполагается примерно следующий план изложения:

1. Предварительные представления о когерентности.
2. Регулярные и случайные процессы. Корреляционные и спектральные характеристики случайных процессов. Статистическое усреднение и усреднение по времени. Теорема Винера-Хинчина [1].
3. Случайные поля. Угловые и частотные спектры пространственно-временных случайных полей [1, 2].
4. Источники рентгеновского излучения (трубки, синхротроны, рентгеновские лазеры на свободных электронах).
5. Полностью и частично когерентные поля. Распространение волн в пространстве (пропагаторы [2]). Корреляционные функции. Пространственная (поперечная) и временная (продольная) когерентность. Опыт Юнга. Теорема Ван Циттерта-Цернике [1].
6. Когерентная рентгеновская дифракция [3]. Осевая (on-line) рентгеновская голография, imaging и влияние когерентных свойств излучения на контраст изображений. Проблема фазы и пути её решения. Примеры решения прямых и обратных задач.
7. Дифракция случайных рентгеновских волн. Особенности влияния когерентных свойств излучения на брэгговскую дифракцию фемтосекундных импульсов рентгеновского лазера на свободных электронах в кристаллах и многослойных структурах [4-6].
8. Изменение функций пространственной и временной когерентности при брэгговской дифракции рентгеновских пучков и импульсов с произвольной степенью когерентности [4-6].
9. Режим self-seeding и его использование для повышения степени временной когерентности случайных импульсов рентгеновского лазера на свободных электронах [6].

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Introduction to the phase space optics as a basis of the X-ray simulations method for the large fields of view

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The simulation of the experiment has always been a frontier zone. This is a boundary at which our knowledge about the nature intersect with what is actually happening. In the X-ray region, the simulation has a number of specific features. On the one hand, the extremely small wavelength makes it possible to conduct studies of micro-and nanoscale objects. On the other hand, an extremely high penetrating ability is of interest in the diagnosis of macro objects, such as, for example, chest fluorography or welding seams at the junction of pipes. Is it possible to combine an angstrom wavelength with millimeter, centimeter, and maybe decimeter, or even more, areas of study in the simulations? A method for modeling X-ray diffraction that can flexibly adjust the numerical scheme depending on the task is presented in this report [1].

Within the framework of the approach based on the use of phase space functions, the method of fragment-by-fragment simulations of the large fields of view was implemented. It is shown that the method of "coupling " wave fields works even in the case of computing interference images. Also, the method naturally takes into account the anisotropy of the source (the difference in size along different directions), which is characteristic of synchrotron radiation sources.

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SiMerge: looking at diffraction in 3D

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With modern 2D detectors it is very easy to measure some part of 3D reciprocal space of some object. One just needs to rotate the sample around almost any axis (except the beam direction) while recording the diffraction patterns and corresponding angles. Later the 3D reciprocal map can be built for further analysis.

The equations for recalculating detector pixel positions into the reciprocal space are very simple, so it's not a problem to build the 3D intensity distribution. The issues are usually "just" technical: how to open files saved in different formats, how to make the program efficient, how to "clean" the images before processing and make different normalizations. Another problem emerges when the resulting 3D volume has to be analyzed: it is usually not so easy to manipulate 3D data and sometimes even to view it.

Previously we have made a program for 3D merging of protein crystallography data [1] and successfully used it for analysis of some new and interesting features that can be observed in 3D reciprocal space [2]. But our program was not scalable and it was rather difficult to modify.

That's why we have developed a new program, SiMerge [3], that is distributed under the GPLv3 and is rather easy to modify. The program is written in C++ and is well optimized for merging even rather big 3D volumes (up to the amount of installed RAM). It can be compilated under Linux, Windows or Mac and has only one optional dependence – HDF5 library. The main features of the program are: it supports plenty of common file formats (HDF5, CBF, TIFF, IMG, EDF, RAW, etc.), it supports almost any detector geometry configuration, supports different background corrections (local and different radials), has an option of distance-weighted interpolated merge, supports arbitrary rotation axis and Euler angles, gain and dark correction, per pattern scaling, different masks and ROIs, polarization and pixel size corrections, and so on. For easier data processing the operations with the resulting 3D volume are separated into one more program [3] and also a 3D viewer written in python (using VTK library) is deposited at [3].

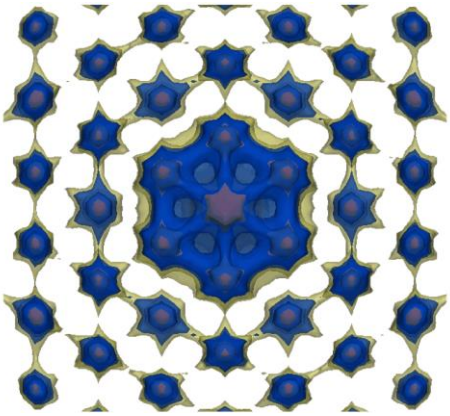

Figure 1. A merge of PS1 dataset measured at LCLS

The talk will cover the basics of 3D reciprocal space mapping, some more complicated aspects (like different ways of background subtraction), examples and general ideas how to measure non-standard diffraction using standard setup or even something that can't be measured.

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X-ray methods for studying incompletely ordered systems: reflectometry, tomography, topotomography, small-angle scattering

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Methods of X-ray reflectometry, microtomography and small-angle scattering are now effectively used to study incompletely ordered materials, which are increasingly used in modern technology.

It is logical to use the method of reflectometry, based on the effect of total external reflection of X-rays, to study the peculiarities of the surface structure of materials. Scattering of radiation under such conditions makes it possible to determine the surface roughness of smooth objects. The paper presents the results of relevant studies, compares different methods of roughness measurement and shows that for most substrate materials modern technologies allow to obtain the RMS roughness height at the level of less than 0.2 nm in a wide range of spatial frequencies. On the other hand, analysis of the angular dependence of the coefficient of total external reflection makes it possible to establish how the electronic density of the samples under study changes in the direction perpendicular to their surface. The results of studies of the structure of lipid layers deposited on liquid substrates carried out by this method are presented. In addition, the possibility of X-ray reflection by a rotating liquid, based on the "whispering gallery" effect, has been demonstrated for the first time.

The method of X-ray microtomography is based on the differences in the absorption of radiation by the studied samples depending on the local density and chemical composition. The results of the study of a number of biological objects are presented. It is shown that elements of the locomotor apparatus of geckos do not undergo changes after staying in microgravity. This distinguishes this animal from other living organisms. Methods of absorption and phase-contrast microtomography have established peculiarities of the structure of human brain epiphysis in norm and under pathologies. This endocrine gland is responsible for regulation of circadian rhythms.

To analyze the perfection of the internal structure of crystals, the method of X-ray topotomography was used for the first time in laboratory conditions. When studying synthetic diamonds, a new type of defects was discovered - cone-shaped defects. They represent dislocations decorated by microinclusions of extraneous phases (probably silicates and/or oxides). The partitioning of a crystal growth face into separate blocks with the subsequent formation of dislocation bundles and interblock boundaries can cause their formation. Using silicon crystals as an example, it is shown that even single dislocations specially introduced into the structure can be detected by this method. The application of the "whispering gallery" effect and tomographic reconstruction methods has created a possibility to detect defects on concave surfaces.

The possibilities of the method of small-angle scattering for the study of various monodisperse systems are shown. Using small-angle scattering, microtomography, helium pycnometry, and mercury porosimetry, a multiscale study of the structure of polylactides, biodegradable polymers used in medicine, was performed.

In conclusion, it should be noted that the methods described in this paper, despite their differences, can be complementary in various studies, which increases the reliability and informativeness of the experimental results.

This work was supported by the Ministry of Science and Higher Education within the State assignment FSRC «Crystallography and Photonics» RAS.

Microvoids in solids: synchrotron radiation phase contrast imaging and simulations

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Solid materials may contain inhomogeneities, such as microcracks, micropipes, minute cavities and pores. Despite the small size, they can be detected using synchrotron radiation (SR). Unlike single crystals in which voids are relatively sparse, some materials, e.g., biocomposites, such as bone tissues or dentin, can contain dense arrays of voids that are proven to play a significant role in toughening.

In the first part of the lecture, x-ray imaging technique to obtain a fairly complete picture of micropores within the single crystal (without destroying it) has been presented. Phase contrast x-ray imaging, based on in-line setup, came into wide use after 1995, when it was shown that 3rd generation SR sources possessed a sufficient degree of coherence [1].

In the second part, the presented method is employed to examine pipes with μm-sized diameters in bulk silicon carbide (SiC), a wide bandgap semiconductor. The principles of one-dimensional (1D) computer simulations of phase-contrast images have been explained on example of SiC [2]. The simulations have been shown to improve understanding new features of dislocated micropipes, the most harmful structural defects in SiC-based devices.

Finally, the approach to 2D objects has been discussed. Experimental images of arrays of tubules in human dentin as well as pores in single crystals having a complex geometry are shown (Figure 1). One can say with certainty that simulation-enriched phase-contrast imaging allows a reliable quantification at the micro-level even for moderate values of the transverse coherence lengths.

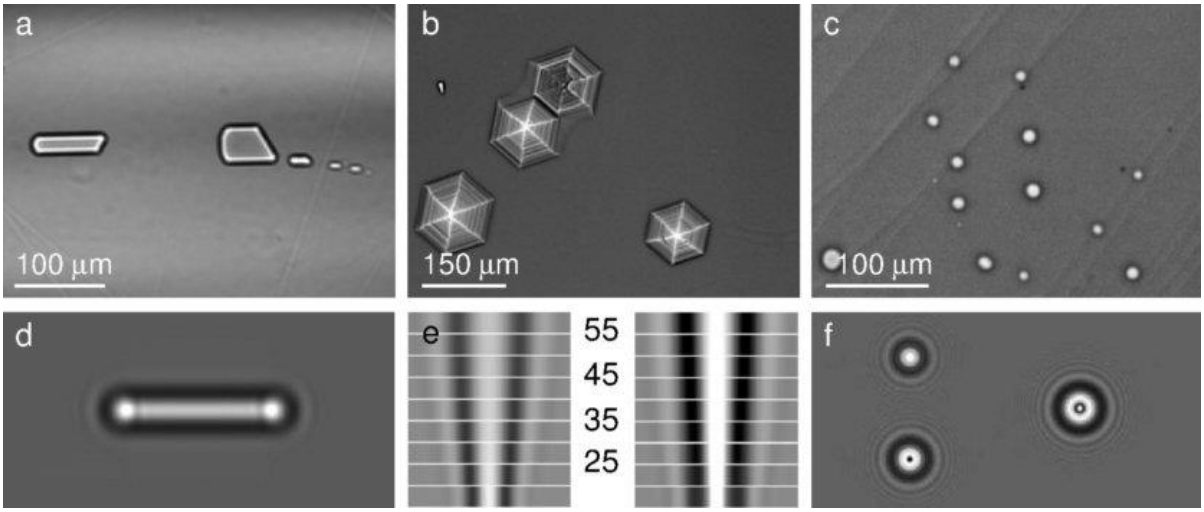

Figure 1. Phase-contrast images of micropores obtained using monochromatic (a) or pink (b, c) SR beam. Simulated images of pores with different shapes in SiC crystals: capsule (d), pipe (sample-detector distances given in cm by numerals) (e), and sphere (f) .

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Thomson Sources and their applications

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Laser-electron X-ray source based on Thomson scattering is a novel type of high spectral brightness X-ray source. Currently, several Thomson sources are fully operational and even commercially available. In all cases, these are multipurpose setups shared by many investigators. Whereas such Thomson generators are 10 to 30 times larger than the conventional X-ray-tube-based sources, they can provide several orders of magnitude higher brilliance which stimulates applications in traditional X-ray technologies like security, therapy and medical diagnostics. The main advantages of Thomson sources that determine their development and applications are a relatively narrow spectrum (with a sharp drop at high energies); high intensity and directivity of radiation; the possibility of spectrum tuning by varying the accelerator magnetic field and (or) by changing a laser wavelength; the possibility to control the temporal structure of the beam in a wide range of pulse durations. The parameters of Thomson source make it possible to implement many "synchrotron" X-ray methods, for which the X-ray tube power is insufficient, i.e., EXAFS (extended x-ray absorption fine structure) spectroscopy, anomalous X-ray scattering to small and large angles ("elemental contrast"), K-edge subtraction angiography, and others [1, 2].

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Collagen materials for tissue engineering

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Biocompatible materials obtained by the technology of structuring connective tissue proteins have high biocompatibility and bioresorption. The technology we have developed for obtaining a new collagen material allows us to obtain collagen matrices that have increased plasticity, biocompatibility with respect to human epithelial cells, and an expanded closed network of pores. The technology makes it possible to obtain a collagen frame material with a thickness of 110 - 150 microns, a strength of 80 - 100 MPa, a relative elongation with a tear resistance of 40 - 47%. The collagen matrix has been successfully used for the cultivation of connective and bone tissue cells, the rheological properties of the collagen matrix allow it to be used for tissue bioprinting. The formation of structures similar to natural collagen is difficult for the technologies that are available today. Electrospinning and deposition technologies cannot establish the long-range orientation of collagen fibers. Collagen films obtained by lyophilization cannot replicate the extracellular matrix of living biological systems. The present invention expands methods for the technological production of collagen materials, and also provides a new collagen matrix that has properties of structural similarity to the human cell matrix. The Oriented Collagen Matrix has a natural resemblance to the human cellular matrix. The collagen layer has a developed rough surface of ordered collagen fibrils and a smooth surface of the matrix structures of collagen and integrin proteins. The creation of scaffold materials based on a collagen matrix is an important application of our technology for tissue engineering. Collagen matrix technology allows the creation of three-dimensional scaffolds with biologically similar structural direction of fibrils [1,2]. The extracellular matrix plays an important role in the morphological development of tissues and organs (G. Avtandilov et al., Journal of Applied Crystallography, 33: 511-514 (2000); P. Lazarev et al., Annual International Conference of the IEEE Engineering in Medicine and Biology - Proceedings, 2000, v. 4, p. 3230-3233; Cuttle et al., Wound Repair and Regeneration, 13: 198-204 (2005)). Biomedical research involves significant efforts to obtain materials with the desired orientation of collagen fibrils by mechanical unidirectional stretching or by structuring liquid crystal collagen. The technological process opens up access to biologically similar structures of collagen fibrils, which improves both the bioresorbtion of the matrix and the receipt of structured growth of living tissue cells.

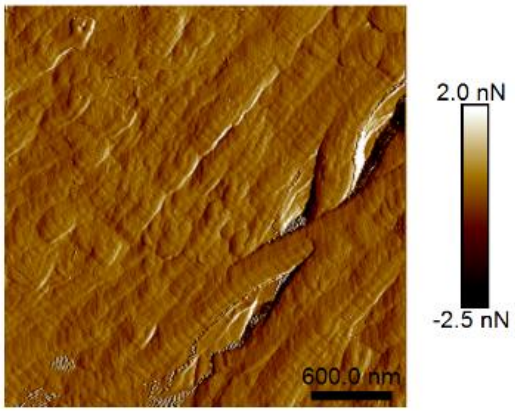

Fig. 1 AFM measurements of the collagen matrix, a directed network of fibrils is visible

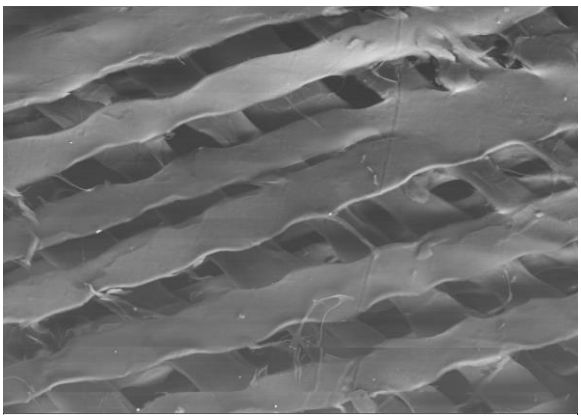

Fig 2. Electron microscopy of collagen matrix

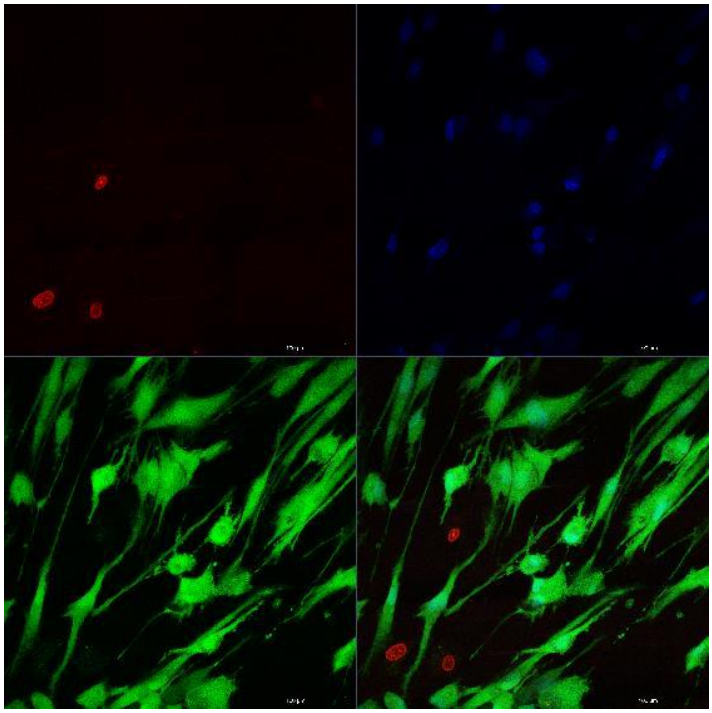

Fig 3. Cell proliferation in the collagen matrix scaffold

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Nanoscale Self-Assembly under Synchrotron Light

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Soft matter and nanomaterials are two largely overlapping notions that greatly revolutionised materials science in the last decades. Here one of the key concepts is the self-assembly, which leads to the appearance of structural features on the scales ranging from a nanometer to microns. In this lecture, I shall mainly focus on synchrotron studies of the self-assembly at the nanoscale. After a short general introduction into the topic, I shall discuss the self-assembly of magnetic and non-magnetic cubic colloids with rounded edges [1,2]. In-situ studies of the self-organisation of semiconductor quantum dots at a liquid interface [3,4] will be illustrated. I shall describe our recent time-resolved study of the formation of microtubes [5,6], which we use as templates for the fabrication of chiral colloids [7,8]. I shall also present examples of recent and on-going research studies [9].

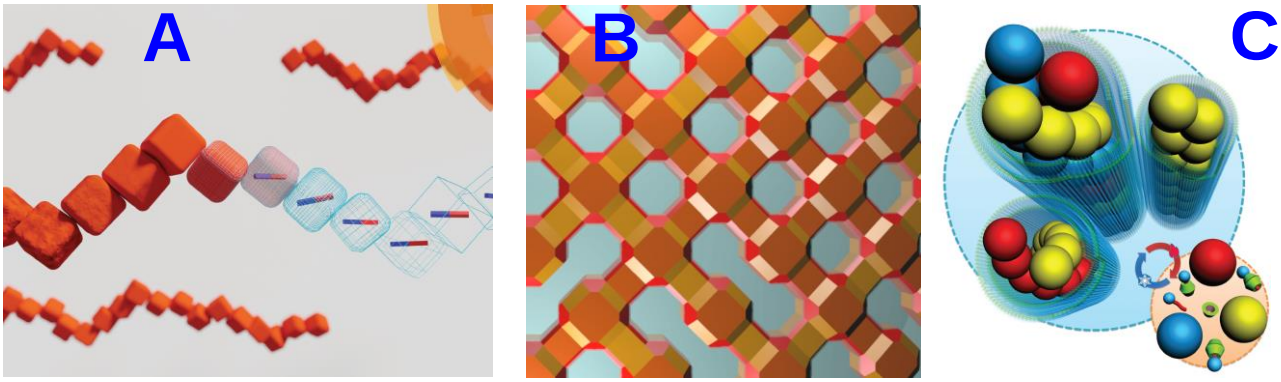

Figure 1. Schematic cartoons of A: 1D assemblies of hematite microcubes constrained by competing shape- and dipole-induced anisotropies; B: 2D lattice of PbSe nanocubes on a liquid surface; C: self-assembly of isotropic colloidal spheres in cylindrical confinement into various structures.

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Advanced Peak Profile Analysis Techniques for Materials Characterization: Advances due to Synchrotron Radiation

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Development of peak profile analysis methods started almost immediately with the application of X-rays for diffraction on crystals. The pioneering work [1] in this field belongs to Paul Sherrer – a famous Swiss physicist. He derived a famous equation which relates the crystallite size with full width at half maximum (FWHM) of diffraction peaks:

$$D = \frac{K\lambda}{\beta \cos\theta}$$

Where D is a crystallite size, K is a constant depending on shape of the crystallites, λ is the wavelength, β is WFHM, and θ is the position of diffraction maxima.

Although Sherrer's consideration was oversimplified as he didn't consider all complexity of materials' structure, his equation is still widely used in materials science and chemistry to characterize the size of the crystallites. It was soon understood that other factors like lattice strain can also contribute to the profile broadening and Stokes and, later, Williamson-Hall approaches appeared.

It is not possible to mention all the important milestones of peak profile analysis history within a short text of an abstract. I would like only to mention here, that in recent decades more versatile models of peak broadening appeared in the literature. The models, which take into consideration the dislocation structure of materials (like modified Warren-Averbach and modified Williamson-Hall methods) seem to be most promising.

Another important discussion is the terminology used in peak profile analysis. For instance, the common term "crystallite size", which is popular in English-language scientific literature is misleading and a more accurate term "size of coherent scattering regions" should probably be used instead. However, it is an important question of how the coherent scattering region looks inside the polycrystal and how it is related to other structural elements like grains, subgrains, etc.

The last important issue is the application of peak profile analysis to understand the evolution of the structure of materials during some external action – deformation, heating, or a combination of these. Thanks to a progress in synchrotron sources, we are now able obtaining the X-ray diffraction patterns in-situ and observe the accumulation of dislocations, their rearrangement and distribution by burgers vectors. As an example of such studies, I can mention recent research done by our group [2]. By application of synchrotron X-ray diffraction, we were able to characterize the structural evolution of materials during the friction and wear.

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Detonation nanodiamonds. Technology properties and applications

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In present-day nanotechnologies, the place, which has thus far been occupied by the traditional, so-called "top-down", processes, is being increasingly claimed by those termed "bottom-up", i.e., processes based on assembly of nanosized building blocks for developing materials with required properties.

Diamond has been well known from time immemorial. Applications of this material, truly unique in its beauty, hardness, and chemical stability, keep expanding as years go by. For hundreds of years mankind has used natural diamonds. It is only from the beginning of the mid-1950s that industrial production of diamond on a commercial scale in reactors capable of maintaining the required high pressures, of tens of thousands of atmospheres, and temperatures of about fifteen hundred degrees Kelvin, was announced to the world.

Presently, when most technologies are moving with confidence from the microscale into the nanometer-scale world, demand has arisen for diamonds of the corresponding size. Such nanodiamonds were first synthesized in the Soviet Union in the 1960s, and their industrial production, initiated in the late 1980s. The starting raw material for nanodiamond synthesis was the carbon, present originally in explosives, and the high pressure and temperature needed for formation of the diamond structure from carbon atoms were reached as a result of the explosion itself. The short duration of the explosion accounts for the small size of the diamond crystallites, which measured a few billionths of a meter only [1].

The presentation discusses technology, properties and applications of diamond nanoparticles produced by explosive method, so called detonation nanodiamonds (DND) and based on the last results obtained by "Nanodiamond group" of Ioffe Institute [2-5].

In the review, I will present:

- an approach for production of stable hydrosol of DND particles with average size 4-5 nm [2],
- an unusual the sol-gel transition in DND suspension [3]
- a method for modification of DND surface with different metal ions [4],
- a new approach for high-pressure high-temperature synthesis of perfect single crystals diamond from DND particles [5]

The presentation will also discuss current and possible applications of detonation nanodiamonds.

The research of "Nanodiamond group" is supported by Ioffe Institute program (project 0040-2019-0013).

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Conductive polymers for smart textile applications

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Smart textiles are one of the most exciting innovative technologies in manufacturing. Smart textiles can be defined as textiles that are able to sense and respond to changes in their environment. They can be divided into two classes: passive and active smart textiles. Passive smart textiles can change their properties depending on the impact of the environment. Shape memory materials, hydrophobic or hydrophilic fabrics, etc. are part of this category.

Active smart textiles are fitted with sensors and actuators in order to connect internal parameters to the transmitted message. They are able to detect different signals from the environment such as temperature, light intensity and pollution to decide how to react and finally to act using various textile-based, flexible, or miniaturized actuators (textile displays, microvibrating devices, light-emitting diode (LED), organic light-emitting diode (OLED)). The «decision» can occur locally in case of embedded electronic devices (textile electronics) to smart textile structures or remotely in case the smart textile is wirelessly connected to clouds containing data base, servers with artificial intelligence software, ect.

Highly functional yarns with carefully developed and specially selected chemical and physical-mechanical properties serve as raw materials for the so-called innovative textiles. Such materials open up the broadest prospects for the technical textiles of the future.

For more than 10 years, the team of authors has been conducting research work in the field of development and research of polymer composite materials in the form of threads/fibers, which are widely used in the creation of smart textiles, both active and passive [1-7]. Within the framework of cooperation with LLC "Ampertex" and NWCTT, the team conducts research in the field of manufacturing textile products from yarns modified with carbon nanotubes, which allows heating and preserving the heat of the human body, without limiting the possibility of its movement, exercise and professional duties, with providing all aesthetic functions.

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