Posters Session B

Tuesday, September 6

PB1

THE INFLUENCE OF Y AND Ce IONS TO CHANGES OF CRYSTAL STRUCTURE OF ZIRCONIA OXIDES

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Zirconia (ZrO_2) is an important ceramic material with an increasing range of applications. Zirconia ceramic components have excellent fracture toughness, very good wear resistance, high corrosion stability, low thermal conductivity and coefficient of thermal expansion in range of steel. All these properties characterize zirconia as a leading structure material for a dental medicine [1, 2] and solid-oxide fuel-cell design [3] and catalytic technologies [4]. This material are drawn attention towards the remarkable structural properties: with increasing temperature the McCullough-Trueblood structure [5] of pure zirconia with monoclinic symmetry ($P2_1/c$) transforms to tetragonal symmetry ($P4_2/nmc$), by approximately 1180 °C [6] and then to fluorite structure with a cubic symmetry (Fm-3m) [7] starting about 2370 °C with melting by 2716 °C [8]. The difference between the cubic and tetragonal structure is the alternating distortion of the O-atom columns along the 4_2 axes i.e. the O-atom displacement along the c axis. The alloying of pure ZrO_2 with CeO_2, or with lower valence oxides such as CaO, MgO, La_2O_3, Y_2O_3 or certain other metal oxides, it is possible to stabilize the tetragonal phase at room temperature. These metastable phases are analogous to those in pure zirconia but have dopant ions which substituted Zr^{4+} ions and a corresponding concentration of oxygen vacancies to retain charge neutrality.

The main aim of the work is the determination of influence of Y and Ce ions on the features of crystal structure and microstructural parameters of zirconia ceramic. The Y and Ce doped zirconia samples have been obtained through the standard ceramic technology, a mixture of yttrium zirconia oxides $ZrO_2 - x\%Y_2O_3$ (x = 0; 4% and 8%) and a mixture of cerium zirconia oxides $ZrO_2 - y\%Ce_2O_3$ (y = 10; 15% and 20%) being used in proportions established *a priori*. After grinding and pressing, the samples were shaped in form of cylinders. Cylindrical samples were finally treated at a temperature of 1500 °C (6 h) in air. The structural analysis of zirconia oxides was performed by using a conventional X-ray diffractometer with CuK radiation and the time-of-flight (TOF) High Resolution Fourier Diffractometer (HRFD) (Fig.1 and 2) at the IBR-2 pulsed reactor in Joint Institute for Nuclear Research, Dubna, Russia [9]. The difference of quality of experimental results

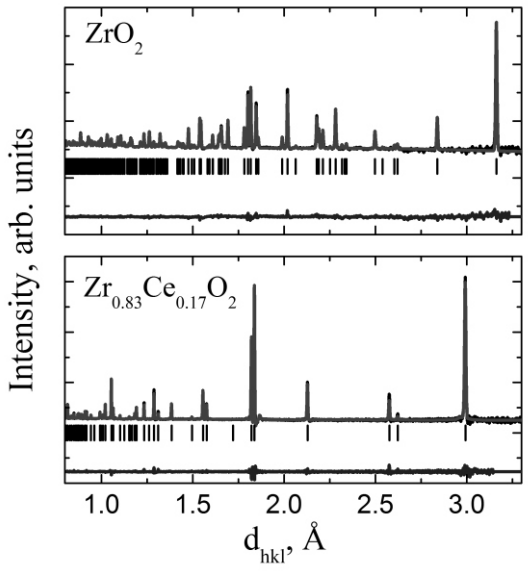

Figure 1. Diffraction patterns for the $Zr_{1-x}Ce_xO_2$ samples measured using HRFD at room temperature, the data were treated using a Fullprof program.

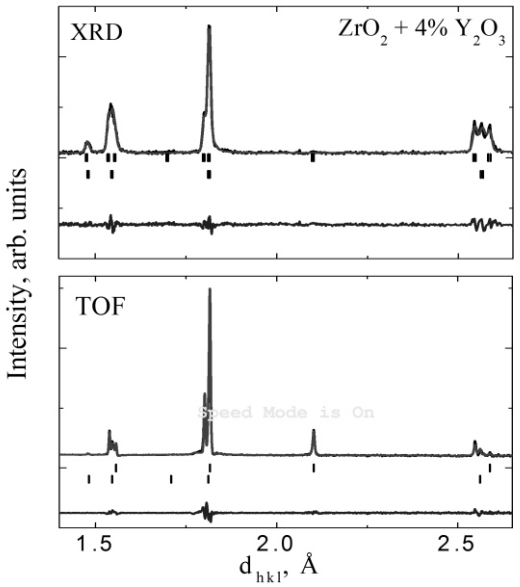

Figure 2. X-ray and neutron diffraction patterns for zirconium oxides measured at room temperature, the data were treated using a Fullprof program.

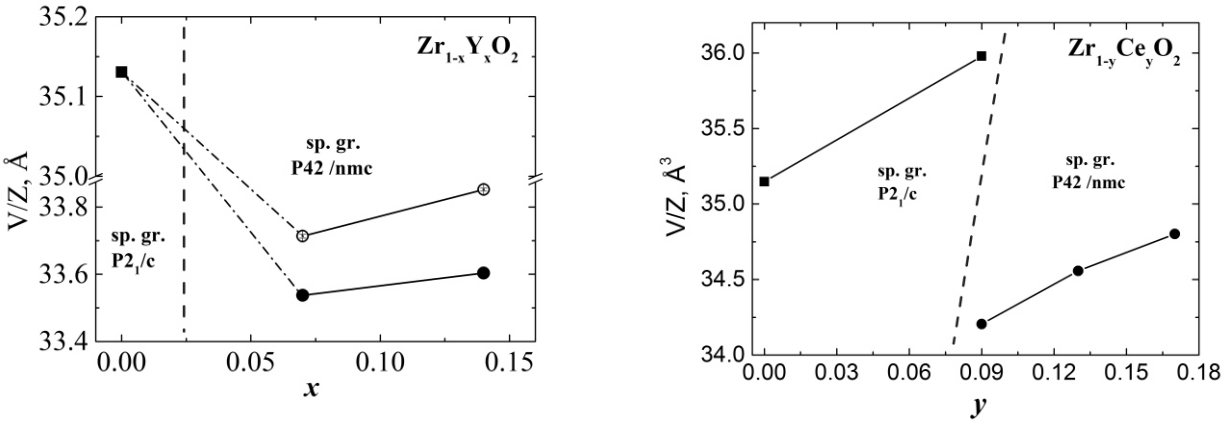

Figure 3. Variation of the unit cell volume of $Zr_{1-x}Y_xO_2$ *(left)* and $Zr_{1-y}Ce_yO_2$ *(right) vs.* concentration (*x or y*) of doped ions. Full square – phase with monoclinic structure, full and empty circles – normal and a weakly distorted tetragonal structures, respectively. Z—the number of molecules in the unit cell.

from conventional X-ray diffractometer and HRFD are shown in Fig.2. Ceramic samples doped with Y ions are inhomogeneous, however the low resolution of conventional X-ray diffractometer do not allow to determine the type of crystal structure of impurity. According to high resolution data an impurity phase has a weakly distorted tetragonal structure. The volume of unit cell increases as the concentration of Y ions is increased Fig.3.

The substitution of the Zr with Y or Ce in zirconium oxides leads to a change of the phase composition, and a transition from the monoclinic to tetragonal structure. Concerning the substitution of Zr with Ce we observed a difference of the phase composition between the surface layer of the sample and the phase composition of the bulk samples. We attributed this difference to the various oxygen concentration in the surface layer and in the bulk sample. The doping of ZrO_2 by Y or Ce ions leads to increase of broadening of diffraction peaks. Such a behavior has been explained by an increase of microstresses and/or a decrease of average size of coherent blocks. The subsequent increase of concentration of Y ions up to 8% leads to an increase of distortion in crystallites of tetragonal phase.

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PB2

INFLUENCE OF HIGH PRESSURE AND TEMPERATURE ON CRYSTAL STRUCTURE OF DOUBLE PEROVSKITES

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Spintronic devices [1-4], such as sensors, spin diodes, spin transistors based on the effects of giant and tunnel magnetoresistance have significant advantages over conventional electronic as they use the spin characteristics of materials.The double perovskites with molar formula $A_2B'B''O_6$ (where A= La; Pr; Sr; Baet al.; B' and B'' = W; Co; Mn; Fe; Moet al.) [5, 6] have been brought recently into thecenter of scientific interest because of their unique physical properties: high value of the Curie temperature (T_c ~ 420 K), large values of the negative magnetoresistance (MR) (up to ~ 30 % at 4.2K), almost 100 % - degree of the spin polarization of conduction electrons. These properties allows to apply double perovskites in equipment of spintronic for creating of spin valvesor magnetic sensors. Such functional properties are the main feature of double perovskites having a tetragonal structure I4/*m* with a superstructure ordering of Fe^{3+} and Mo^{5+}cations.

The magnitude of the spin effect of these materials is highly dependent on the degree of spin polarization and on the density of states at the Fermi level. In turn, both valence state and average size of cations B' and B'' influence onto the density of states at the Fermi level. In the ideal double perovskite structure, the ordered arrangement of FeO_6 and MoO_6 octahedra is observed with accommodation of Sr^{2+}cations in the voids between them. In this case, the antiferromagnetic interaction between the spins of iron cations Fe^{3+} ($3d^5$, S=5/2) and molybdenum Mo^{5+} ($4d^1$, S=1/2) is realized with the formation of ferrimagnetic structure, which determines the half-metallic state complex oxide with two separate conduction bands formed by hybridized states of $4dt_{2g}$-orbitals of the molybdenum ions and $3dt_{2g}$-orbitals of the iron ions. This half-metallic nature of the electronic states leads to an almost complete polarization of the conduction electron spins in strontium-molybdenum double perovskites.

The main aim of this work is definition the regularitiesof influence of external factors (low- and high temperatures: from 10 to 400 K, high pressure: up to 5 GPa) onto crystal and magnetic structures of double perovskites $Ba_{2-x}Sr_xFeMoO_6$. All samples have been prepared by solid-state method at 1200 C (10 h) in flow 5 % H_2/Ar. The investigation of crystal and magnetic structures have been performed by neutron diffraction time of flight (TOF) method at High Resolution Fourier Diffractometer (HRFD) [6] (see Fig.1) and at the specialized spectrometer

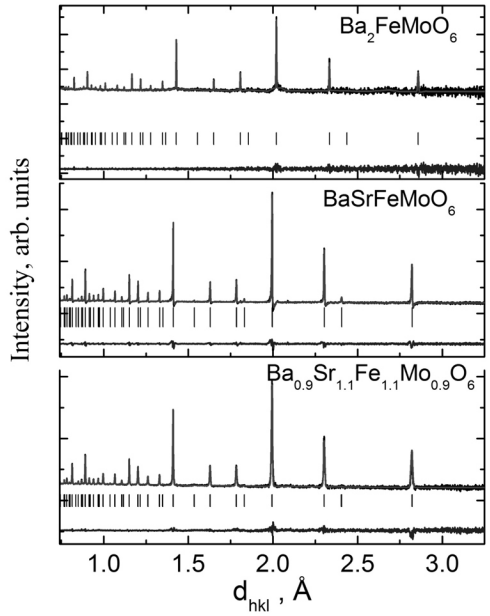

Figure 1. Diffraction patterns for $Ba_{2-x}Sr_xFeMoO_6$ samples measured using HRFD in paramagnetic region, the data were treated using a FullProf program.

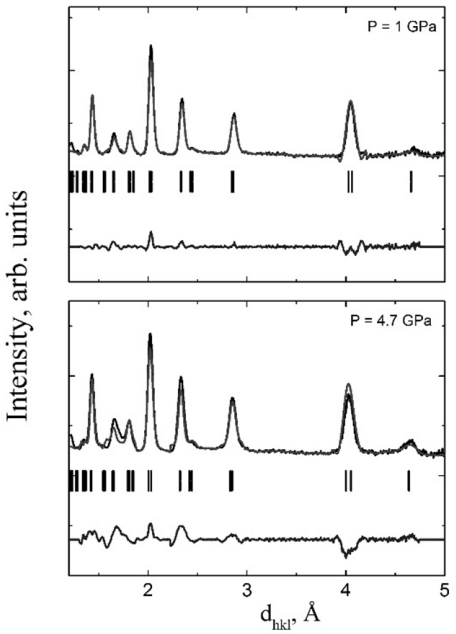

Figure 2. Diffraction patterns for Ba_2FeMoO_6 samples measured using DN-6 at different pressures, the data were treated using a FullProf program.

DN-6. The neutron TOF diffraction data under a high pressure are shown in Fig.2. The Curie temperature was determined by ponderomotive method.

According to neutron diffraction data all investigated samples are homogeneous. The substitution of Ba by Sr ions leads to changes of crystal structure: cubic (F*m*-3*m*) orthorhombic (F*mmm*) tetragonal (I4/*m*). The decrease of ambient temperature leads also to change of type of crystal structure from cubic (F*m*-3*m*) orthorhombic (F*mmm*) or tetragonal (I4/*m*). The volume of unit cell and the Curie temperature decreases as the concentration of strontium ions are increased (Fig. 3). The increase of ambient pressure leads to decrease of volume of the unit cell (Fig.4).

The application of high resolution of HRFD has allowed determining also microstructural parameters. The increase of concentration of Sr ions leads to increase of values of microstresses that can be explained by influence of difference of ionic radii of barium and strontium ions due to their statistic distribution.

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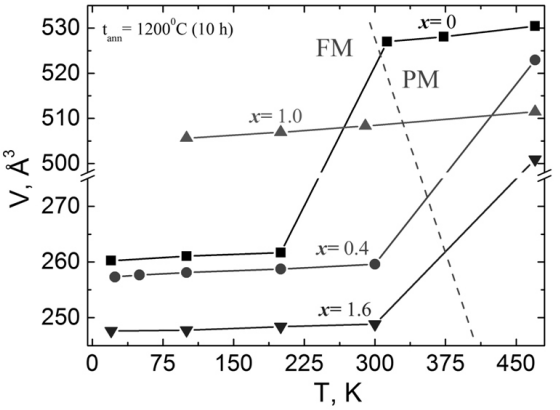

Figure 3. The concentration dependence of volume of unit cell of $Ba_{2-x}Sr_xFeMoO_6$ vs temperature.

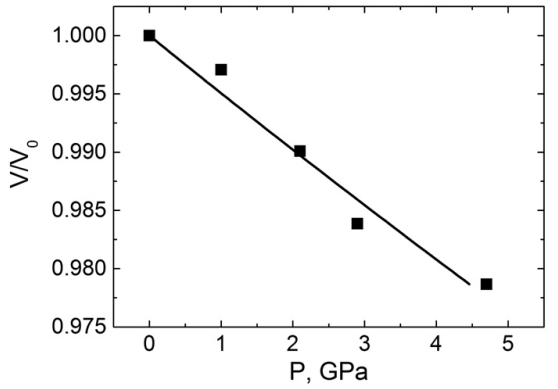

Figure 4. The dependence of volume of unit cell of Ba_2FeMoO_6 vs pressure.

PB3

THE REFINEMENT OF ATOMIC AND MAGNETIC STRUCTURE OF $BaFe_{12-x}In_xO_{19}$ (x = 0.1 – 1.2) BY NEUTRON DIFFRACTION

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The barium and strontium hexaferrites and their solid solutions that doped by Al^{3+}, Sc^{3+}, In^{3+} and Ga^{3+} ions [1, 2] attract much attention of researches due to their unique physical properties. These materials have large magnetocrystalline anisotropy, high Curie temperature, relatively large magnetization, as well as its excellent chemical stability and corrosion resistivity. Such excellent functional properties made it possible their practical applications as permanent magnets [3], magnetic recording media [4], and microwave devices. They provide a wide range of potential applications, such as multiple-state memory elements, novel memory media, transducers and new functional sensors are needed. Hexagonal ferrites are successful used in absorption of centimetric radio waves. Such technology is well known as "Stealth" technology. Recently was reported that Co/Ti-substituted hexagonal M-type ferrite is an excellent material for the high-frequency multilayer inductors. Their magnetic structure are well described by the Gorter's model [5], according with this all magnetic moments of Fe^{3+} ions are ordered along with easy magnetization axis that coincide with hexagonal axis.

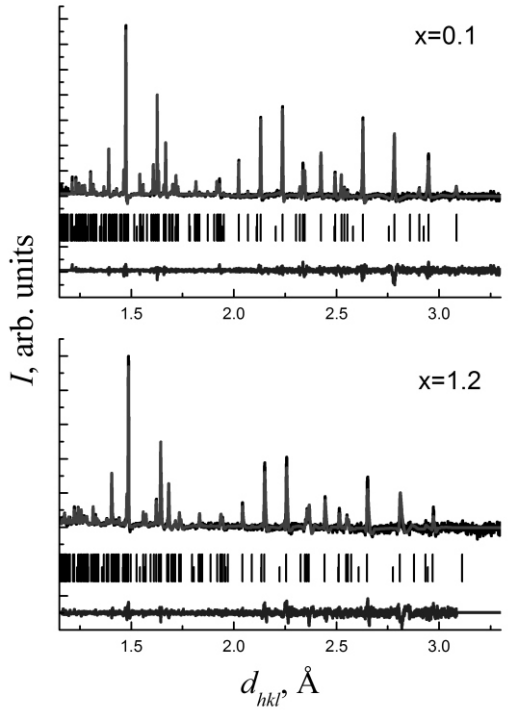

Figure 1. Powder neutron diffraction patterns for the $BaFe_{12-x}In_xO_{19}$ (x = 0.1 and 1.2), sample, measured at 300 K and calculated by Rietveld method.

Recently, in $PbFe_{12}O_{19}$ ceramics [6] with hexagonal structure was found large ferroelectric polarization. Dual ferroic properties of a strong magnetism and large ferroelectricity have been observed in un-doped barium $BaFe_{12}O_{19}$ and strontium $SrFe_{12}O_{19}$ hexaferrites fabricated by a modified ceramic technique. Therefore, barium hexaferrites are new potential multiferroic candidates. It is necessary to point that materials with coexistence of ferroelectricity and ferromagnetism are rare and mostly provided rather weak ferromagnetism. The perovskite $BiFeO_3$ [7] shows weak magnetism, which somehow limit its practical application. Therefore, preparation of a material in which large ferroelectricity and strong ferromagnetism coexist would be a milestone for modern electrics and functionalized materials [8]. The analysis of experimental data of multicomponent oxides such as hexaferrites shows that their physical properties are directly depended versus concentration of diamagnetic substitution, type of crystal structure, crystalline size and even anion stoichiometry.

Therefore, the main aim of our work is determination of correlation between crystal and magnetic structures of barium hexaferrites because of doping In diamagnetic ions. The main interest for investigation of $BaFe_{12-x}In_xO_{19}$ (x = 0.1 – 1.2) is associated with higher meaning of ionic radii In^{3+} (r = 0.94 Å) unlike of Fe^{3+} (r =0.645 Å) [9], therefore, one may expect the significant influence of In ions to crystal and magnetic structures. The investigated $BaFe_{12-x}In_xO_{19}$ (x = 0.1; 0.3; 0.6; 09 and 1.2) samples have been fabricated by the conventional ceramic method. After synthesis at 1200 °C (6 h) samples annealed at 1300 °C (6 h) in air. The crystal and magnetic structures were determined by neutron time of flight method at High Resolution Fourier Diffractometer (HRFD, Dubna) [10] in broad temperature range (10–730 K). Field dependences of specific

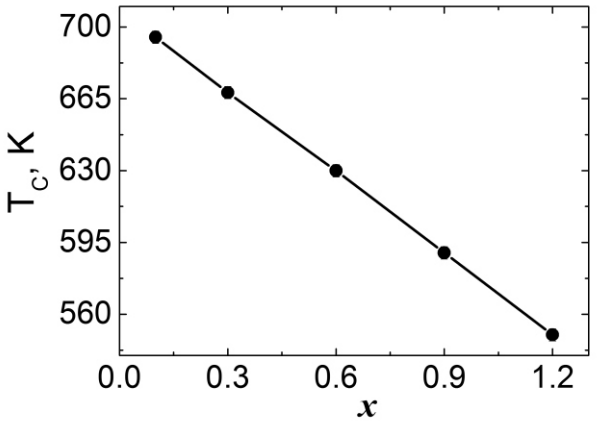

Figure 2. The concentrational dependence of the Curie temperature *Tc* for all the compositions.

magnetization were measured at 300 K by Liquid Helium Free High Field Measurement System (VSM).

According to neutron data all investigated samples are homogeneous and possess a hexagonal structure with space group ($P6_3/mmc$) with two molecules in the unit cell (Z = 2). The pattern of neutron diffraction for the $BaFe_{12-x}In_xO_{19}$ (x = 0.1 and 1.2) is shown in Fig. 1. The volume of unit cell of the In-doped barium hexaferrites is higher than pure $BaFe_{12}O_{19}$ composition. While the indium ions concentration increases the volume of the unit cell is increased. It is due to the bigger ionic radii of the In^{3+} ion (0.940 Å) unlike of the Fe^{3+} (0. 645 Å) [9] ion. With increasing of concentration of the diamagnetic In^{3+} ions in the solid solutions of barium hexaferrite, the number of neighbors of magnetically iron ions is reduced so that the magnetic order is destroyed at lower temperatures. This substitution leads to broke exchange interaction between the magnetic sublattices and to a decrease in the value of their magnetic moments. As result, the specific magnetization decreases from 49.8 emu/g (x = 0.1) to 36.6 emu/g (x = 1.2) at room temperature and the Curie temperature decreases from 695 K to 550 K (Fig. 2.) as the concentration of In ions is increased. A slight change in the magnetic moment of the different compositions appears to reduce the magnitude of the sublattice magnetic moments of the iron ions located at positions 2a and 2b may be due to inhomogeneities in the distribution of indium ions on crystallographic positions in the preparation of the samples. The high resolution of HRFD allowed us to determine the microstructural parameters of our ceramic samples. The calculation of microstresses have been performed for isotropic approximation. The microscopic microstresses increases as the concentration of In ions is increased that can be explained by rising of the system disorder, as a result of the statistical distribution of indium ions in magnetic sublattices, that can make different contributions to the total deformation.

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F15d-003) and Joint Institute for Nuclear Research (Grant No. 04-4-1121–2015/2017).

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PB4

STRUCTURE STUDY OF THE SHAPE MEMORY ALLOY Ni_2MnGa dopped with In

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Shape memory alloys belong to Heusler alloys, which exhibit many interesting electronic and magnetic properties strongly dependent on crystall structure. Magnetic shape memory and magnetically induced reorientation (MIR) are properties, which allow easy straining of these materials in moderate magnetic fields < 1 T. The martensitic transformation is the key concept for deep understanding of these efects.

Ni_2MnGa is good model system where the above mentioned effects can be well observed. Moreover, different behavior can be reached due to the off-stoichiometric composition (with respect to default 2:1:1), as well. Above that, all of this may be accompanied by the creation of the modulated structure. Opossite to above mentioned, another of Heusler alloys Ni_2MnIn does not have to exhibit any martensitic transformation. Therefore, the study of the samples which are doped with In instead of Ga will be the key benefit for understanding the effects connected to the martensitic transformation.

Several samples of with different composition with respect to In have been grown by Bridgeman method. Complex studies of their structure have been performed by high-resolution X-ray scattering and by measurement of diffraction in low and high temperatures.

PB5

PHASE PARTICLES IN Ti – 8.1 at.% Mo ALLOY STUDIED BY ANOMALOUS X-RAY DIFFRACTION

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Nanoparticles of metastable phase form in a certain composition range in metastable â titanium alloys, i.e. Ti alloys in which martensitic transformation from the high temperature body-centred cubic (bcc) phase to low temperature hexagonal close-packed phase is suppressed and the bcc phase is retained in a metastable state. The particles are several tens of nanometres large and are homogeneously dispersed in the matrix. It has been shown recently that the particles are ordered along directions . The particles form during quenching by a displacive transformation during which two neighbouring (111) planes collapse to their intermediate position, creating a hexagonal structure of the phase. The collapse can be only partial and its magnitude is composition-dependent. During ageing, the particles grow by a diffusion-assisted, displacement controlled process during which the solute elements are rejected from the particles into the matrix.

In this research, the chemical composition of the particles and its dependence on ageing conditions was studied in single crystals of Ti – 8.1 at.% Mo alloy by anomalous X-ray diffraction (AXRD) measured on (8-4-42) diffraction peak. The energy dependence of the diffraction peak was studied on a series of samples aged at temperatures 300 °C, 335 °C and 370 °C, with ageing times ranging from 15 min to 512 h. The AXRD measurements were done on beamline BM02 at ESRF, Grenoble, France. The studied

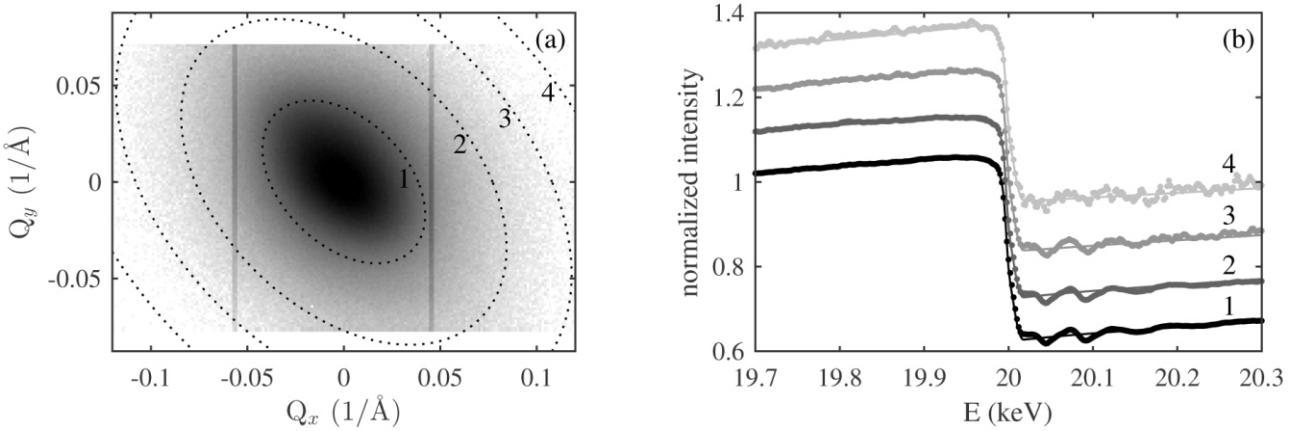

Figure 1. (a) A typical diffraction maximum; ROIs are denoted by dotted lines. (b) Intensity dependence on the energy of the primary beam for individual ROIs (indicated by numbers). Experimental data are indicated by dots, solid lines denote simulated curves. The curves are shifted vertically for clarity.

energy range was near the Mo absorption edge (20.0 keV). The peak exhibited an elliptical shape, which is related to the shape of the particles in the alloy (prolate ellipsoids). Fig. 1(a) shows an diffraction maximum divided into four elliptical regions of interest (ROIs) denoted by dotted lines. Fig. 1(b) shows intensity evolution with increasing primary beam energy within individual ROIs. The mean Mo concentration in the alloy influences mainly the penetration depth of the X-ray radiation, i.e. the size of the step at the Mo edge. The Mo content in the particles corresponds to the roundness of the curves just below the energy of the Mo edge – with increasing Mo concentration in the ů particles the curves are rounder.

The evolution of diffraction maxima intensity with increasing primary beam energy was simulated by a core-shell model in which the mean Mo concentration in the particle was smaller than the average concentration in the alloy and the centre of the particle was slightly richer in Mo than its periphery. For each aged condition of the alloy, Mo concentrations in the core as well as in the shell of the particles were calculated. Moreover, the mean particles size (i.e. two half-axes of the prolate ellipsoid) was determined from the shape of the diffraction maximum.

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PB6

STRUCTURE DETERMINATION OF A COMPLEX ZEOLITE BY COMBINING ROTATION ELECTRON DIFFRACTION, HRTEM AND PXRD

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Zeolites are porous materials with important industrial applications [1]. Their structures are complex, and sometimes disordered which makes their structure characterization difficult using conventional methods. Electron diffraction (ED) combined with high resolution transmission electron microscopy (HRTEM) is a very powerful method for determination of complex or disordered structures from nano-and submicron-sized crystals. In this work, we used Rotation Electron Diffraction (RED) [2] combined with HRTEM imaging and powder X-ray diffraction (PXRD) to

solve and characterize the structure of the complex germanosilicate IM-18 [3]. The RED method combines discrete goniometer tilt steps (2.0-3.0°) with fine beam tilt steps (0.05-0.20°) to collect 3D ED data from a single particle. More than 1000 ED frames can be collected in less than one hour. Moreover, both sharp spots and diffuse streaks indicating the disorder could be seen from the 3D reciprocal lattice reconstructed from the RED data. IM-18 was first prepared more than 8 years ago, but its structure remained unsolved [3]. RED data of IM-18 consisting of 649 ED frames were collected covering a tilt range of 119.46° with a tilt step of 0.20°. RED data were collected at 200 kV using the software *RED – data collection* on a JEOL JEM2100 TEM. The *RED data processing* software was used for unit cell determination, indexing, and intensity extraction.

The unit cell parameters of IM-18 were obtained from the 3D reciprocal space reconstructed from the RED data, which were also confirmed from the PXRD data. We have found that the average structure is orthorhombic (*Imma*) with a = 5.168 Å, b = 14.984 Å, c = 16.965 Å. Three different polymorphs (*P*2/*m* with a = 10.336 Å, b = 14.984 Å, c = 17.734 Å, = 106°; *Pmna* with a = 14.984 Å, b = 10.336 Å, c = 16.965 Å and *Pnnm* with a = 16.965 Å, b = 10.336 Å, c = 14.984 Å) can be built from the average structure. The difference between the models is the arrangement of double 4-ring (D4R) units within the layer. HRTEM images can provide additional information about the local structure.

We have shown that the average structure of the complex zeolite IM-18 could be obtained directly from the RED data. Finally, it is possible to confirm the structural model by using the PXRD data.

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PB7

XRD AND TEM CHARACTERIZATION OF POLAR GaN LAYERS GROWN BY HTVPE ON SAPPHIRE SUBSTRATES

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Gallium nitride (GaN) is nowadays considered as one of the most promising wide-bandgap semiconductors for numerous applications. Due to the lack of native substrates, the GaN layers are usually grown by heteroepitaxy on foreign substrates. Recently, a modified setup for high-temperature vapour phase epitaxy (HTVPE) was proposed [1] that consists of a cold-wall reactor equipped with a novel Ga evaporation cell. Using this setup, a series of c-oriented GaN layers having the thickness of 5 m was deposited by HTVPE directly on sapphire substrates.

We investigated the effect of selected growth parameters such as growth rate or ammonia flow during the deposition on the quality of produced layers. The density of screw and edge threading dislocations (TDs) was determined using a combination of high-resolution X-ray diffraction (HRXRD) and Monte Carlo simulation [2]. For the latter, we suggested a novel description of the dislocation bunching that is based on the transmission electron microscopic (TEM) observations [3]. The density of TDs was correlated with the residual stress in the samples that was determined using a modified $\sin^2$ method [4].

TEM (Fig. 1) revealed furthermore a change of the contrast between the [0001]-oriented columns that were attributed to GaN grains with Ga and N polarities. The grains with inverse polarities arise already during the nucleation process at the GaN/sapphire interface and their boundaries look like antiphase domains. In symmetrical XRD geometry, these defects cause additional XRD line broadening in azimuthal direction and thus affect the accuracy of the screw TDs determination.

The effect of the antiphase boundaries on the determined TDs densities and on the correlation between the defect density and the residual stress will be discussed.

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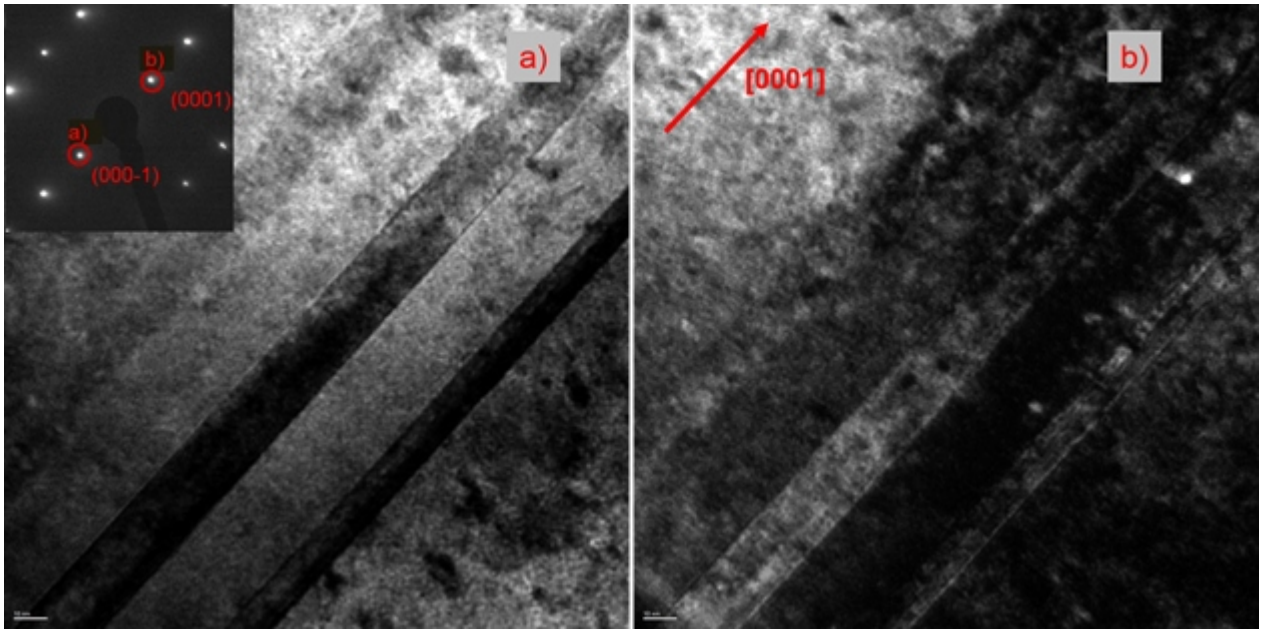

Figure 1. Dark field TEM images recorded in the diffraction spots +0001 (a) and –0001 disclosed domains with inverse polarities. The selected diffraction spots arisen due to the multiple scattering are marked in the electron diffraction pattern (left).

PB8

BEHAVIOR OF 2-MERCAPTOBENZIMIDAZOLE UNDER HIGH-PRESSURE CONDITIONS

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2-Mercaptobenzimidazole, (MBi), is used as an inhibitor of corrosion of metals, owing to its ability to coordination and formation of a protective layer on the metal surface. The compound can be present in the thion, thiol and zwitterionic forms

Single crystals of MBi have been *in situ* grown in a Merrill-Bassett diamond-anvil cell (DAC) [1] and the structures were determined by X-ray diffraction. The crystal structure of this compound was determined for the first time in 1976 [2]. High-pressure crystallizations at isochoric conditions from methanol solution lead to the unsolvated crystals. 2-Mercaptobenzimidazole crystallizes in monoclinic space group $P2_1/m$ up to 2.5 GPa and it's the same phase as that obtained at ambient conditions. In all crystal structures molecular packing is governed by NH···S hydrogen bonds.

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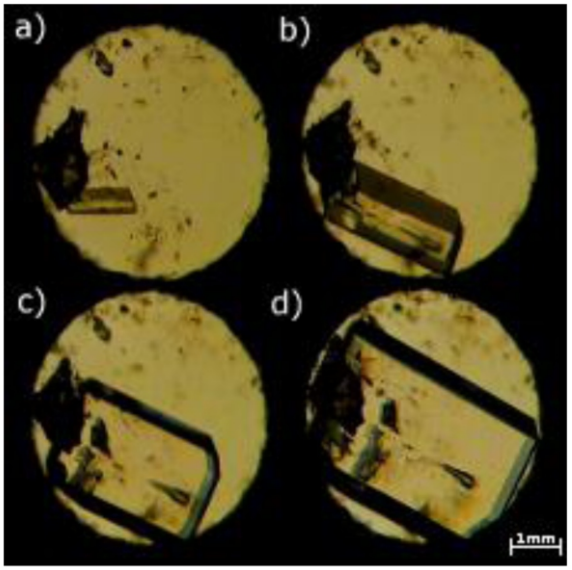

Figure 1. Stages of isochoric growth of MBi at 1.30 GPa: (a) one seed at 423 K; (b) at 393 K; (c) at 323 K; and (d) single crystal of MBi at 296 K/ 1.30 GPa.

PB9

HIGH PRESSURE STUDY OF 3-HYDROXY-4,5-DIMETHYL-1-PHENYLPYRIDAZIN-6-ONE

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3-Hydroxy-4,5-dimethyl-1-phenylpyridazin-6-one (**1**) is a derivative of maleic hydrazide, known in three polymorphic forms, an analogue of nucleic bases, applied as the growth inhibitor in agriculture; derivatives of **1** are used as antiviral drugs. At ambient conditions 1 precipitates as monoclinic crystals of space group C2/*c*, with four symmetry-independent molecules (Z'= 4) (polymorph) [1] and $P2_1/c$, with Z'=1 (polymorph).

Currently in the Cambridge Structural Database there are about 10% of crystal structures with Z' >1; more than 66% of them have one symmetry independent molecule (Z'= 1) in the structure, in 8.04% structures there are two independent molecules (Z'= 2), in 0.45% Z'= 3, in 0.44% Z'= 4 and in 0.04% Z'= 6. Also fractional Z' is quite frequent for symmetric molecules: Z'= 0.25 is present in 1.56% of deposited structures, Z'= 0.33 in 0.65%, Z'= 0.5 in 22.4% and Z'= 1.5 in 0.25% of deposited structures.

However, the significance of the Z' number for the properties of crystals is still puzzling. We have shown that in the crystal of **1** pressure clearly favours the low Z polymorph . However, phase requires recrystallization above 0.4 GPa and it could not be obtained at ambient conditions, even though the crystal of phase ă have been kept in open vials for one year at least. The Z' reduction in the high-pressure polymorph of **1** contrasts with the phase transitions in thiourea [2] from its ambient pressure phase V (Z'= 1) to high-pressure phase VII (Z'= 3); as well as the transition of urea [3] from the ambient-pressure phase I (Z'= 0.25) to phase III (Z'= 1) above 0.48 GPa; above 2.8 GPa urea transforms to phase IV (Z'= 0.5).

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PB10

SKEW ASYMMETRIC ARRANGEMENT OF X-RAY DIFFRACTION FOR STRUCTURAL DIAGNOSTICS OF MULTI-LAYER SEMICONDUCTOR MATERIALS

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Berg-Barrett topography technique is effectively used even today to estimate the quality of the structure of semiconductor materials. This paper shows that some modification of this technique can increase by an order of magnitude its sensitivity to the peculiarities of the morphological structure of surface and its structural distortions.

This diffraction geometry is different from traditional schemes in that normal to crystal entrance surface does not lie in the diffraction plane [1-3].

This experimental scheme opens up new opportunities for a layer by layer visualization of structural changes in the subsurface crystal layers, and also allows increasing the determination of distribution of strain types in the subsurface layers using a series of diffraction reflection curves. Among possible diffraction planes (*hkl*) for the proposed diffraction geometry preference should be given to those for which the difference between the values of angles and is insignificant, i.e. – 1 (– Bragg diffraction angle, – disorientation angle of entrance and reflecting crystallographic surfaces).

There occurs a kind of X-ray optical increase of topogram resolution (by about an order of magnitude) due to a reduction of X-ray penetration depth and a stronger influence of the subsurface structural defects on the formation of diffraction pattern.

This geometry of topography there is some kind of increase of X-ray optical resolution on topograms (almost by an order of magnitude) due to the decrease of the penetration depth of X-rays and a strong influence of surface structural defects on the formation of the diffraction pattern.

Some examples show that application of reflection topography geometry gives an opportunity to perform selective studies of structural changes in the subsurface layers of

crystalline compounds after different external influences with sufficiently small step (0.01-0.1 μm).

Implementation of X-ray skew asymmetric topography provides a way to obtain different defect projections on the entrance crystal surface in the same (*hkl*) reflex. This gives possibility:

- to select optimal experiment geometry for the most complete interpretation of defect type, to study its orientation and elastic strains, the features of strain field distribution;

- to readily estimate the degree of structural disordering of surface layers under different external influences (laser irradiation, ion etching, ion implantation, etc.);

- to investigate the main features of X-ray diffraction contrast formation, as well as to perform quantitative diagnostics of surface relief, i.e. to estimate the height and step parameters of the asperities.

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PB11

LONG-RANGE INTERACTION OF RESIDUAL DEFORMATION FIELDS IN THE X-RAY TRIPLE-CRYSTALLINE LLL-INTERFEROMETER

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This paper presents experimental and theoretical research of long-range interactions of extended sources of residual strains formed by the action of the diamond indenter on the initial surface of analyzer of silicon LLL-interferometer. We present new approaches of solution of inverse problems in definition of possible sources of residual strains and mutual influence at a considerable distance from them.

Simulation of moiré images with known deformation field is based on algorithm of Takagi equations numerical solution, presented in [1]. We performed the analysis of formation of moiré intensity distributions from interaction of deformation fields of two rows of distributed local concentrated forces with different positions: 1) placed parallel or perpendicular to the diffraction vector **H** and spaced at different distances from each other; 2) intersecting or touching (Fig. 1b,c); 3) placed at an angle to each other (Fig. 1e,f).

Interaction of scratches deformation fields or concentrated forces rows appears on experimental and calculated moiré images by the formation of the characteristic distribution of moiré stripes, as well as the presence of mutual moiré stripes that begins on one of scratches and end on the other. Especially, it is well observed on fig.1e. As a rule, the formation of new deformation moiré fringes occurs in alternating signs (compression-tension) deformation area near the concentrated forces, where the maximum rate of phase change is presented. In general, the shape of the moiré stripes usually reflects the resulting displacement field of deformations sources.

The introduction of phase object (Fig. 1c, f) as original indicator of strains allows to detect and specify the mechanisms of formation of moiré stripes near sources of deformations and determine the contribution of each of them. In addition, the phase moiré significantly influences on formation of image in the area between scratches. We observe here some changes not only with shape and contrast of deformation moiré stripes (dark-white to white-dark), but also with their number.

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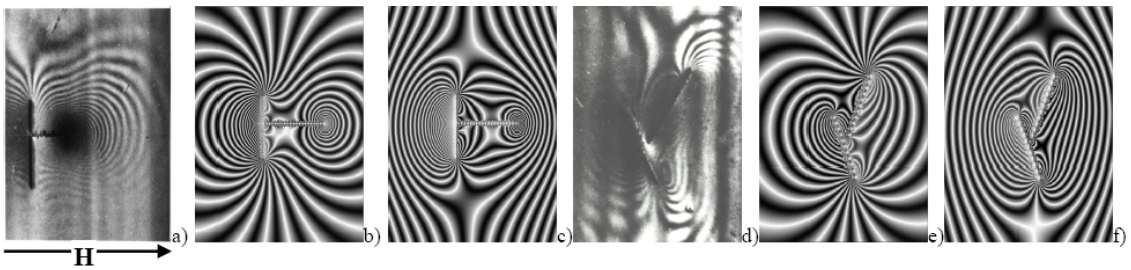

Figure 1. Experimental moiré patterns (220), Cu$K_{\alpha 1}$, (a, d) and corresponding them ones, calculated without (b, e) and with phase object (c, f) (= 1800 m).

PB12

STUDY OF SINGLE DISLOCATIONS BY TOPO-TOMOGRAPHY WITH X-RAY LABORATORY SET-UP

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Manageable dislocation structure formation in crystalline materials allows to extend their industrial applications, *e.g.,* for creating novel X-ray sensors. On the other hand, as is well-known, various external loads drive the dislocation motions that change the crystal properties. In this report the study results of the crystalline lattice disorder in Si due to individual defects (introduced dislocations) are presented. To characterize 3D structure defect the topo-tomography method is used [1, 2]. On the example of the Si samples and using the X-ray laboratory setup, all the measurements were performed in the Laboratory of reflectometry and small angles scattering, Shubnikov Institute of Crystallography, RAS, Russian Federation. The procedure for introducing dislocations in Si-single crystals is fully described. The dislocation topographic contrast experimentally obtained is analyzed based on the numerically simulated 2D- and 3D-images. The reconstruction results of the topo-tomography images are discussed in the terms of the displacement elastic field being around dislocations [3].

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PB13

QUANTITATIVE SYNCHROTRON TOPOGRAPHY APPLICATION FOR CRYSTALS OF GROOVED Si AND $ZnGeP_2$

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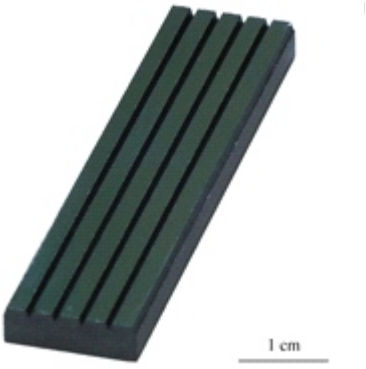
Quantitative topography is a novel technique which makes possible not only visualization of the real structure of crystal but also obtaining information on the rocking curve of any region of interest [1]. We present the results of investigation of two samples of different origin.

In the field of high energy physics the new device for multiple deflection of the proton beam was developed recently [2], it consists of several bent strips of silicon. In the device the successive bending of silicon strips at the surface of a thick plate is achieved due to internal stresses in the material of the crystal as a result of applying mechanical grooves. Method of quantitative topography at synchrotron radiation was applied for measurement of bending of



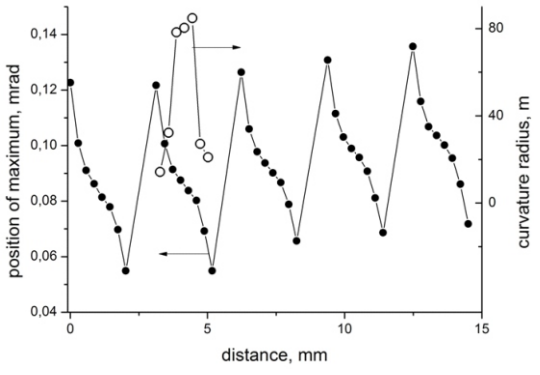


Figure 1. View of the deflector (left) and the dependence of the position of local maxima of the rocking curves on the coordinate across the crystal and the bending radius of a stripe, depending on the location (right).

the individual strips of the deflector and the crystal as a whole (Fig.1). Silicon (511) asymmetric monochromator and symmetric (333) Bragg reflection of the sample were used. The results of the measurement are compared with the results obtained in the proton beam.

$ZnGeP_2$ crystals have nonlinear behaviour in the infrared region and their performance depends on the crystal quality [3]. Several crystals were studied using the quantitative topography at synchrotron radiation. Silicon (511) asymmetric monochromator and the (336) reflection of the sample were used with almost 0 dispersion. Crystals displayed macroscopic curvature, subgrains, growth striation and other defects. Nevertheless the rocking curves width in some regions demonstrated high crystalline perfection. The results will be useful for improvement of the growth process.

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PB14

IMAGING AND DENSITY ESTIMATION OF ICY MATERIALS UNDER TEMPERATURE-CONTROLLED CONDITIONS USING PHASE CONTRAST X-RAY COMPUTED TOMOGRAPHY

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X-ray micro-computed tomography (CT) by means of the absorption contrast X-ray imaging technique has been effectively used for imaging of gas filled pores within materials using X-ray CT. On the other hand, one of major difficulties is detecting changes in water phases (water, ice and hydrated water) within materials such as food product, biological materials. Because of low density and small density differences in these materials and devices, the quality of the imaging dataset is not high enough to visualize multi-phase materials without a contrast agent.

Density resolution of phase-contrast X-ray imaging is much higher than that of the conventional X-ray transition imaging since the light elements, which are composed of such molecules as hydrogen, carbon, and oxygen, have approximately 1000 times larger phase-shift cross sections than their absorption cross sections. Additionally, this technique made it possible to obtain density mapping of crystals or biological tissues with a density resolution up to several mg/cm^3.

We have applied the phase-contrast X-ray CT to non-destructive imaging of gas hydrates including guest gas molecules, such as methane, which is being expected as unconventional natural gas resource. In this presentation, experimental method under temperature-controlled conditions using phase-contrast X-ray imaging method will be presented.

PB15

PHASE CONTRAST TOMOGRAPHIC IMAGING OF POLYMER COMPOSITES

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X-ray computed tomography (CT) is a non-destructive method for 3D imaging of inner structure of objects. However, classical CT imaging is restricted to materials with not negligible X-ray absorption. Using the phase contrast imaging technique, observation of samples consisting of a low absorption material becomes possible, moreover materials with similar absorption properties can be distinguished. For a long time, phase contrast imaging has been restricted to synchrotron radiation sources or special techniques requiring a spatial coherence of radiation. Along with recent development of X-ray micro and nanofocus X-ray tubes and X-ray detectors a phase contrast imaging becomes available also with laboratory systems [1]. Single-distance propagation based method of phase imaging is widely used on synchrotron radiation sources. Thanks to its simplicity against the other phase imaging approaches, it is possible to apply this method with laboratory CT devices with partially spatial coherent X-ray sources. For a quanti-

tative information about phase shift of X-rays transmitted from the object, phase-retrieval algorithms have been derived [2, 3].

Here, we report on utilization of phase contrast X-ray imaging and application of phase retrieval on data from RIGAKU Nano3DX CT system, specifically on sample of carbon reinforced polyester (PE) fibres. RIGAKU Nano3DX is equipped with 3300 × 2500 pixel^2 X-ray CCD camera and Cu rotatory target working at acceleration voltage 40 kV and current 30 mA. The CT measurement was performed with the optical head with 20× magnification, enabling the field of view 0.9 × 0.7 mm^2. A binning 2 was set which determined the linear voxel size of the CT data at 0.54 μm. The exposure time was 5 s resulting in the time of scanning 1.1 hour. The reconstruction was realized using CT reconstruction module within VGStudio MAX. Phase retrieval algorithm was applied using AnkaPhase software [3].

In a tomographic section (Fig. 1), edge enhancement caused by phase effects is visible. In this image, a segmentation of different phases of material is complicated. Application of phase retrieval algorithm on X-ray images leads to the CT data with better contrast and lower signal to noise ratio (SNR), and consequently allows easier segmentation (Fig. 2). Moreover, another phase of the studied material, having probably slightly different density, becomes visible (in Fig. 2 pointed by white arrow).

In this paper, we showed the possibility of phase contrast X-ray imaging with RIGAKU Nano3DX device, which was demonstrated on a polymer-carbon composite sample.

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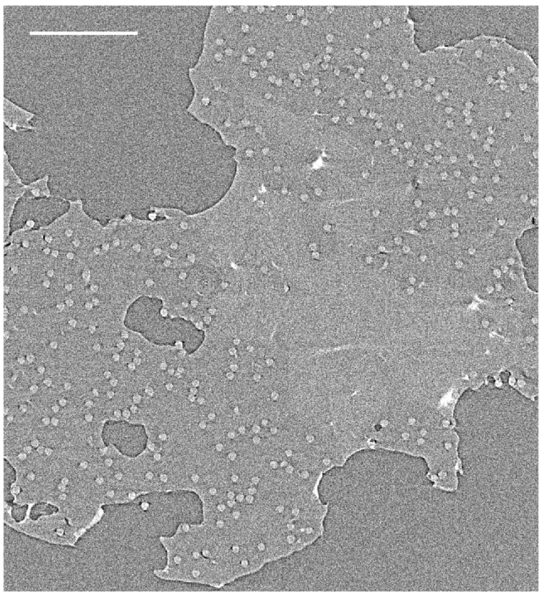

Figure 1. Tomographic section of carbon reinforced PE fibres. Length of a scale bar is 100 μm.

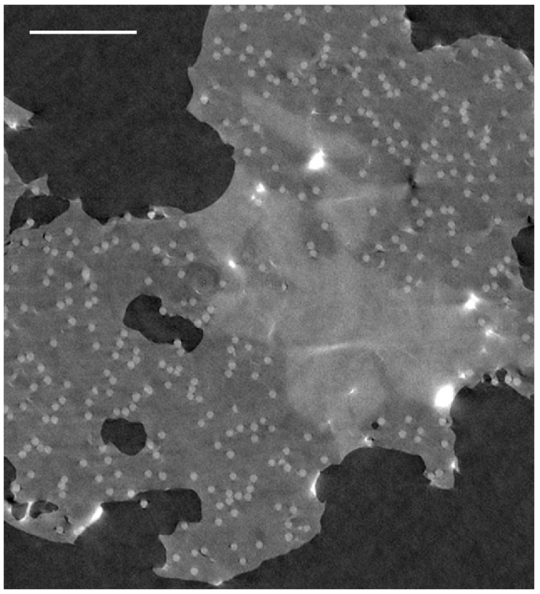

Figure 2. Phase retrieval on tomographic section of carbon reinforced PE fibres. Length of a scale bar is 100 μm.

PB16

OPTIMIZATION OF CHANNEL-CUT X-RAY OPTICS FOR HIGH-THROUGHPUT AND HIGH-RESOLUTION LABORATORY SMALL-ANGLE X-RAY SCATTERING EXPERIMENTS

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The latest generation of solid and liquid-based microfocus X-ray sources coupled to multilayer optics provides wide collimated X-ray beams with high flux of 10^9 photons/s and low divergence close to 0.5 mrad. Here, we present new solutions of Ge channel-cut crystal optics for the next generation of high-throughput X-ray collimators to fully utilize potential of the new microfocus sources, particularly for SAXS expriments. We performed comprehensive ray-tracing simulations of various beam-compressing channel-cut collimators and their combinations. Two principal versions of the channel-cut collimators are presented: i.) the high flux configuration based on a single V-shaped channel-cut compressor and ii.) the high-resolution dispersive configuration which consist of a compressor and a symmetric parallel channel-cut monochromator. We discuss also the most promising high-flux hybrid solution based on the quasi-dispersive configuration of two channel-cuts with different working diffractions. The numerical simulations are supported by experiments preformed on a liquid-jet Ga microfocus X-ray source. The Fig. 1 shows a comparison of the conventional Kratky collimator with the above mentioned channel-cut collimators. Both channel-cut collimator designs clearly outperform Kratky collimator in terms of the output flux at the same resolution and vice versa.

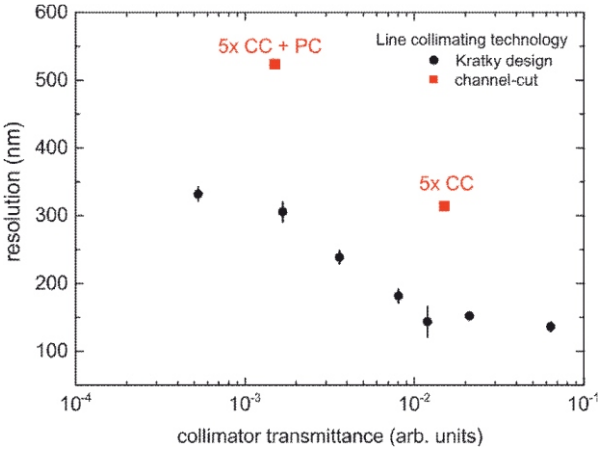

Figure 1. Experimentally measured resolution and transmittance of Kratky and selected channel-cut line collimators.

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PA17

PERFORMING HIGH-RESOLUTION IN-PLANE BRAGG SURFACE DIFFRACTION USING MULTIPLE-BEAM DIFFRACTION

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In-plane X-ray diffraction is critical for nondestructively understanding misfit strains and their relaxation of epitaxial structures, surface nanostructure patterns, etc. Tremendous efforts have been made to develop extremely asymmetric diffraction schemes, particularly grazing-incidence diffraction (GID), to achieve partial in-plane diffraction. In addition to their experimental complexities and difficulties, the various GID schemes all unfortunately suffer from many severe difficulties, including (i) low diffraction efficiency (due to X-ray total specular reflection), (ii) low angular resolution (associated with the broad diffuse grazing diffraction peaks), and (iii) extremely low spatial resolving resolution for surface mapping and imaging due to the large footprint of the grazing-incident beam on the sample. These drawbacks have significantly hindered the applications of high-resolution X-ray diffraction although more powerful X-ray sources, particularly fourth-generation synchrotrons with small two-dimensionally collimated

X-ray beams, are available or emerging. Here, we present a simple scheme to solve the long-lasting difficulties of in-plane diffraction. It only utilizes the symmetric reflection geometry with large incident angles (corresponding to small X-ray footprint and high spatial resolution) but can produce two individual diffraction peaks through the x-ray multiple-beam diffraction (MBD) effect. These two peaks correspond exactly to the out-of-plane and in-plane diffraction processes, respectively, such that the two kinds of structural information can be revealed simultaneously but independently. In particular, if the primary out-of-plane diffraction is a forbidden reflection, only pure in-plane diffraction will be activated. Although it involves no grazing incidence, the intermediate diffracted waves in the MBD process propagate almost exactly parallel to the surface. Therefore, this scheme is extremely sensitive to surface structures, and thus can be used to study and image a wide range of epitaxial (as well as bulk) materials, including ultrathin films and multilayers, lateral surface nanostructures, and bulk crystal phase transitions [1,2].

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PB18

HIGHLY EFFICIENT AND TUNEABLE SILICON DIFFRACTIVE LENSES FOR HARD X-RAY MICRODIFFRACTION EXPERIMENTS

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Many currently available structural and chemical imaging techniques such as micro X-ray diffraction (microXRD), X-ray absorption spectromicroscopy (microXAS) or scanning X-ray fluorescence microscopy (SXFM) require highly efficient focusing of hard X-rays. Such applications widely rely on performing experiments with micro- and nanometer size beams, with the wish of having an extremely bright beam on the sample under investigation in order to obtain the best signal to noise ratio. This holds true in particular in macromolecular crystallography, since increasing difficulties are observed to grow large crystals for experiments aimed at solving the structure of challenging targets, such as G-protein coupled receptors, a major drug target. It is therefore of paramount importance to design focusing modules with ultra-high efficiency, yet showing focusing capability for reaching beams with micro- and nanometer size.

One material of choice for fabricating lenses in this context is silicon, since it has no absorption edge at hard X-ray energies, and thus shows substantial transparency. We developed silicon lenses based on the zone plate equation which present the ideal kinoform profile for efficient X-ray diffraction [1] thereby allowing a theoretical efficiency reaching unity, only limited by material absorption. To address the challenge of fabricating the ultra-high aspect ratio nanostructures required for efficient hard X-rays diffraction, we used the metal-assisted chemical etching technique (MAC-Etch), based on a wet-etch process [2-4]. We here optimized the MAC-Etch approach to fabricate lenses which consist of diffractive structures with pitch down to 150 nm and a depth of up to 6 m (see Fig. 1). The adopted design relies on a pair of tilted linear lenses, thus decoupling focusing in both dimensions, which additionally shows great flexibility in tuning the central operating wavelength [5].

Microfocusing experiments were conducted at microXAS - X05LA beamline of the Swiss Light Source, Paul Scherrer Institut and an average efficiency of 63% was obtained for 1D focusing at 12.4 keV (see Fig. 2). Such diffraction efficiency is substantially higher than the ones expected from binary diffractive structures, inherently limited to 40.5% for pure phase structures.

The presented results will range from the design of our optics with optimized optical performances, the development of the nanofabrication process followed by the efficiency characterization at hard X-ray energies. The significance of the silicon lenses developed will be presented in context of hard X-ray micro-focusing experiments

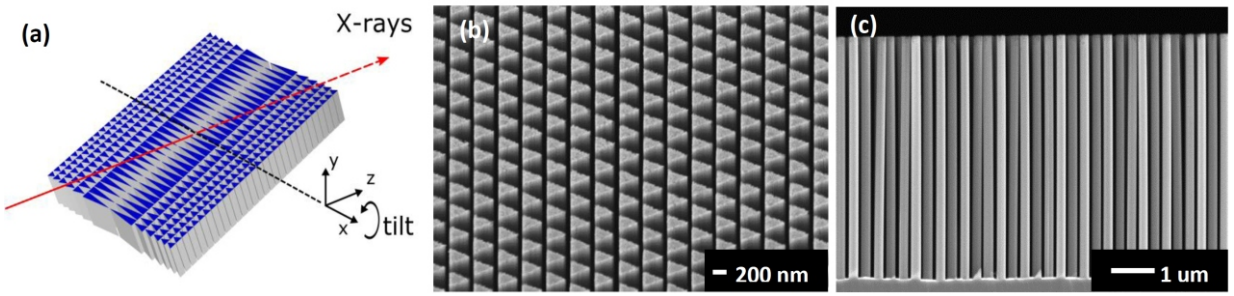

Figure 1. **(a)** View of a kinoform tilted lens, consisting of a periodic arrangement of small triangular pillars acting as lens elements for hard X-rays. SEM pictures of the silicon linear lenses realized with the MAC-Etch technique: **(b)** 20° tilted view of 200 nm pitch zones. **(c)** Cross section of same nanostructures with aspect ratio of 30:1.

performed at the Swiss Light Source, Paul Scherrer Institut.

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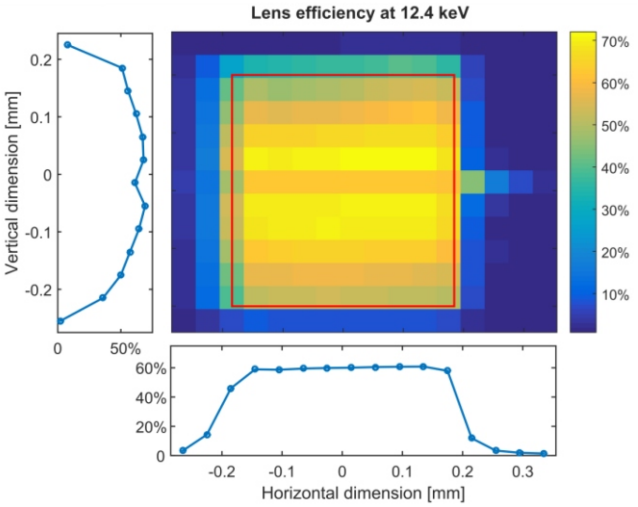

Figure 2. Space-resolved diffraction efficiency for 1D focusing of a kinoform silicon lens measured at 12.4 keV (see colorbar), obtained by raster scan with a pencil beam. The red frame indicates the lens footprint with 400µm x 400µm aperture. The two sub-figures show the averaged efficiency along each dimension over the lens footprint, to evidence the good homogeneity obtained.

PB19

COMPLETE CHARACTERISATION OF REFLECTION GRATING PROPERTIES BY ATOMIC FORCE MICROSCOPY (AFM), X-RAY DIFFRACTION (XRD) AND GRAZING INCIDENCE X-RAY FLUORESCENCE ANALYSIS (GIXRF)

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For the characterization of the profile used in diffraction gratings one usually refers to scans being made by use of scanning electron microscopes (SEM) or atomic force microscopes (AFM). These techniques cover only very small areas at the sample, which make difficult to extrapolate the grating performance in terms of expected diffraction efficiency. In addition they only report the surface topography and do thus not allow us to learn about the properties of eventually buried layers and interfaces. When subjecting a sample to X-ray grazing incidence conditions, the investigated area at the grating is significantly increased. By combining angularly resolved Grazing Incidence X-ray fluorescence spectroscopy (GIXRF) and X-ray reflectivity (XRR), one then also has access to the depth composition of the material, given by the angularly resolved fluorescence contributions for the different constituents of the sample under investigation and to its optical parameters, respectively.

This study wants to demonstrate the performance of GIXRF in the characterization of such periodic structures. A grating with laminar profile has been measured systematically depending on the grazing angle of incidence and on the orientation angle of the structure around its pole. In this case when the trajectory of the probing beam is parallel to grooves with rectangular profile, i.e. with flat side walls, the system could be looked at as an assembly of many parallel micro-mirrors. The periodicity of the structure will necessarily lead to diffraction. In this case the diffracted intensity will be found on a cone symmetrically oriented around the plane of incidence. Then the totally diffracted intensity should be identical to the reflectivity of a plane mirror with the same coating. And likewise one should not be able to distinguish the angularly resolved GIXRF from that of the plane mirror. This is infact observed and indicates thus the successful realization of the groove profile with the indicated shape.

When now the grating is rotated around its pole such that the beam trajectory is inclined with respect to the grooves, then the fluorescence intensity from buried layers should exhibit significantly different angularly dependent behaviour. This is observed and it will be shown that the now more structured angular dependence is in agreement with predictions employing geometrical ray-tracing calculations. X-ray diffraction data confirm the layering of the system as deduced from the the previously described experiments.

PB20

MULTILAYER LAUE LENS FABRICATION AT FRAUNHOFER IWS DRESDEN

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The fabrication of multilayer Laue lenses (MLL) is a challenging task. At Fraunhofer IWS we have implemented all necessary steps to manufacture these lenses completely in our own laboratories.

For the deposition of the multilayer stack a method is required, which allows for precise thickness control and smooth interfaces between individual layers in particular. Magnetron sputtering is the state of the art technique to obtain thick multilayer stacks with nanometer precision.

For MLL usually multilayer systems based on two materials with $MoSi_2$ or WSi_2 as the absorber and Si as the spacer have been used. The result is a multilayer system dominated by compressive stress of the amorphous materials. This might lead to delamination of the multilayer stack or cracking of the substrate during the deposition process. We have developed a three-material system, which can significantly reduce the internal stress of the multilayer structure. We have chosen to use Mo as absorber material., which features tensile stress. The stress can be balanced out against the compressive stress that is contributed by Si. To obtain nearly optimal relative thicknesses a third material, Carbon, is introduced as a transition layer. It prevents a significant interdiffusion between Mo and Si and substitutes part of the spacer layer to obtain a nearly optimal lines to spaces ratio.

A laser micro structuring process is utilized to cut the whole lens module out of the multilayer stack. The contour to be cut consists of the narrow lens element (tip) and a wider base structure. With approximately 80 μm width and 200 μm length the contour is very close to the final net shape which is minimizing the efforts in the subsequent fine polishing process. A laser wavelength in the ultraviolet spectrum (343 nm) was chosen to ensure efficient coupling into the multilayer material and a minimum laser spot size of approximately 10 μm.

A new FIB at Fraunhofer IWS is used for the final processing step. Part of the tip is ion milled (Ga+) to the final dimensions of the MLL. The optimal section thickness depends on the X-ray energy of the designated experiment. For 12 keV it is approximately 7 μm.

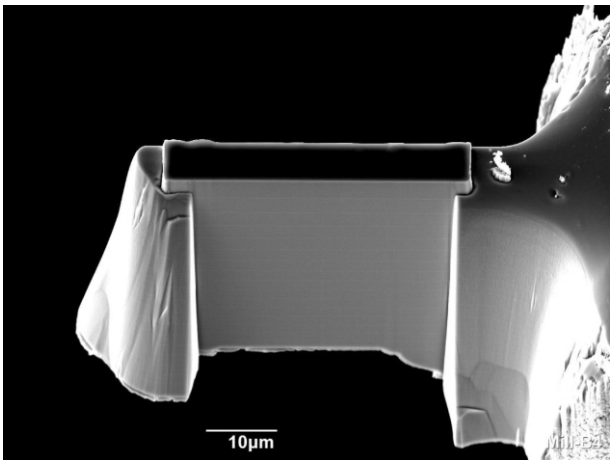

Figure 1: Final shape of the multilayer Laue lens.

PB21

ACHIEVING POINT AND LINE FOCUS USING MULTILAYER LAUE LENSES AT ESRF BEAMLINE ID13

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Understanding the behavior of materials and how their properties change after or during deformation or heating is an important part on the way to improve many industrial materials. Thin films for instance, can show characteristic depth-dependent properties, which are related to structural variations across the thickness, worth to be studied in detail and with high lateral resolution. Local characterization of the crystallographic orientation and stress distribution of such nanocrystalline materials can be analyzed with X-ray methods such as wide-angle X-ray scattering (WAXS). In this case, a high-resolution characterization technique normal to the interfaces is desired and a line focus improves diffraction statistics for homogeneous layers.

We have used multilayer Laue lenses (MLL) with a total aperture of 50 μm to focus hard x-rays to a focal spot size of ~ 30 nm in vertical and horizontal directions. A de-

sign representing 7000 zones according to the zone plate law was deposited with alternating layers of WSi_2 and Si starting with zone number 850. The focal length equals 8.2 mm at 13 keV x-ray energy [1]. The working distance from the order sorting aperture to the sample is design to be 2.7 mm. The lenses were manufactured as flat lenses first. Subsequently an additional SiO_2 layer was deposited on one side of the lens structure. This adds a defined amount of stress to the structure, which is deformed in defined way to obtain a wedged MLL structure [2]. The focal spot size of around 30 nm was determined by ptychography. The geometry of the lens was designed to allow switching between a point and line focus. This was possible by changing the slit position upstream of the lenses only.

Since it is planned to characterize crystalline structures of layered samples in-situ during annealing procedures and due to the small distance between the lenses and the heat source MLL could be exposed to higher temperatures. Therefore, thermal stability tests of MLLs have performed. The lenses have been measured before and after heat treatment and in-situ [3].

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PB22

NON-SCANNING X-RAY FLUORESCENCE SPECTROMICROSCOPY WITH A LABORATORY X-RAY SOURCE

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For many years, energy-dispersive X-ray fluorescence analysis has relied on the use of a single or multi-element semiconductor detector. This technique also has capability of imaging by combining with XY scans of X-ray microbeam on the sample. On the other hand, the advent of high-resolution 2D area detectors like CCD and sCMOS cameras makes the projection-type X-ray imaging much quicker than the conventional XY scan scheme [1-2]. In the present research [3], we have studied the use of CCD camera in ordinary X-ray fluorescence spectra measurement as well as simultaneous imaging of the corresponding elements. In the final, a non-scanning X-ray fluorescence spectromicroscopy with a laboratory X-ray source was developed.

The present X-ray fluorescence spectromicroscopy uses a full-field beam from laboratory X-ray tube (copper target, 1.5 kW, with monochromator) as light source, a micron pinhole collimator as imaging system [4] and a commercial CCD camera as detector. For this setup, its photon energy sensitivity is realized by maintaining the CCD camera working in *single photon count mode* [5]. This microscopy is able to offer X-ray fluorescence spectra upon any region of interest in the observation field; also it supports simultaneous fluorescence-energy-selective imaging of different elements in the sample. The energy resolution is 150 eV at 5.9 keV; the spatial resolution can reach 20 m when a 12 m pinhole is employed. Time-resolved observation is also applicable to chase the element movement process.

Figure 1 shows one application of the microscopy in chemistry experiment. The sample is a self-assembly structure named chemical garden, grown from crystal seeds of manganese sulphate and cobalt chloride in sodium silicate solution. This time, the microscopy gave the X-ray fluorescence spectrum over the whole observation field, as well as the respective element imaging of manganese and cobalt.

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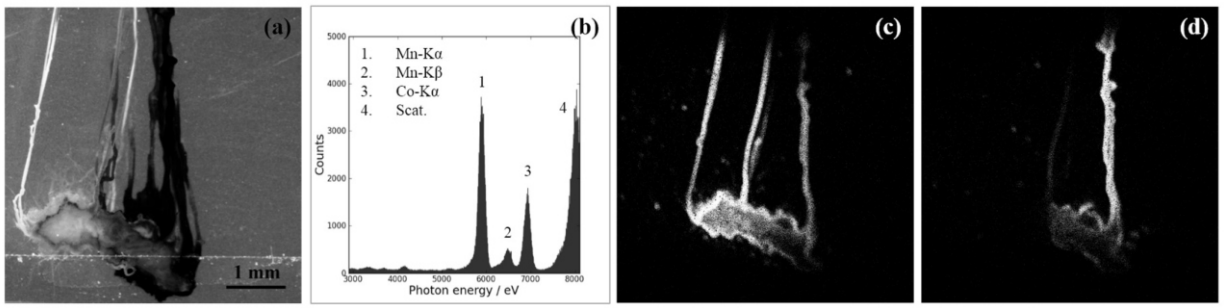

Figure 1. Element imaging of chemical garden branches in surface layers in solution. (a) Optical microscopy photon, (b) Fluorescence spectrum, (c) Imaging of Mn, (d) Imaging of Co.

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PB23

STRUCTURE ANALYSIS OF DRUG DELIVERY SYSTEMS WITH SAXS IN THE LABORATORY

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Small-Angle X-ray Scattering (SAXS) draws increasing attention in the field of pharmaceutical engineering. SAXS is a versatile technique used for shape and size characterization of nanostructured materials between 1 nm and 200 nm. Biological samples, like proteins or viruses are already well known to be investigated with SAXS. Furthermore drug delivery systems like drug loaded vesicles (see example in Fig. 1), where size and shape parameters of the vesicle and the drug are found or granulate powders, where the internal surface obtained by SAXS correlates with the tablet hardness, are interesting examples of applications in pharmaceutical research.

In this contribution we present select applications of biological samples, employing a multifunctional laboratory Small and Wide Angle X-ray Scattering (SWAXS) system, the SAXSpoint. The SAXSpoint system enables SAXS and WAXS studies at ambient and non-ambient conditions, GI-SAXS, in-situ tensile SWAXS experiments and satisfies the advanced user with a wide range of dedicated sample stages, full experimental flexibility and highest resolution. The system provides simple operation, short measurement times and excellent angular resolution, enabled by a smart beam formation concept which includes a brilliant X-ray source, advanced X-ray optics and optimized scatterless collimation while maintaining a laboratory-friendly compact size and small footprint.

Different scattering studies on biological and pharmaceutically relevant samples were performed on the presented SAXSpoint system. Some of the samples required high resolution, i.e. a very low minimum scattering angle in order to resolve large structural dimensions. The unique sample-positioning mechanism enabled WAXS measurements to determine crystallinity without re-aligning any part of the SWAXS system. The presented studies clearly show that high-resolution and high-quality SWAXS data can be obtained with a laboratory SWAXS system.

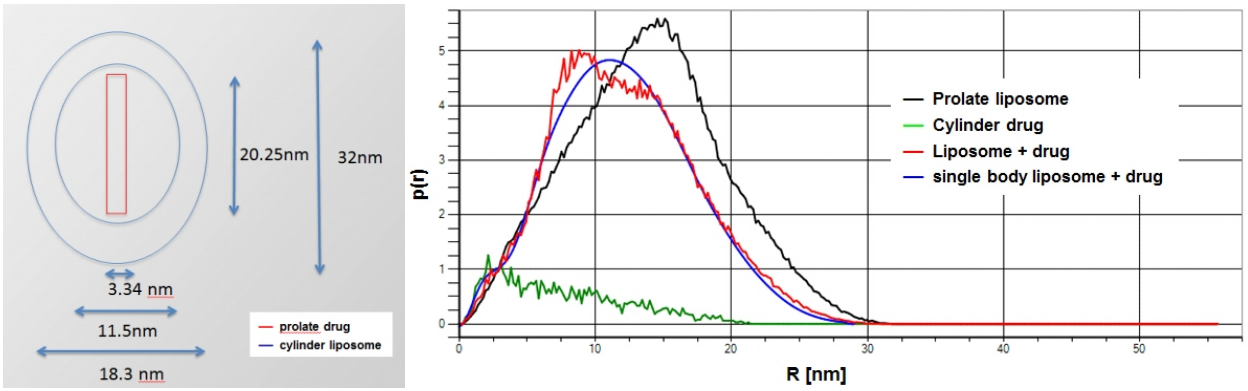

Figure 1. Study of a liposome drug carrier system. Data, obtained from a SAXS-measurement, yielded with aid of single body simulation (simulated Pair Distance Distribution Function on the right side) in the depicted model of the drug-loaded liposome (on the left side).

B24

FIRST TESTS OF X-RAY IMAGING USING PIXEL DETECTORS BASE ON SEMI-INSULATING GaAs

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Semi–insulating (SI) GaAs is one of the most important candidates for fabrication of semiconductor X– and –ray detectors applicable in digital radiology instrumentations [1-4]. Advantage of the bulk SI GaAs is the possibility of fabrication of a monolithic strip or matrix detectors in one substrate due to the creation of the space charge region under each blocking contact, which represents the active part of a pixel detector. In comparison with widely used silicon detectors, SI GaAs reach 20 times higher detection efficiency, faster charge collection due to higher electrical field in sensor. It has no or minimal polarization effect contrary CdTe of CdZnTe detector and linear absorption coefficient for X-rays is smooth in interesting energy range from 15 to 80 keV.

We fabricated pixel detector using SI GaAs substrate with thickness of 350 m. The detection area has a size of 14.1 × 14.1 mm^2 with 256 × 256 pixels and was connected with Timepix readout chip. At first we characterized the detection system and performed tests of homogeneity in term of count rate, test of stability of counting rate and spectrometric resolution of pixel detector. As a source of radiation we used ^{241}Am which generates -ray with 59.5 keV and In for X-ray induce emission of 23.7 keV (K). The detected spectrum is shown on Fig. 1.

Following we tested imaging performance of pixel detector using X-ray source with micro-focal spot size (about 8 m). We used gold testing object with precisely fabricated patterns. The size of the testing object is 0.7 × 0.7 mm^2. In tests we utilize geometrical magnification from 17 up to 35 and the effective size of pixel detector was about 1.6 m. The X-ray image of testing object obtained by pixel detector is in Fig. 2. In the image processing we used only flat field correction. The black vertical line in the left part of the picture represents bad connection line between the pixel detector and read out chip, which can be easily removable.

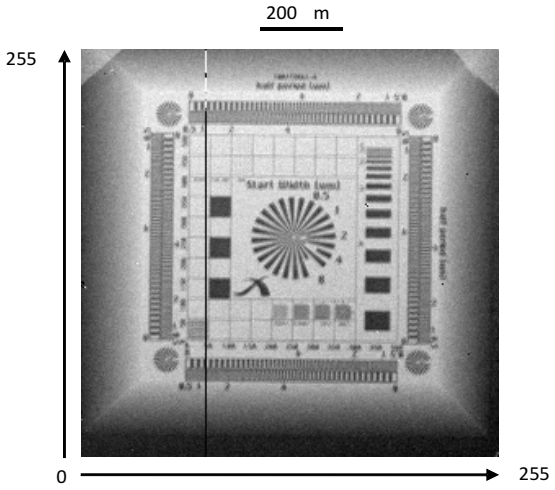

Figure 2. The X-ray picture of testing object. The accelerating potential was 50 kV and current 0.1 mA.

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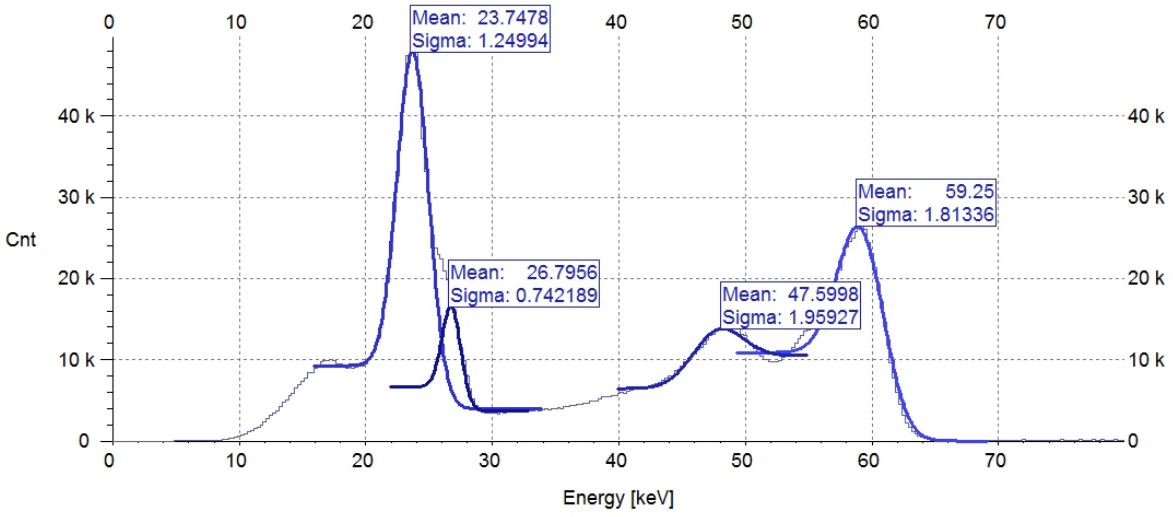

Figure 1. The detected spectrum of - and X-rays from ^{241}Am+In by SI GaAs pixel detector.

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PB25

THE X-RAY BI- AND MULTICHROMATOR – THEORY AND ALIGNMENT

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The X-ray bichromator is a novel crystal device which delivers two arbitrarily chosen wavelengths in one beam. The principle has been already published in previous paper [1]. The method is based on simultaneous diffraction on two different crystallographic planes and on using two properly oriented channel-cut crystals. In this poster we describe detailed procedure how to systematically tune the two chosen wavelengths starting from several basic orientations of crystals. The X-ray multichromator gives three and more wavelengths in one beam. An example of such device is also presented.

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PB26

HIGH-QUALITY ACTIVE SURFACES FOR X-RAY CRYSTAL OPTICS

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We utilize nano-machining methods, in particular single point diamond turning (SPDT) and fly cutting (FC) [1, 2, 3] for preparation of high-quality active surfaces of X-ray crystal optics. We are focusing mainly on Ge or Si, however, other materials such as Al, Cu, Ni or plastics can be utilized for IR optics [4].The nano-machining allows very good form accuracy (0.15 μm over 75 mm dia.) and surface roughness (RMS 3 nm). These values are sufficient e.g. for IR optics, however, X-ray optics needs an additional polishing process. Our goal is to achieve the surface roughness (RMS) far below 1 nm and deviations from planarity in the nanometre range over the lengths of millimetres at maximum suppression of the sub-surface damage (SSD) of the crystal lattice. In addition to the open planar surfaces, we want to address also harder accessible inner walls in the channel cut monochromators.

To optimize machining parameters, more than 100 flat samples were prepared and evaluated so far. The AFM image shows the results obtained on the surface machined with the feed rate of 2 mm/min and surface finishing of 2 μm removal depth that exhibited 0.35 nm RMS roughness in Fig. 1. Micro Raman spectroscopy shows periodic stress variations in the sub-surface area of crystal lattice (<35 nm) in Fig. 2 which follow the surface morphology (ripples) showed in AFM image. These ripples are not desirable for X-ray optics, hence a subsequent polishing is required to approach the goal of 0.1 nm RMS roughness [5] and to suppress the sub-surface damage. TEM analysis on cross sectional specimens thinned down to electron transparency by Ar ion bombardment as the final step confirms a non-invasive effect of the nano-machining as there are no dislocations or other type of damage up to the very surface.

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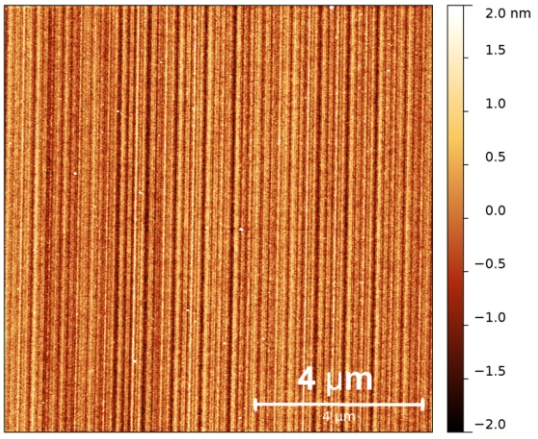

Figure 1. AFM image of low dislocation Ge(220) prepared by flycutting is exhibiting 0.35 nm RMS roughness.

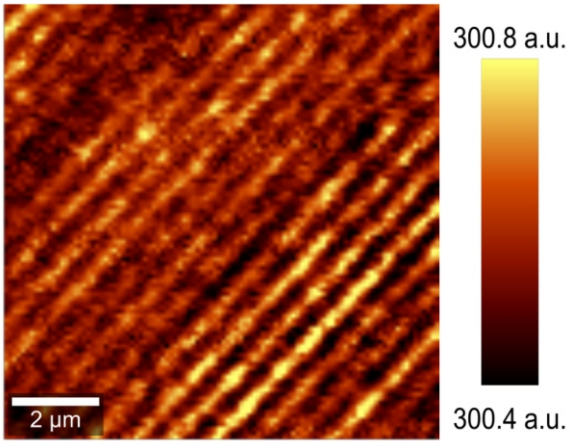

Figure 2. Shift of the peak centre (brighter areas) in micro Raman image corresponds to higher stress in the crystal lattice.

PB27

AN AUTOMATIC SAMPLE POSITIONING SYSTEM FOR NANO-BEAM X-RAY DIFFRACTION MULTI-SCALE MAPPING

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An automatic sample positioning system has been developed that adjusts an arbitrary position of a sample with non-flat surface to the rotation centre of a goniometer. In general, samples with flat surface are adjusted to the rotation centre of a goniometer using a technique that sets samples surface parallel to the incident X-ray and translate samples to intersect the X-ray until transmitted X-ray intensity becomes half. The technique, however, is not available for a sample with non-flat surface. Because, it is difficult to set the sample surface parallel to the incident X-ray and also X-ray is absorbed at the most projecting parts of the sample along the X-ray path. Therefore, the developed system consists of following two steps. Figuration of a sample surface is measured in advance. Using the figuration data, arbitrary position on the sample surface is automatically adjusted to the centre of a goniometer. This system enables synchrotron nano-beam X-ray diffraction multi-scale mapping from 100 nm to a few mm.

The figuration measurement was performed using a laser confocal displacement sensor (LT9010-M, 10 nm resolution, KEYENCE Co.) on an offline goniometer. A sample is set on a goniometer head that has a kinematic mount. Sample positions at the offline and an online goniometers are synchronized prior to the measurement. The sample position at the online goniometer is automatically adjusted to the rotation centre using the data measured at the offline. Fine tuning of the sample position is done by a miniature ultrasonic linear actuator stage with a built-in linear encoder (XDT35-044, 100 nm resolution, Technohans Co., Ltd.). Evaluation of the system is per-

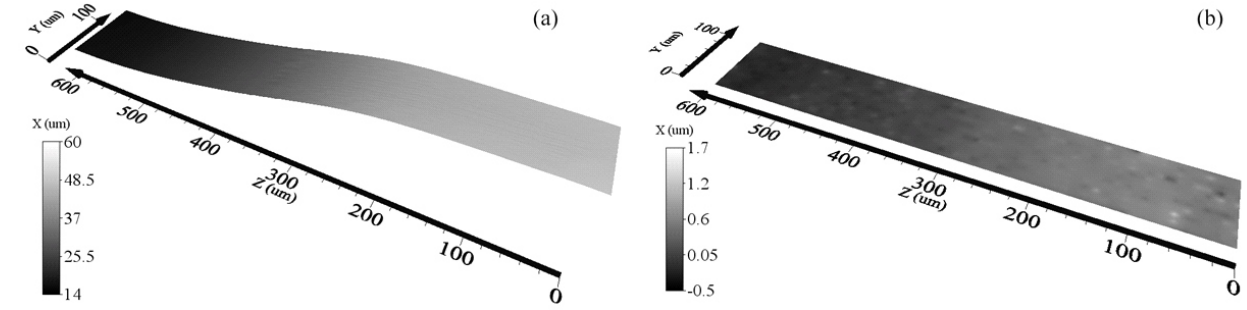

Figure 1. Measured displacement of a sample surface from the goniometer centre without (a) and with (b) the automatic sample positioning system. The sample was cleaved section of a silicon wafer.

formed using a cleaved section of a silicon wafer as a test sample. Fig. 1 (a) shows a measured figuration of the sample. Fig. 1 (b) shows measured displacement of the sample surface from the goniometer centre with the automatic sample positioning system. The sample surface area of 600 × 100 m^2 was 14 ~ 60 m apart from the rotation centre, and the system adjusted the position to an error of -0.5 ~ 1.7 m automatically. The error is sufficiently small so that X-ray irradiation positions will not move more than 100 nm while rotating samples for a few degrees. The development of the automatic sample positioning system independent of sample surface configurations enabled multi-scale (100 nm ~ a few mm) mapping of nano-beam X-ray diffraction.

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PB28

RECENT PROGRESS IN DESIGN AND TECHNOLOGY OF CHANNEL-CUT MONOCHROMATORS

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In flat X-ray monochromators, it is important to steadily improve shape precision (flatness) and to decrease the surface roughness, subsurface damage (SSD) and elastic strains [1] because of increased demands on conditioned beam parameters such as flux density, integrated intensity, reduced wavelength dispersion, degree of collimation, coherence and beam homogeneity. For this, the technique of chemomechanical polishing (CMP) with planetary motion in two dimensions is fully applicable as final technological step because of open surfaces.

Two-bounce channel-cut monochromators (CCMs) preserving beam direction and three-bounce CCMs acting like analysers are widely used in laboratory and synchrotron applications for metrology and partly for imaging applications. The inner active surfaces of CCMs are usually constrained in one direction by the bottom of the channel. The CMP is not possible to be fully used and hand polishing followed by a wet polish etching is applied. Due to generated surface unevenness (e.g. waviness and "orange peel", there may be problems with beam coherence, wavefront flatness and image distortion in imaging monochromators [2,3]. Reverse procedure starting with wet etching is applicable as well. Sophisticated geometrical design (Z-monochromator) allowing to use a modification of CMP was presented in [4]. This technique is applicable for smaller Bragg angles and/or larger channel widths. In some cases this technique is applicable to polish the asymmetric channel-cut monochromators (ACCMs). Typology of symmetrical and asymmetric CCMs for beam compression and expansion was presented in [3]. Beam compressors are suitable for metrology (diffractometry and small angle scattering, SAXS), beam expanders are used to improve resolution in high resolution X-ray imaging including phase contrast imaging.

Recently, a special automated polishing machine imitating hand polishing of CCMs was presented to mimic the manual polishing of CCMs by using a similarly sized and shaped tool, combined with repeatable figure-eight motion and fixed down pressure [5]. The tool for hand polishing was fastened to an X-Y translational stage with a possibility to program several kinematics.

This contribution reports another approach - the single point diamond technology (SPDT), namely technique of flycutting, which was used to process the active surfaces

Figure 1a). SPDT processed surface, flycutting technique.

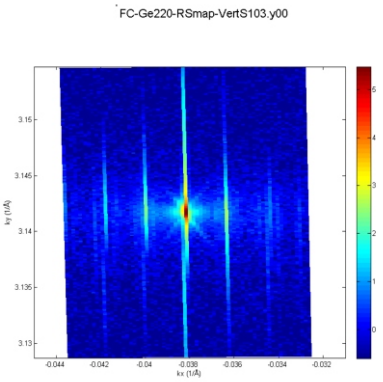

Figure 1b). Reciprocal space map of Ge(220) sample with vertical tool traces perpendicular to the plane of diffraction.

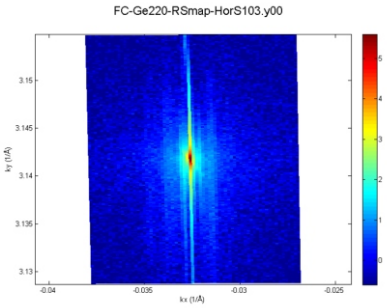

Figure 1c). Reciprocal space map of Ge(220) sample rotated in its plane by 90 , with horizontal tool traces.

inside channel-cut monochromators. The SPDT technique is based on precisely shaped single crystal diamond tool, extremely precise CNC controlled slides and air spindle in a nanomachining centre. It is commercially available and is used to produce free-form optics with the surface quality sufficient for infrared optics without any postpolishing. Our first experimental results of SPDT processing for X-ray flat optics were presented in [6]. In this communication it will be shown that SPDT with special tools can provide very precise flat walls inside CCMs. For channel widths around 2 mm there was a problem – rod shaped tool shank thinned down to 1.8 mm with diamond aside got into vibrations generating surface waviness at spindle speed of 2000 rpm. Fig. 1 shows "artificial" symmetrical Ge(220) CCM used for testing of technology and reciprocal space maps in directions perpendicular and parallel to toolpath traces (using 3 mm thick tool shank with diamond aside).

At this stage of technology development, slight chemical polishing was applied to remove tool traces visible in AFM, micro Raman and RSM with promising results. Experiments with very fine end polish etching, separate CMP using hand tools and, preferably, on-machine postpolishing, are in progress and the results obtained will be presented.

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PB29

HIGH-RESOLUTION MICRO- AND NANO-BEAM DIFFRACTION SYSTEM AT BL13XU OF THE SPRING-8

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Among of the X-ray diffraction techniques, a measurement of intensity distribution in a reciprocal space, which is so-called a reciprocal space map (RSM) measurement, is effective for characterizing strain status of an epitaxial layer because lattice tilt is separated from lattice spacing [1]. If we can measure the RSMs with using an X-ray micro- or nano-beam, it is expected to be a more powerful tool for characterizing the local strain distributions in detail.

To realize this prospect, we have developed high-resolution micro- and nano-beam diffraction system at SPring-8 undulator beamlines for more than 10 years before [2-4]. Since fiscal year 2005, the system was installed at BL13XU experimental hutch 3 (EH3), which was shared by ultra-high vacuum surface X-ray diffractometer, and was opened for public users. As the result of continued improvements, we recently realized rapid local RSM measurements using focused beam with the size of about 200 nm produced by phase zone plate [3,4]. On the contrary, drift of sample position due to temperature fluctuation in the experimental hutch became a serious problem for stable measurements.

We therefore constructed a new experimental hutch 4 (EH4) and then the micro- and nano-beam diffraction system was transferred from EH3. In EH4, the temperature was controlled within ± 0.05 °C for one day by using a precise air conditioner and thermal insulating walls of phenolicfoam, which were already used at nano-scale analyses beamlines BL37XU and BL39XU of the SPring-8 [5,6]. Furthermore, fluorescent lamp type LED lighting device was adopted for in-hutch lighting to decrease heat emission. Consequently, we became able to use nano-beam more stably. Minimum beam size at that moment was 110 (horizontal) × 160 (vertical) nm^2 for 8 keV X-rays.

The details of the the high-resolution micro- and nano-beam diffraction system and typical examples of the experimental results [7-10] will be presented.

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PB30

DANMAX - THE DANISH BEAMLINE FOR IN SITU MATERIALS STUDIES AT MAX IV

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DanMAX will be a world-leading materials science beamline dedicated to in situ and operando experiments on real materials. The beamline will be built at the MAX IV 3 GeV storage ring[1] and operate in the 15-35 keV range. The beamline will have two end stations: one for full field imaging and one for powder X-ray diffraction. With a large and diverse user community there will be a focus on high throughput, advanced sample environments and extended provision of data analysis tools.

The radiation source will be a 3 meter long in-vacuum undulator, ensuring a very bright and well-collimated beam. The beam size is adjusted using CRLs from approx. 3 m FWHM to 3 mm FWHM. Two different modes will available: high intensity/lower energy resolution (E/E ~ 10-2) or high energy resolution (E/E ~ 10-4) with lower

intensity. The beamline will strive to operate and continually develop a large range of advanced sample environments. Open standards will be available, both mechanical and software, for fast and easy integration of custom-built sample environments at the beamline. To enhance the user experience and the success of experiments at DanMAX, the user software will be tailored to give the users direct feedback, i.e. on-the-fly integration of 2D data and on-the-fly modelling DanMAX will start to accept early users in 2019.

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PB31

BIOMEDICAL IMAGING AT MAX IV

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The Max IV Laboratory was inaugurated on the 21.6. 2016 with the initial portfolio of 14 beamlines. These cover mainly spectroscopy and diffraction while full-field imaging capabilities will be added in the upcoming years. The establishment of coherent imaging techniques oriented towards bio-medical applications is motivated at Max IV by the consideration that the high coherent flux of this diffraction limited synchrotron source will facilitate the application of various phasing techniques. There is good hope that the radiation damage may be reduced under certain circumstances as more coherent photons contribute to the image formation.

The first beamline devoted to bio-medical and soft matter applications is in its design phase. The BioMedMAX beamline has its main design goal to perform longitudinal in vivo studies on small animals at the micrometer scale. It is currently under consideration to widen the scope towards deep sub-micrometer tomography and element sensitive imaging of biological tissue. The poster presents challenges and proposed solutions for a best use of the high coherent flux of Max IV in view of bio-medical imaging.

PB32

AUTOMATED, EFFICIENT AND HIGH-THROUGHPUT DATA ACQUISITION AT THE ESRF BIOSAXS BEAMLINE BM29

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Small Angle X-ray Scattering of macromolecules in solution (bioSAXS) continues to increase in popularity and is used by an ever more diverse research community. Dedicated beamlines such as BM29 at the ESRF [1] provide an optimised setup for high throughput data acquisition; have had a large impact on structural biology.

The bioSAXS Sample Changer [2] enables high throughput screening of samples for functional studies of how a macromolecule behaves under varying conditions. Online size exclusion chromatography (SEC) coupled with Small-angle X-ray Scattering [3] enable measurements from samples otherwise not amenable for SAXS though depending on the column used can come at expense of throughput. Automatic switching between SC and SEC is controlled via the data acquisition software allowing optimal use of available beamtime. All data acquired from both modes are processed automatically to yield standard invariants of radius of gyration, maximal particle distance, volume and molecular mass estimates as well as ab-initio models. Data and pipeline analysis results [4] are logged into the ISPyB database [5] and can be accessed from anywhere via the web based GUI.

High-throughput screening with the SC is not ideal in terms of sample consuming and optimal beam usage as more time is spent cleaning the exposure cell than for data acquisition. To make an efficient system with low sample consumption digital (droplet) microfluidics devices were introduced to the beamline. Droplets generated on the microfluidics chip with adjustable protein additive concentrations are exposed to X-rays in a fused silica capillary of 300 m diameter. The capillary is mounted into the standard sample holder pods and is connected directly to the exit of the microfluidics chip. The exposure and data acquisition are synchronized such that only droplets and not the carrier oil are exposed to avoid radiation damage of the oil and total reflection on droplet walls. The chip design alternates droplets with and without protein to enable monitoring of the background scattering. This could be an effective

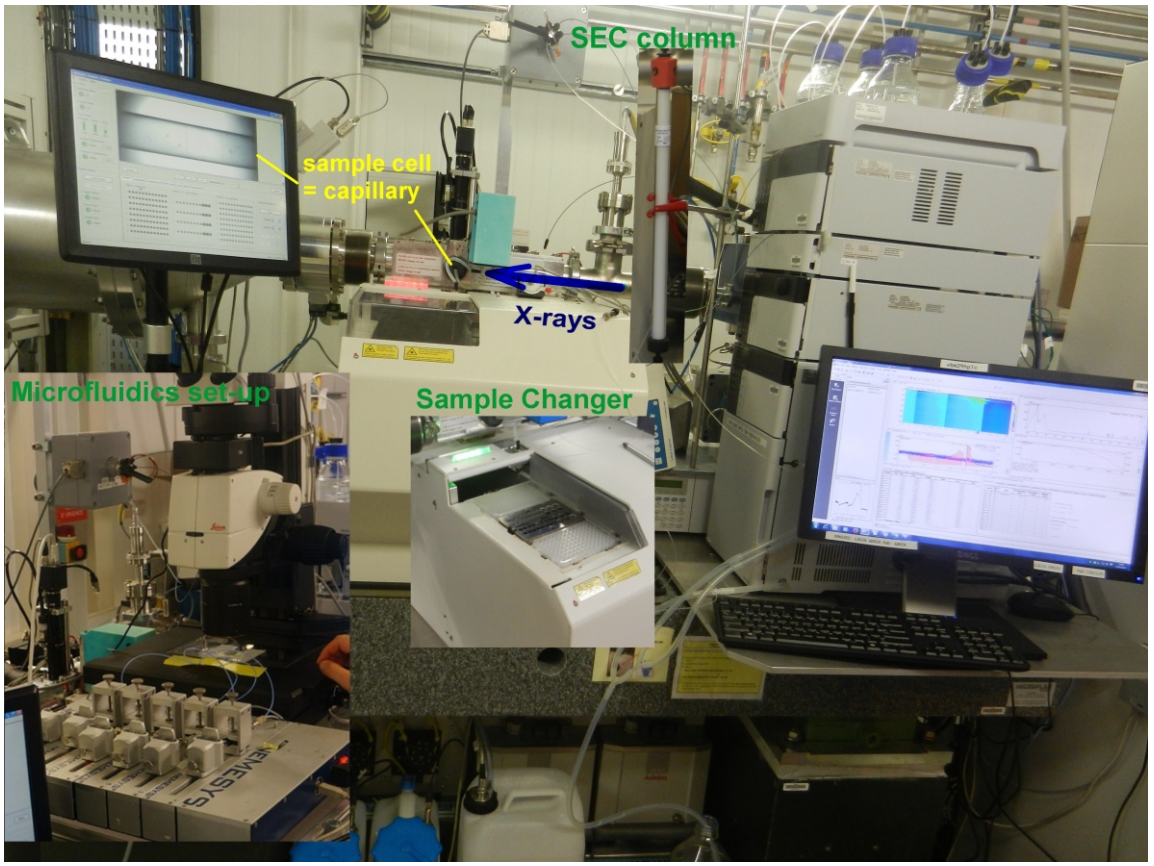

Figure 1. Sample Changer, Size Exclusion Chromatography and microfluidics set-up at BM29 beamline.

delivery mechanism for not only serial SAXS but also serial crystallography. The variable sample illumination available recently on the beamline opens a new stage of the scientific case for photosensitive samples.

The ESRF bio-SAXS beamline BM29 set-up characteristics (Fig.1) together with the examples of user data obtained will be given.

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PB33

THE TOMCAT HARD X-RAY FULL FIELD TXM

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The TOMCAT Full Field Transmission X-ray Microscope (TXM) is working from 8 to 20 keV. It is composed of a custom designed condenser [1] producing a top-flat illumination in the focal plane [2], a series of Fresnel Zone Plates (FZP) objectives, with different diameters on the same frame (to be selected according to the working energy), which will focus the transmitted X-rays from the sample onto a CCD. Thanks to the improvements brought by Zernike [3] for optical application, phase contrast can be achieved by placing a phase shifter at the back-focal plane of the FZP. In our case, a series of Zernike phase shifter rings (PR) have been produced on the same frame to match the working energy.

The recent improvement of the design of our instrument comprises a reduction of the typical artifacts in Zernike phase imaging [4], the possibility to easily and quickly change the X-ray energy for the full-field microscope in the energy range of 8-20 keV and a better stability of the FZP/PR support. This instrument has been built in such a way that the sample position remains fixed: for a working energy, the condenser is placed at the appropriate focusing distance and the diameter of the FZP is chosen accordingly. In this way, the magnification is also fixed, resulting in a constant pixel size of 80 nm for all energies. With our XSight^{TM} uRapid camera, a tomography acquisition time for about 1000 projections is now comparable to a standard tomography acquisition at TOMCAT: about 5 min with our multilayer monochromator (energy bandwidth of 10^{-2}) and about 30 min with the Si(111) double crystal monochromator (energy bandwidth of 10^{-4}).

Our Full Field TXM setup reaches a spatial resolution down to 150 nm both in absorption and phase contrast mode. Having the capabilities to work at such high energy range opens a wide range of applications from biology application to materials science.

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PB34

SIMULATION OF RESOLUTION EFFECTS IN HRXRD BY SEMI-ANALYTICAL RAY-TRACING

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Modelling of resolution effects is important both for the analysis of measured X-ray diffraction data and for the planning of new experiments. In this contribution we present a generalization of our recently reported approach to ab-initio simulation of instrumental function [1]. The aim of the designed semi-analytical ray-tracing approach is to combine both universality of numerical methods [2] and performance of analytical ones [3]. The method reported in Ref. [1] is based on the assumptions of factorized transmission function of optical elements and of rectangular shape of its spatial part. These assumptions are not valid for such elements, as Göbel mirrors, focusing monochromators, collimators with round-shape apertures. In the paper [1], only an approximate description of such elements has been proposed.

The proposed generalization of the method is free of these limitations and provides a consistent description of optical elements with correlated spatial, angular and spectral parts of the transmission function. An improved estimation of beam shape enables preserving high performance of the method without the assumption of rectangular spatial part of the transmission function of elements. On the basis of the developed approach, the problem of efficient simulation of the detected signal for a given sample response and diffractometer configuration is analysed. We show that the effect of finite resolution can differ significantly from its best approximation by a convolution of the sample response with certain instrumental function profile and provide an approach for more accurate, but still fast calculation of the detected signal. This

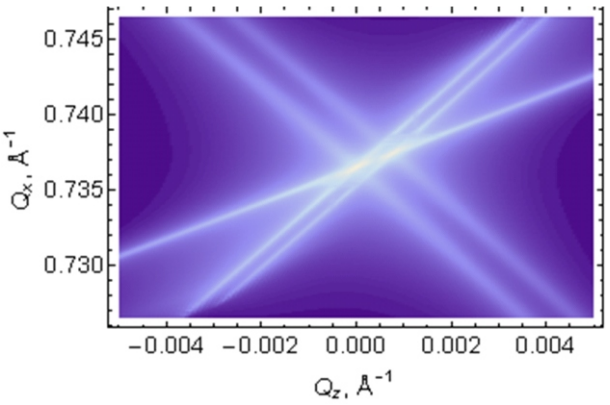

Figure 1. An example of reciprocal space map of instrumental function, simulated by the developed method (diffractometer configuration with 0.2 mm wide incident slit and Ge(220)x1 analyser; Bragg reflection 004 is considered).

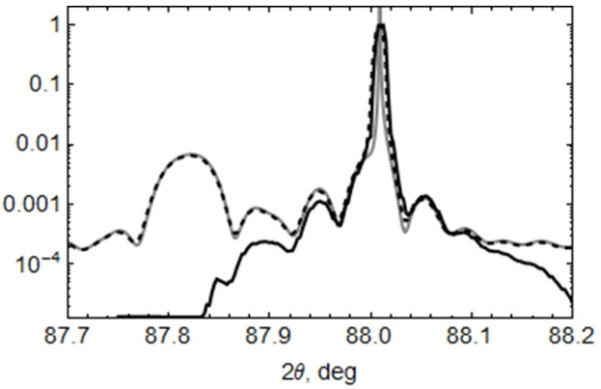

Figure 2. Comparison of simulated 2 scans for 224 reflection of a sample with Si and $Si_{0.86}Ge_{0.14}$ layers on Si substrate. Solid grey line – simulation without resolution effects; dashed line – convolution-based simulation; solid black line – accurate simulation for diffractometer with Ge(220)x4 monochromator, 0.2 mm incident slit and 1 deg receiving parallel slits.

effect, illustrated by Fig. 2, is crucial for correct planning of experiments.

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PB35

COMPLEMENTARY DATA FROM PHOTOEMISSION IN HARD X-RAY REFLECTION EXPERIMENTS

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Electric self-detection of X-ray-induced photoemission from an object can be considered as non-invasive monitoring of the radiation flux because optimization of detection of the generated electric carriers is focused on creating efficient charge collection in the exterior of the object [1]. The measured electric current creates opportunities for monitoring interaction of X-rays with matter including not only measurements of the incident X-ray flux, but also gaining insights on the reflected radiation flux and the structure of the material exposed to X-rays. The photoemission yield is defined by the photon-electron attenuation properties of the material and is modulated by the reflection/transmission properties, which may strongly depend on the characteristics of the incident radiation such as the angle of incidence and the photon energy. This dependence is most prominent in the resonant conditions such as x-ray diffraction in crystals, multilayers and total external reflection from smooth surfaces [1,2]. Angular-dependent electric response of an x-ray mirror enclosed in a gas flow ionization chamber can be used for diagnostics of the X-ray optics, while the same approach can provide easy access to complementary X-ray transmissivity data in X-ray reflectivity experiments [1]. Furthermore, using hard X-rays the approach can be applicable for non-destructive evaluation of surface structures with deeply buried layers [3]. In this presentation practical information about the sample, which can be gained from the self-detected photoemission yield in X-ray reflection experiments will be discussed along with relevant advantages and limitations.

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PB36

DTXRD - COLLECTION OF SOFTWARE TOOLS FOR EVALUATION OF SINGLE CRYSTALS USING X-RAY DIFFRACTION

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A specialized software package DTXRD includes several utilities for evaluation of single crystals using methods of X-ray diffraction. The basic utility "dtxrd" in the package is based on the dynamical theory of X-ray diffraction for a monochromatic wave in the 2-beam approximation. The utility provides calculations of reflectivity/transmissivity for temperature-dependent models of several single crystals commonly used for X-ray optics and in the semiconductor industry. A more advanced code "throughput" is included in the package to perform such calculations in a multi-crystal geometry using basic models for the X-ray source with variable divergence and a user defined spectrum (e.g., [1]). Another code "rctopo" provides calculations of X-ray rocking curve topographs based on a sequence of X-ray diffraction images collected at different angles on the rocking curve of a single crystal under examination (also known as rocking curve imaging [2]). The use of this code to process sequential X-ray topography data in the pseudo plane wave geometry was found particularly useful for detailed characterization of X-ray crystal optics (e.g., [3]). Additional scripts are available to assist with data handling and evaluation. The code is written in python and is available under the Open Source licence [4]. The software at present has command-line interface with graphical output based on the python-matplotlib library. The detailed software documentation is available online [5].

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PB37

COHERENT DIFFRACTION BASED IMAGING AT THE DIAMOND LIGHT SOURCE BY UTILISING I13'S UNIQUE COHERENCE PROPERTIES

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I13 is a 250 m long hard X-ray beamline (6 keV to 35 keV) at the Diamond Light Source [1]. The bealine comprises two fully independent and simultaneously operating experimental branches: one for imaging in direct space currently providing high resolution in-line phase-contrast tomography and one for imaging in reciprocal space using coherent diffraction based techniques like Ptychography, Bragg Coherent Diffraction Imaging (Bragg-CDI) and Photon-Correlation Spectroscopy (XPCS). In addition, the design of the latter branch provides an outstandingly large lateral coherence length beyond 200 m [2], lending itself to unique research in the field of coherent xX-ray optics, the development of novel instrumentation and imaging techniques in relation to interferometry, holography and speckle tracking.

Techniques currently provided at I13 on a regular basis are experiments in the forward scattering direction, like Ptychography (Figure 1) and Bragg-CDI (Figure 2) in reflection geometry. Ptychography lends itself to the study of specimens, which have hardly any absorption-contrast over a large field of view of up to ~100 um with a resolution of a few 10 nm. On the other hand Bragg-CDI permits the investigation of strain and phase-transitions in single crystals with a size of up to 10 m, due to temperature changes or irradiation with VIS laser light.

In this paper we will detail the current prospects and recent progress concerning these two key experimental techniques and complement them with examples of more specialised optics experiment exploiting the large coherence length.

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We acknowledge the support of Oscar Branson (Cambridge), who provided the calcified Plankton shell.

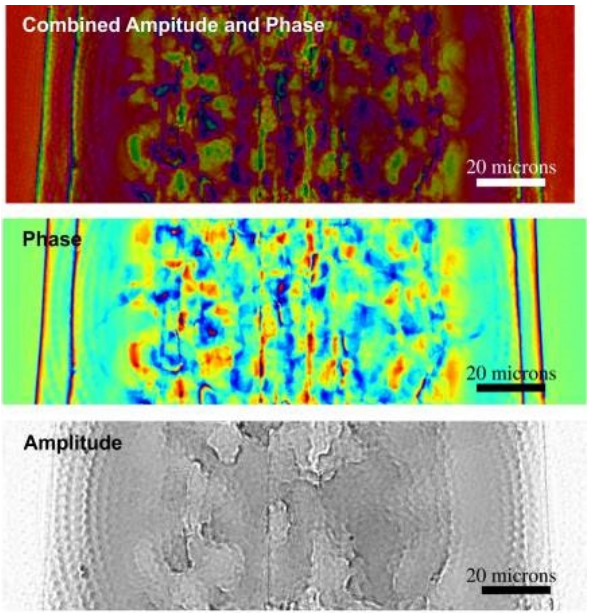

Figure 1. Ptychogram of a calcified Plankton shell.

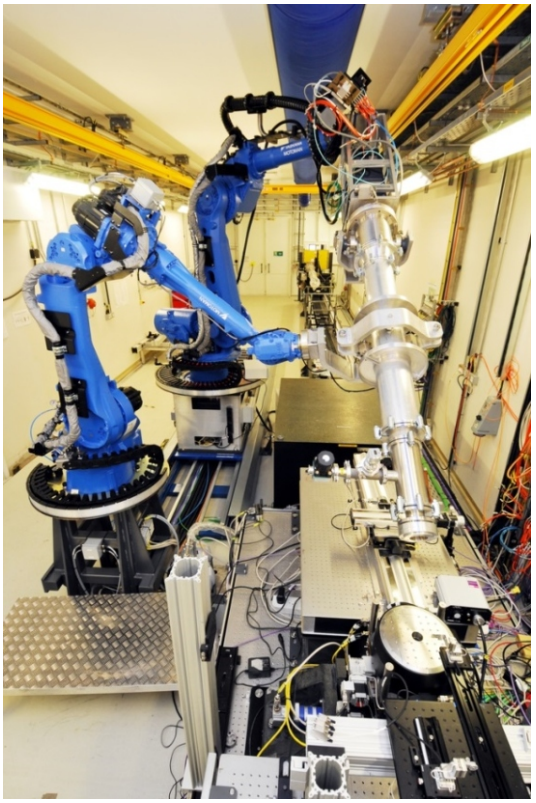

Figure 2. Robot Diffractometer.

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DIAMOND MANCHESTER BEAMLINE (I13-IMAGING) AT DIAMOND LIGHT SOURCE (DLS) – NEW DEVELOPMENTS

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The Diamond-Manchester Imaging branchline of the I13 beamline routinely performs real space imaging and tomography with micron resolution. The Experimental Hutch is about 250 m from the synchrotron source: the large distance provides the X-ray beam with intrinsic high spatial coherence. The branchline is capable of handling both pink and monochromatic beams in the energy range 8-30 keV. The pink beam flux allows fast imaging for the investigation of rapid dynamic processes. There are two monochromators available in the beamline: a Double Crystal Monochromator (DCM) and a recently installed Multi-Layer Monochromator (MLM). The latter is currently under commissioning. The MLM provides a flux about 20 times higher than the DCM. The monochromators are suited for element specific imaging and probing the chemical environment.

In the beamline microtomography is routinely performed and the development of Transmission X-ray Microscopy (TXM), which is in the commissioning phase, will allow for nanometer scale imaging.

Here we present the preliminary results for TXM with MLM X-ray beam. The TXM in Zernike phase contrast imaging mode with spatial resolution in about 100 nm range is shown. The instrument provides a large field of view and working distance. The latter is important for the implementation of customised sample environments. The full installation of MLM will help to extend further the capabilities of I13-branchline.

PB39

HIGH PRECISION X-RAY MULTILAYER MIRRORS FOR CUSTOMIZED SOLUTIONS

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A variety of X-ray analysis methods is available for laboratory applications. In most cases, the X-ray beam needs to be tailored with suitable X-ray optics to enhance the performance and to allow for suitable working distances. X-ray multilayer mirrors are often first choice for the application at typical laboratory X-ray sources since a beam with a requested dimension and divergence can be provided and a sufficient monochromatization is achieved. We show a typical workflow of the optimization of customized X-ray multilayer mirrors and examples for specific applications.

Several aspects have to be considered regarding the design of the entire X-ray system. This includes the choice of an appropriate X-ray source (e.g. type and size of the X-ray focus, anode material) and X-ray optics type (e.g. one- or two dimensional operation, focusing or collimating behaviour, monochromaticity/ bandwidth). These conditions define the general shape of the X-ray mirror that is coated with a high precision multilayer stack of alternating materials to permit Bragg diffraction. Different multilayer systems are available to achieve a narrow-band or broad-band behaviour, or to tune the reflectance. These specular properties can be predicted by respective simulations. The influence of the finite size of the X-ray source, imperfections of the curvature of the X-ray mirror, or effects due to non-negligible energy difference between the characteristic $K_{\alpha 1}$ and $K_{\alpha 2}$ photon energy at higher photon energies can be studied with raytracing simulations of the entire optical path, taking the specular behaviour into account. Thus, a complementary optimization of the geometry of the X-ray mirror, the choice of appropriate substrate parameters, and the properties of the multilayer is possible.

Examples are shown for the fields of high-resolution X-ray imaging and X-ray diffraction.

Two-dimensionally focusing multilayer X-ray optics are suitable condenser optics for full-field X-ray microscopy with multilayer Laue lenses [1]. This configuration is characterized by a better matching of the numerical aperture of condenser and MLL, and of the size of the illuminated region to the field of view of the X-ray lens. Direct beams, bremsstrahlung and K_{β} radiation are efficiently suppressed to improve imaging conditions. In addition, this configuration enables the application of photon energies $E > 10$ keV for full-field X-ray microscopy at high spatial resolution using laboratory X-ray sources.

In case of X-ray diffraction, some aspects become more important, if high photon energies, large working distances, or small X-ray sources are present. A comprehensive example is the design of a focusing X-ray optics for Ag-K_{α} radiation. The splitting of both $K_{\alpha 1}$ and $K_{\alpha 2}$ lines ($E = 0.17$ keV) as well as the lateral extent of a typical microfocus X-ray source have to be considered to provide a homogeneous illumination.

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PB40

STRUCTURAL STUDIES OF THE GROWTH AND ORIENTATION OF M- AND Y-TYPE FERRITES PREPARED BY CHEMICAL SOLUTION DEPOSITION METHOD

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Oriented M-type hexagonal ferrite (magnetoplumbite structure) thin films were prepared by chemical solution deposition on $SrTiO_3$ (111) (STO) substrates and used as templates (seed layers for oriented Y-type $Ba_2Zn_2Fe_{12}O_{22}$ thin films. Several M phases with different chemical composition, magnetic character and mainly lattice misfits in the range of -0.8 % to -7 % were investigated by X-ray diffraction (XRD), electron backscatter diffraction (EBSD)

and atomic force microscopy [1]. XRD analysis was performed in both conventional Bragg-Brentano symmetric setup as well as in parallel beam with the Eulerian cradle. In order to find appropriate reference value of the lattice parameters of different used M-phases, some data analysis of PDF-4 database had to be performed with respect to the statistics and/or also to the stoichiometry. Structural studies were focused on the investigation of preferred orientation by symmetric and asymmetric XRD scans and by EBSD and also on the analysis of possible residual stresses.

Seeding M-layers and corresponding Y-films showed always strong out-of-plane (000*l*) orientation with some differences in its degree. More significant differences were found in the observed in-plane orientation. This largely depended on misfits between the M-interlayer and substrate and also M-interlayer and Y-film. The best in-plane orientation of top Y-layer was obtained if the misfit values between the seed layer and substrate, and between the seed layer and Y-layer are similar and also if the surface of seed layer is formed by hexagonally shaped grains. This corresponds to the following chemical compositions of seed layers: $(BaSr)(GaAl)_{12}O_{19}$, $Ba(FeAl)_{12}O_{19}$ and $SrGa_{12}O_{19}$. Then single domain perfect hexagon-on-hexagon orientation was observed for M-film while for the Y-layer the in-plane orientation $(001)_Y \parallel (111)_{STO}$ and $[100]_Y \parallel$ [2-1-1]STO shows double number of maxima in -scans than it would correspond to the plane multiplicity. This indicates in-plane obverse/reverse twinning of the Y-films. Examples of these scans are shown on Fig. 1 for one of the best thin film architecture with $(BaSr)(GaAl)_{12}O_{19}$ interlayer and on Fig. 2 showing multiple domains for $(BaSr)Fe_{12}O_{19}$ interlayer.

This work was supported by the Grant Agency of the Czech Republic under Grant No. 14-18392S.

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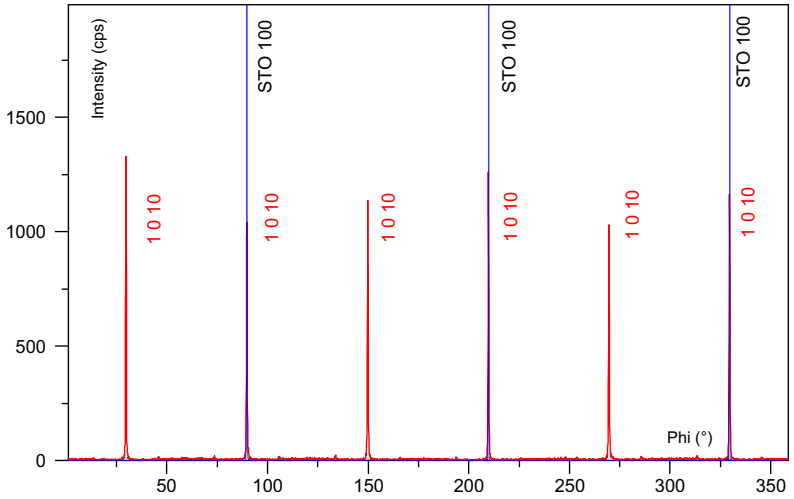

Figure 1. -scans on 1 0 10 reflection of Y phase on STO substrate and M $(BaSr)(GaAl)_{12}O_{19}$ seed layer.

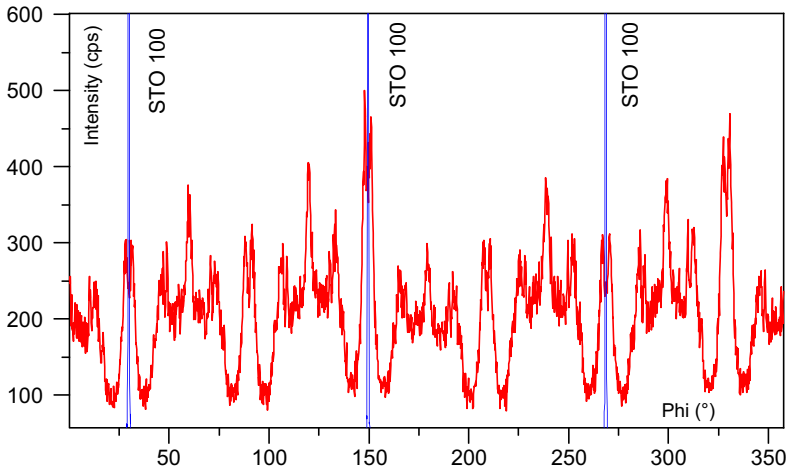

Figure 2. -scans on 1 0 10 reflection of Y phase on STO substrate and M $(BaSr)Fe_{12}O_{19}$ seed layer.