

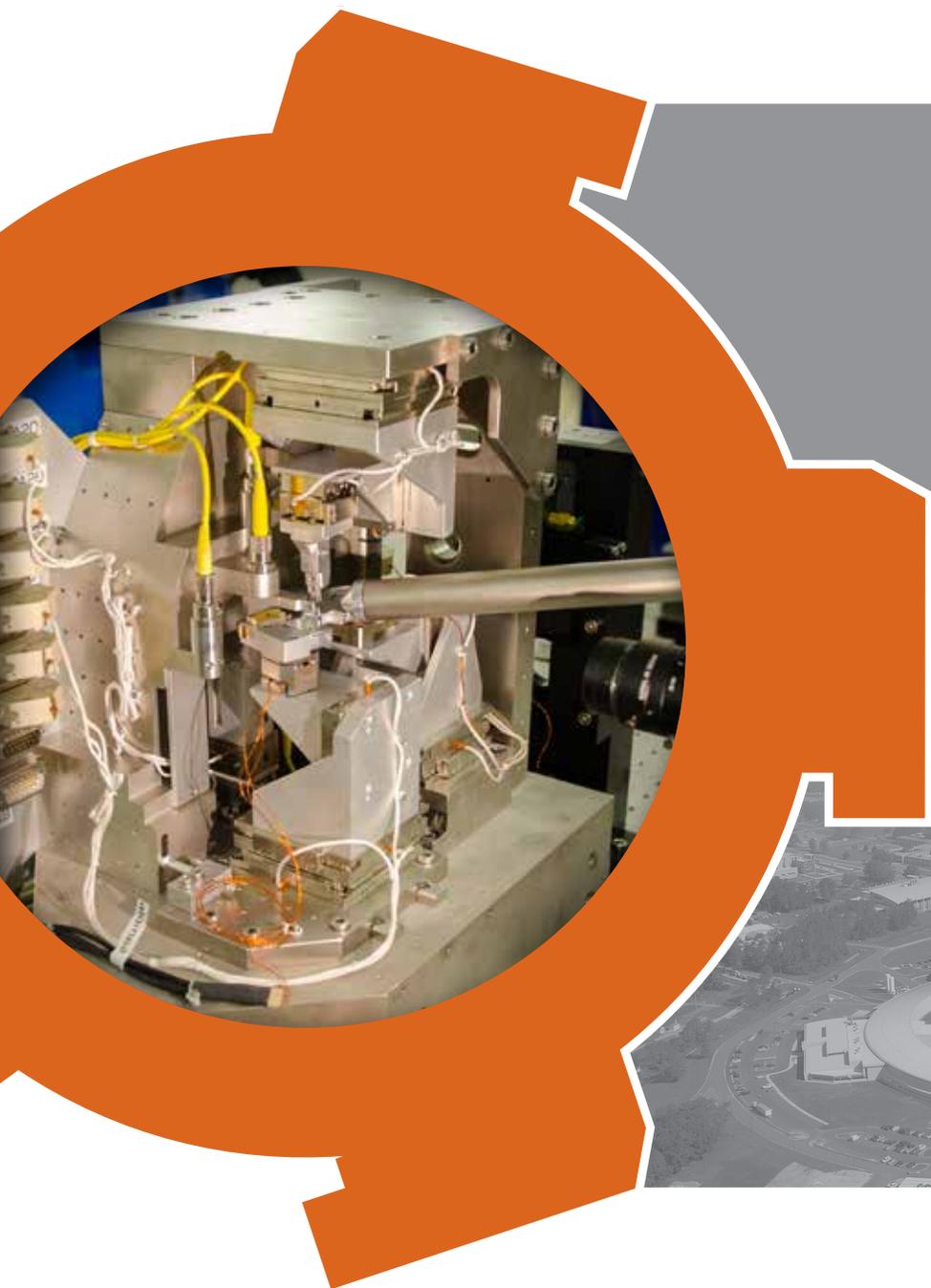
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Poster
Abstracts



Transition Metal Distribution in the Brain and Spinal Cord of a Rat Model of Myelin Loss

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Introduction and Objectives

Transition metal concentrations in the central nervous system (CNS) are implicated in neurodegenerative diseases such as Alzheimer's, Parkinson's and Multiple Sclerosis. A common symptom of these diseases is demyelination, which is the degradation of the myelin sheath that encapsulates the neurons in vertebrates. A dysmyelinating rodent model (Long Evans Shaker (LES) rodents) was used to characterize the transition metal concentrations in the central nervous system compared to its age-matched controls, to elucidate the contingency between transition metals and myelination in the pathogenesis of neurodegenerative diseases. The concentrations of manganese (Mn), iron (Fe), copper (Cu), and zinc (Zn) in regions of grey matter and white matter have been compared between Shaker rodents and their age-matched Long Evans (LE) controls in the cerebellum and spinal cord, using micro probe Synchrotron Radiation X-ray Fluorescence (μ SRXRF).

Results and Discussion

In the cerebellum, the concentration of all elements were significantly increased in the white matter of the Shaker model, and decreased in the gray matter of the Shaker model in comparison to their age and region matched controls. In the spinal cord samples, concentrations of all metals were higher in white matter and grey matter of Shaker rat spinal cord compared to those in the control rat spinal cord. This study demonstrated that μ SRXRF sensitivity is sufficient to discriminate between the elemental distributions of gray and white of the brain sections and spinal cords in the two groups. The observed significant increase of Mn, Fe, Zn and Cu in the white matter of the Shaker animals in the cerebellum and spinal cord compared to controls could be the result of astrocytic glial cells replacing the myelin in the CNS [1]. Unlike other imaging techniques, the fine resolution of μ SRXRF enables specific regions of gray matter structures namely, the molecular layer and the granule layer to be identified in the rat CNS, and their transition metal concentrations to be quantified.

Conclusions

This work will further establish μ SRXRF as a powerful analytic technique for compositional studies in brain sections from models of brain disease. If further μ SRXRF studies could be carried out, a final atlas could be created to represent the expected levels of transition metals in central nervous system of healthy animals, which is currently missing in literature.

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Micro-X-Ray Fluorescence Spectroscopy Mapping on Electrodes for Li-ion Batteries

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Introduction and Objectives

Capacity fade caused in part by leaching of transition metals is one of the major challenges for promising high energy battery materials such as the high voltage $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$ (LNMO) spinel. The leaching process produces chemical and morphological inhomogeneities and such heterogeneities were identified as major contributions to capacity fade and aging of the battery. Fast scanning micro-X-ray fluorescence spectroscopy (Micro-XRF) at medium spatial resolution (500 nm) and over millimeter ranges utilizing a Maia fluorescence detector was used to characterize the effects of cycling rate and state of charge on the elemental distribution (Ni, Mn) for $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$ /carbon composite electrodes in LNMO/Li [1]. Charge distribution is imaged by mapping the Ni-oxidation state by acquisition of a stack of elemental maps at multiple energies in the vicinity of the Ni K-edge (XANES mapping). The large solid angle provided by a 384 detector element array in combination with streamlined data handling and processing yields exposure times in the millisecond range and thus makes such detailed investigations feasible.

Results and Discussion

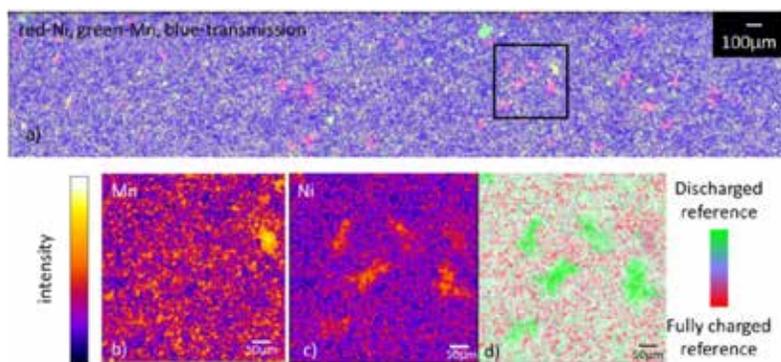


Figure 1a) shows the inhomogeneous distribution of Ni and Mn over a large area of the LNMO composite electrode cycled to an intermediate voltage. More detailed maps of Mn, Ni and charge distribution of the marked region in a) is shown in b), c) and d). The Ni distribution map shows areas with higher Ni concentration, so called “hot-spots” which show less

participation in the oxidation reaction upon charge, hence a better fit to the XANES standard obtained in the discharged state. The origin of the inhomogeneous distribution, possibly caused by side reactions with the electrolyte or structural changes, has yet to be understood better, but these regions are unlikely to participate in the charge/discharge cycle reducing the capacity of the battery cell.

Conclusions

While these measurements give a first overview and characterize the ongoing processes of degradation, we envision further studies *in operando*. This will allow us to follow the mechanism causing the inhomogeneities and characterize it in more detail. In addition, micro-XRF in 2 and 3 dimensions is a valuable tool to characterize the leached transition metals, which are partially deposited on the graphitic anode and thereby further reducing the capacity of the battery.

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An Energy Tunable X-ray Zernike Full Field Nanoscope up to 20 keV

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Introduction and Objectives

To improve the contrast between structures that have very similar absorption, phase-contrast imaging approaches have been developed. Based on the rings system used by Zernike [1], the Tomcat nanoscope is composed of: a custom designed beamshaper [2] producing a top-flat illumination in the focal plane [3], a Fresnel Zone Plate (FZP) acting as an objective lens and a Zernike phase shifter placed at the back-focal plane of the FZP. Zernike Phase Contrast (ZPC) is used mainly in biological applications at low energy range (below 8 keV). Nevertheless having the capabilities to use this tool at higher energy would open new applications in material science. In this presentation, we will show that, after solving some challenging technical problems, the applications in hard X-rays can provide interesting results in this field.

Results and Discussion

The new design of our instrument comprises a reduction of the typical artifacts in Zernike phase imaging [4] and the possibility to easily and quickly change the X-ray Energy for the full-field microscope in the energy range of 8-20 keV [5]. This opens the possibility to fine tune the acquisition energy according to the sample and also to distinguish different materials by K-edge imaging. The setup is built in such a way that the sample position remains fixed and the condenser is placed at the appropriate focusing distance for the working energy. The magnification is also fixed, i.e. a constant pixel size of 50nm, thanks to the design of a set of FZPs with different diameters for the used photon energy range. In the particular case of Nb₃Sn filament, used in superconductive wires, we will show that high energy ZPC provides 3D nanostructure details not accessible before and give complementary information to absorption imaging.

Conclusions

Our Full Field ZPC setup is working from 8 to 20 keV with a spatial resolution down to 150 nm both in absorption and phase contrast mode. This opens a wide range of application in materials sciences, as it will be shown in different applications during this talk.

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Ray Tracing and Wave Propagation with xrt

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Xrt is an open source framework for ray tracing in x-ray regime [1]. We provide basic classes for realistic synchrotron sources and various optical elements (mirrors, crystals, gratings, zone plates, compound refractive lenses etc.) and the engine for accurate calculation of the x-ray beam propagation. Due to high efficiency of the code and no limitation on the number of rays, in a reasonable time frame (seconds to minutes) the user can obtain both high resolution beam profiles and reliable estimation for the total intensity/power/flux values. Color coding can be used to demonstrate spectral characteristics of the beam on the same figures simultaneously with intensity distribution.

In the most recent version we present the full wave propagation mode that accounts for the diffraction effects on any optical element via the Kirchhoff integral calculation. We introduced the dedicated single electron wave generation mode to address partially coherent beam propagation. As the transition from rays to wave formalism leads to exponential growth of computational complexity, most critical calculations can now be delegated to the GPU via CUDA or OpenCL.

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GI-XRF/XRR and TXRF-XANES Analytical Applications of a Multi-purpose IAEA X-ray Spectrometry Facility at Elettra Sincrotrone Trieste

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Introduction and Objectives

The International Atomic Energy Agency (IAEA) has recently commissioned jointly with the Elettra Sincrotrone Trieste a multi-purpose X-ray spectrometry facility as the endstation of XRF bending magnet beamline. Exciting energies from about 3.8 - 14 keV are currently available using a Si(111) double crystal monochromator with a resolving power of $1.5 \cdot 10^4$, whereas certain upgrades are foreseen for the near future [1].

The endstation facility was developed to operate primarily under ultra-high vacuum (UHV) conditions, but non-UHV compatible samples can be also accommodated. The main instrument of this facility is a motorized 7-axis manipulator allowing 3 linear translations (x/y/z) and 2 rotational (theta/phi) degrees of freedom for the sample alignment. A coupled theta-2theta goniometer offers the option for performing simultaneously GI-XRF and XRR scans using several photo-diodes mounted on a separate linear stage. An ultra thin window SDD detector is employed for X-ray fluorescence experiments, whereas an intuitive GUI based on LabView software allows controlling all UHV chamber actuators/detectors. The endstation is based on a prototype designed by the Physikalisch-Technische Bundesanstalt (PTB), Braunschweig and the Technical University of Berlin, Germany (TUB) [2], whereas its technical specifications were evaluated by the PTB [3].

Results and Discussion

First results from the application of GI-XRF/XRR and TXRF-XANES analytical methodologies for the characterization of various nano-structured materials and environmental samples will be presented and discussed to highlight the analytical merits and capabilities of this new X-ray spectrometry facility that is available for external users [4].

Conclusions

Research projects from the IAEA member states are very welcome.

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The Most Numerous and one of the Tiniest Neurons in a Human Brain Investigated by X-ray Tomography

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Introduction and Objectives

Within this examination we visualized granule cells from a human brain. Granule cells - predominating in the granular layer of cerebellar cortex and rendering its characteristic microscopic picture, possess one of the smallest somas of all neurons (ca. 4 μm) make them most numerable population of neurons in the whole brain, estimated even to one half of all brain neurons. A small piece of cerebellum was scanned with a full-field X-ray tomography in a cryogenic-vacuum environment, a new development available for users at PETRA III P06 beamline at DESY. The scanning, emission X-ray fluorescence tomography was employed to find any inhomogeneities and fine structures in the element composition at a sub-tissue level.

Results and Discussion

An X-ray attenuation contrast below 12% in tomograms, recorded at a small radiation dose of ca. 10^3 Gy, with a spatial resolution of 1.1 μm , was sufficient enough to distinguish between different sub-structures where mass density varies relatively less than 10%. Cell nuclei from granule neurons are clearly visible in tomography slices (Fig. 1) as well as in the 3D visualization of cerebellar cortex (Fig. 2).

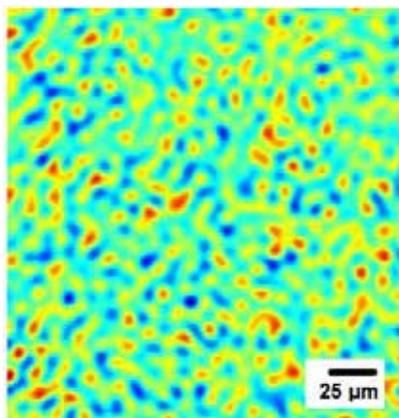


Fig. 1. Cell nuclei (red) from granule neurons. Void regions (dark blue) are due to autolysis.

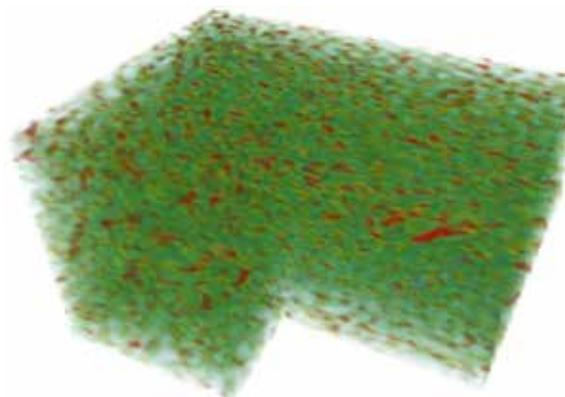


Fig. 2. Granular layer in a 3D vision. Cell nuclei (red) are apparent in cytoplasm (green).

Conclusions

The experiment proves that cell nuclei are slightly more dense than the surrounding cell plasma. Autolysis in the granular layer was confirmed. This experiment opens up the way for any future investigation to clarify e.g. the reason of brain death in forensic pathology. We have also proven that the new cryogenic-vacuum environment installed at PETRA III P06 beamline at DESY is a reliable instrument for bio-imaging with hard X-rays.

Automated Adjustment of Confocal X-ray Microscope

F.A. Darin, D.S. Sorokoletov, Ya.V. Rakshun

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They at the SR XFA station on VEPP-3 have developed an X-ray confocal microscope (XCM) with pair of polycapillary lenses used as X-ray optical elements. These lenses are very sensitive to adjustment of the angles of beam incidence. Since the station is intended for a variety of tasks besides the XCM, the position of the front lens may vary uncontrollably. Therefore, prior to experiments of elemental mapping it is necessary to adjust the X-ray unit.

We have developed a two-coordinate unified procedure of periodic adjustment of the polycapillary lenses by fluorescence signal. The automated adjustment of the first lens is performed based on two angle coordinates relative to the SR beam; the second lens is adjusted by two linear coordinates in a plane perpendicular to the cross section of the lens. The adjustment process takes less than one hour. The optimality of the angle settings can be determined from the signal maximum; the position adjustment can be assessed from the shape of the curve of the instrument function.

Some problems under study at the experimental station require disalignment of the XCM for a larger confocal volume. With a non-optimal setting of beam position relative to the axis of the front lens, the instrument function is asymmetric. This dependence is well approximated with expression [1]:

$$Sig(x, y, E) = \frac{A}{w\sqrt{2\pi}} e^{-\frac{y^2}{2w^2}},$$

where $A=A(E)$ is the maximum recorded signal value; $w=w(x,y)\equiv\{w_1(x): y<0; w_2(x): y\geq 0\}$ is the waist width parameter; x is the coordinate of the wire along the incident radiation beam relative to the focus of the lens; y is the transverse coordinate of the wire.

The asymmetry of the first lens is defined by the linear displacement of its axis with respect to the SR beam axis. Performing one-dimensional scanning with the wire at first in the horizontal plane of the beam cross section and then in the vertical plane, we obtain the profile of the distribution of asymmetric Gaussian beam. Using the FWHM values of the approximated dependencies, one can obtain correction factors for lens alignment. Using this procedure, one can adjust the lens in five coordinates for problems of X-ray confocal microscopy with disalignment.

This work was supported by RFBR grant number 14-02-00631.

1. D.S. Sorokoletov, Ya.V. Rakshun, F.A. Darin, Hardware features of polycapillary lens and confocal X-ray microscope in restructuring of its confocal volume (in Russian). / *Avtometriya* 2015 V51 No.3.

New SVD Algorithm for Deconvolution Problems in Micro XFA with Large Widths of Instrument Function of Confocal Scheme

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X-ray fluorescence analysis on SR beams with polycapillary lenses (micro XFA) enables elemental analysis in volumes of linear dimension within the focal spot diameter. The use of two lenses in the confocal scheme confines the area under study to a "pixel" of about 15x15x15 microns [1].

In some micro XFA problems (investigation into biological cells, dust mites, layered samples etc.) it is desirable to attain to a spatial resolution of a few microns, still using polycapillary optics with their advantages, including the high radiation transmission ratio and compactness. The relevant methods are based on the solution to a three-dimensional deconvolution equation, which is reducible to a block system of linear equations [2]. There are a lot of computational methods for solving such inverse problems, and good results can be achieved by a number of ways. In the widespread Tikhonov's method of regularization, the use of the singular (SVD) or spectral decomposition of the resulting block matrix can significantly reduce the computation time [2], and the solution may depend on the chosen decomposition basis (depending on the source of the initial signal and noise characteristics). Comparison of the results of the method with different bases of decomposition may be of interest.

The SVD and SVDs procedures of the Matlab program do not work properly when applied to block matrixes that correspond to an instrument function with large widths ($\text{FWHM} > 6$). In particular, they overstate the singular values. We have attempted to identify the source of the error and then to develop an algorithm that would be free of the above drawbacks. We relied on the SVD function of the Jama package [3] adapted for the programming language C++, this function giving similar results. In the course of the study of this algorithm in the environment Visual C++, we identified the problem fragments that gave error because of incorrect rounding-off, which in turn was caused by underflow. Moreover, the error accumulated fast because of the nature of the code. These fragments were fixed via introduction of big-width numbers using the package BigNumber [4], which we adapted to the case of real numbers.

The new SVD algorithm is at the stage of debugging yet. It is expected that the application of the algorithm in the Tikhonov regularization method to model problems with very low noise (below 10⁻³%) will increase the resolution up to three times (which corresponds to a minimum detectable object of 3 microns). This will enable better results of deconvolution at actual noise levels of about 1% (assuming that the noise is close to white).

This work was supported by RFBR grant No.14-02-00631.

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The Color X-ray Camera - A pnCCD Based Benchtop System for XRF, TXRF and XRD

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Introduction and Objectives

Laboratory scale or “Benchtop” X-ray analysis systems are used in many laboratories for materials characterization. Such instruments tend to be specialized for one specific measurement type, whether XRF based imaging, total reflection X-ray spectrometry (TXRF) or X-ray diffraction (XRD). In this work, we will present the Color X-ray Camera (CXC) as a flexible benchtop instrument, based on a pnCCD full field imaging X-ray spectrometer, which is capable of performing all three experimental methods.

Results and Discussion

pnCCDs have been used as imaging X-ray spectrometers installed on X-ray satellites and at synchrotron sources for a number of years [1]. These energy dispersive, high-spatial resolution area detectors record both the position and energy of an X-ray event [2]. With readout rates of 1000 Hz and maximum count rates over 200 000 counts per second, these imagers are a flexible tool for a variety of X-ray measurements [3]. Because the position and energy are recorded, experiments requiring spatial

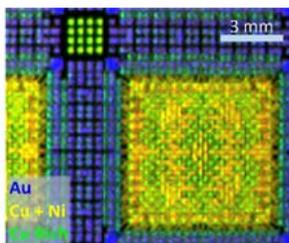


Figure 1: False color image of electronic circuit board created using the CXC with polycapillary optics.

resolution, such as XRF imaging and XRD can be performed. The intrinsic position resolution due to the pixel structure is 48 μm , and can further improved by subpixel calculations below 10 μm . With an imaging area of approximately 1.2 cm x 1.2 cm, efficient, high solid angle X-ray spectrometry can also be performed. Using the standard model CXC, a series of experiments have been done to characterize the performance of the pnCCD for use in XRF (see Figure 1), TXRF and XRD, and this work will present the opportunities and advantages of the pnCCD as a flexible full field imaging spectrometer.

Conclusions

Formerly, several different systems were necessary to examine a sample with different X-ray methods. With the Color X-ray Camera, a variety of different X-ray detection methods like XRF, TXRF and XRD can be performed using only one detector with high read-out speed and superior spectral resolution.

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X-Ray Spectroscopy Study of Local Microstructures in CdSe Quantum Dots Prepared by UV Photolithography

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Introduction and Objectives

The fabrication of optoelectronic devices with self-assembled quantum dots (QDs) is costly due to the nature of the growth techniques and the difficulty of controlling their size and uniformity. As an alternative, highly luminescent quantum dots is becoming increasingly demanding for optoelectronic applications, one of such candidate is CdSe QDs. Not only that the UV-photolithographic patterning [1] is possible but also by changing exposure time, the size of CdSe QDs can be tuned. With this technique, CdSe QDs and CdSe QDs/polymer matrix systems were grown on glass substrate and investigated the local microstructure by the extended X-ray absorption fine structure method (EXAFS), X-ray absorption near edge structure (XANES) and X-ray fluorescence to determine the effect in local environment surrounding the Se atoms.

Results and Discussion

The different size of CdSe QDs was formed by changing UV-exposure time between 30 – 60 minutes, the average sizes are found to be 10 nm and 14 nm. In addition, CdSe QDs were fabricated both on glass substrate and on polyvinylcarbazole (PVK) polymer matrix. The local neighborhood of Se atoms is examined to determine the effect of microstructure characteristics in those systems. The chemical nature or electron density at the vicinity of Se atoms in the CdSe QDs samples patterned with exposure time of 30 and 60-minute is the same. The CdSe QDs patterned on PVK matrix clearly exhibits a shift in position of the absorption edge and peak absorption position towards higher energy, resulting from the modification of the electronic structure. The shift of energy position of this sample relative to the energy position of SeO₂ indicates the chemical nature is different from the SeO₂. The EXAFS results indicate that in the samples of CdSe deposited on PVK matrix, some of the Se atoms exist next to other (impurity) atoms than Cd. Cd-Se bond length calculation from Fourier transform XANES spectra is consistent with EXAFS results.

Conclusions

According to both XANES and EXAFS results the CdSe QDs patterned on PVK matrix exhibit different micro-structural properties than that of CdSe QDs on glass substrate.

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PVD-made Thin Films for Quantitative X-ray Fluorescence Analysis

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Introduction and Objectives

For many years physical vapour deposition (PVD) techniques such as magnetron sputtering (MSD) or ion beam deposition (IBD) have been applied in the fabrication of multilayer mirrors and high precision coatings for X-ray and EUV applications. With analysis methods like (μ -)XRF and GI-XRF reaching lower limits of detection and stretching into new application fields, suitable reference and test samples are needed. PVD can be useful here due to its high precision, scalability and reproducibility[1].

Results and Discussion

Examples being fabricated and tested are:

For quantification of very small masses in (T)XRF, reference samples of known composition have to be measured together with the specimen. Masses below ng-range or pm-“thicknesses” were fabricated. Characterization was performed using high sensitivity methods such as AAS, ICP-OES and various synchrotron based XRF techniques at DESY, PTB at BESSY[2] and ANKA. The lateral structure of these samples was analyzed with different electron and X-ray scanning techniques.

XRF measurements in grazing incidence geometry (GI-XRF) taking advantage of X-ray standing waves formed inside and above reflecting samples[3,4] have come into the focus of research by advances in synchrotron technology and computing power. GI-XRF is a promising tool to analyze layered structures and buried materials but strongly depends on reliable optical constants and simulation software to interpret measured data. In order to test and improve simulation software, well defined layered structures have to be manufactured, measured and reconstructed by simulations. In cooperation with research groups in the GI-XRF field such samples have been designed and fabricated.

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Development of Readout Systems for X-ray Diamond Beam Position Monitors

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The mechanical, optical, electronic and thermal properties of diamond make it an ideal material to address the x-ray beam monitoring needs of modern synchrotrons. Diamond Beam Position Monitors (DBPMs) have demonstrated to yield position resolutions of 25 nm for stable beams and have shown linear flux responses of at least 11 orders of magnitude [1]. Readout electronics tailored to suit the performance and integration needs of DBPMs are needed to fully harness the potential of the technology. Sydor Instruments LLC in collaboration with Brookhaven National Laboratory (BNL) has advanced novel readout electronics packages based on BPM readout systems developed for the National Synchrotron Light Source II (NSLSII). Two systems were developed under this collaboration, the SIEPB4 compact electrometer and the SIEPA3P advanced electrometer. Both systems are 4-channel electrometers with internal power supplies to operate DBPMs. In addition, the systems' controls are Ethernet based and compatible with the Experimental Physics and Industrial Control System (EPICS), utilizing custom built Control System Studio (CSS) user interfaces. A general overview of the systems and their functionalities will be presented. In addition, results will be presented of tests performed at the Cornell High Energy Synchrotron Source (CHESS) and at Brookhaven National Laboratory with monochromatic DBPMs previously developed by Sydor Instruments.

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In-situ X-ray Microscopic Study of Materials for Electrochemical Energy Conversion Application

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In-situ characterization combining transmission electron microscopy (TEM) and X-ray imaging provides the capability to investigate materials under real reactions with multi-length scale, from atomic structure to micron size overview. In this work, we have designed an in-situ cell, which is capable of performing miniature electrochemical reaction with the presence of electrolyte and electric bias added, and it is suitable for both TEM and X-ray microscope investigation (full-field and scanning). We have preliminary results on the in-situ investigation of the structure evolution of Li-battery electrode material (AgVO_2PO_4) under battery operation, to demonstrate the functionality and capability of the designed cell. In addition, we are looking forward to applying the cell for other systems such as electrochemical catalysts to give insightful understanding of the underlying mechanism from multi-length scale.

Synchrotron Microprobe Analysis of Biometal Changes in the Aging Retina

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Introduction and Objectives

The presence, in tightly regulated levels, of the essential metals iron, copper and zinc, is important for the physiology of a number of organs including the retina. The retina is composed of several layers with distinct structure and function. In the work presented here a synchrotron microprobe has been used to localise and quantify the three trace metals but also the minor ones P, S, Cl and K, in the retinas of n=5 young and aged mice. The aim is to determine whether there is sustained modification of the levels of these metals with age as associations between afflicting conditions and elevated metal levels is an emerging area of interest in retinology.

Results and Discussion

The x-ray microprobe of beamline I18, at the Diamond Light Source, England, was used to localise and quantify the biologically significant minor and trace elements with a resolution of 2 microns. The elemental maps allow distinction of most of the different anatomical structures of the retina; they are 9 in total in a cross-section of approximately 300 microns. Regions of interest were drawn in each of the delineated regions and average concentrations for each region were compiled for the two groups of samples. A trend of significant elevation of Fe, Cu, Zn and S in the outer retinal region of the aged animals has emerged, varying between 2-fold and 4-fold depending on the element. The common difficulties in such measurements are the extremely dilute nature of some of the elements (Cu < 20 ppm) and the statistical spread of concentrations within the same group of specimens, either due to statistical noise associated with weak signal or because of inherent biological variability. In these measurements the intra-group statistical variation of concentrations in the outer retina was insignificant compared to the degree of change between groups so these first results are very encouraging. The metals play a significant role in regulating the function of a number of proteins therefore the apparent correlation with elevated sulphur concentration is also potentially meaningful. The region that displays this age-related modification is in fact a complex of three layers that are not clearly resolved, the Retinal Pigment Epithelium, Bruch's Membrane and Choriocapillaris. The fact that some of these boundaries remain too small for the size of the beam presents a challenge for future experiments.

Conclusions

Initial results suggest age-related accumulation of essential biological metals in the retina with potential implications in macular degeneration. This work is a prime example of how x-ray microscopy can provide invaluable information in current biological problems. Emerging challenges point to the need for even more advanced x-ray techniques, specifically the increased spatial resolution that novel synchrotron nano-probes can provide.

In-situ Experiments in Laboratory Transmission X-ray Microscopy

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Introduction and Objectives

In the last years full field hard x-ray microscopes have earned their reputation not only at synchrotron beam lines but also as a reliable laboratory equipment for imaging and tomography, with a resolution better than 50 nm. Using a system setup for hard x-ray transmission microscopy with long distance of more than 10 mm on each side between condenser optics, sample and objective lens it is possible to include additional instruments for in-situ experiments into the beam path. We present three approaches for such in-situ experiments: (i) recording of a time lapse image sequence of an oxidation reaction at elevated temperatures, supplemented by recording tomography data under inert atmosphere before and after the reaction; (ii) crack propagation studies in on-chip interconnect structures of microchips by double cantilever beam test and (iii) instrumented micro indentation and compression tests.

Results and Discussion

All experiments were carried out inside a laboratory X-ray microscope using Cu-K α radiation (Xradia nanoXCT-100).

The morphology change of agglomerates of iron powder at elevated temperatures up to 773 K were imaged during heating the sample inside a water-cooled reaction chamber with windows for x-ray imaging, electrical connections for resistive heating, temperature control by a thermal couple and gas supply to provide an inert or reactive atmosphere [2,3]. This chamber is positioned using a separate xyz-stage above the sample. Highly reactive iron powder with a particle size less than 100 nm was used for an advanced hydrogen storage cycle based in the steam-iron-process. Hydrogen is released during the oxidation of iron in water steam, and inversely, hydrogen was used to reduce the iron oxide to metal. We studied the loss of storage capacity by imaging the oxidation process for a loose agglomerate in wet nitrogen atmosphere. The time lapse series shows the formation of an oxide layer at the surface of the agglomerate, and tomograms recorded after the reaction show the three-dimensional evolution of the distribution of material.

The mechanical tests were carried out with custom-built piezo-driven mechanical stages that were specially designed to fit inside the beam path, allowing to record tomographic data sets with no or minor missing data. The miniaturized dual cantilever beam (DCB) test was performed at a multi-level on-chip interconnect structure of a microchip, with Cu interconnects and a so-called low-k dielectric material. The local pathway of cracks in this stack and the effect of crack stop structures was shown [4].

The third in-situ experiment uses a compact modular stage for instrumented micro indentation and compression tests. Phase contrast imaging of crack propagation in multi-component ceramic materials are demonstrated.

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Investigations of Tomographic Elemental Distribution in a Strand of Hair by X-ray Fluorescence Analysis Combined with Transvers Scan of an X-ray Microbeam

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Computer tomography combined with X-ray fluorescence (XRF) analysis is attractive for obtaining tomographic elemental distribution in a soft matter. However, the attenuation of XRF within the sample generally makes rigorous analysis of elemental distribution difficult. We have investigated simplified analysis of tomographic elemental investigation of a strand of hair. The method utilizes the transverse scan of X-ray microbeam over the strand of hair, and the projected one dimensional image of the cross section was compared with the model calculation that consider the axisymmetrical distribution of the element and the attenuation of XRF in the hair.

The experiments were carried out on the BL05SS of SPring-8 by using micro X-ray fluorescence spectrometer designed for forensic investigations. The dimension of the microbeam was ca. 2 micron in diameter, and a silicon drift detector was used for XRF measurements. The feasibility study was carried out, and the obtained projected one dimensional image of the hair was consistent with the XRF images of the cross section of the same sample.

Fig. 1 shows examples of one dimensional images of a hair 20 mm from root. The incident and takeoff angle was 45 deg., and the diameter of the hair was 77 μm . It was found that the method is suitable to determine the distribution morphology and the concentration of the element of interest along with the hair. The obtained elemental information is directly connected to the daily or weekly nutrition.

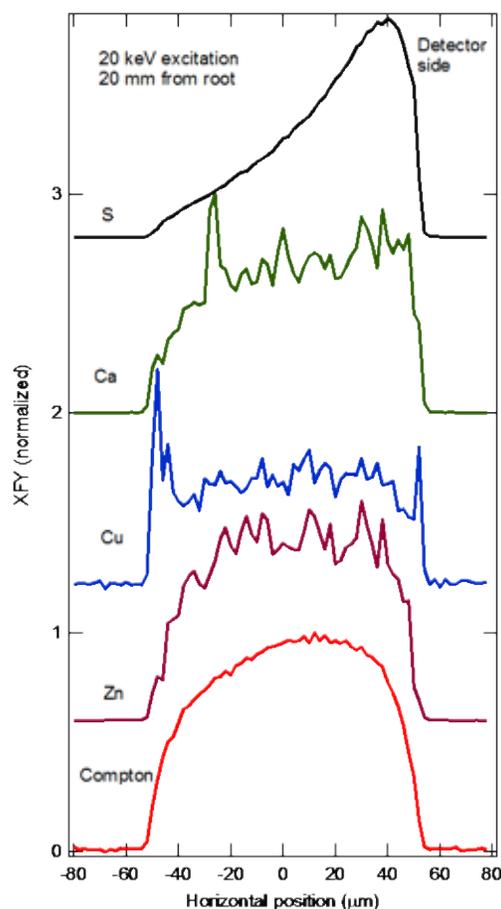


Fig. 1 One dimensional X-ray fluorescence and Compton scattering images of a strand of hair 20 m from root.

PDF Method Development at SSRF

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So called RAPDF (Rapid Acquisition of Atomic Pair Distribution Function) method has been realized using high energy (at about 70keV) X-ray at the 13W imaging beamline at Shanghai Synchrotron. The data quality is extremely high and data collection speed is relatively fast. A comprehensive method platform based on this total scattering method has been built up at Shanghai Synchrotron.

In this poster we describe the setup for both the beamline itself and the PDF platform. Quality evaluation of the data collected using standard sample is described. Limiting effects of low/high temperature and in situ apparatus are also discussed with possible ways to reduce such effects. Data of some typical real samples of interest are present, with focus on our efforts to develop PDF application on low Z materials.

Also we describe our effort to develop PDF method using micro size focused high energy X-ray beam. This has potential application on high pressure experiments using diamond anvil cell (DAC) and also on other fields involve microanalysis. Very preliminary results on effort to fabricate kinoform lens designed for focusing high energy X-ray will be showed.

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High Reflectance Cr/V Multilayer Mirror for Water Window Applications

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Imaging and spectroscopy in the “water window” ($\lambda=2.3-4.4\text{nm}$) has been long pursued in the fields of biology and material science, driven by the natural contrast between carbon and oxygen, and the high spatial resolution provided by the short wavelength. Significant progress has been achieved in producing high flux and coherent light sources in this wavelength region, using synchrotron or free electron laser [1], and high harmonic generation [2]. Besides the high quality sources, multilayer mirror is another key component for the water window microscope [3]. Due to the short working wavelength, the d-spacing of the multilayer is only 1-2nm. This imposes a severe challenge for the fabrication of such multilayer mirrors. Cr/V multilayer is one of the promising candidate working near the V-L edge ($\lambda=2.4\text{nm}$). To develop high reflectance multilayer mirror for this region, the physical structure inside the multilayer with different layer thicknesses were investigated. Interface engineering was further applied to this metal/metal system which produced a maximum reflectance of 24% near the V-L edge at 42° grazing incidence.

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X-ray Lens Characterization using Ptychography

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Quantitative wavefront measurement is extremely valuable for boosting the development of high-resolution X-ray optics. It evaluates the optics performance and analyzes lens defects to provide feedback for fabrication process; it also provides a functional survey on alignment of optical components for optimized operation condition. A diffraction-based imaging technique, ptychography, is recently emerging as a robust and precise analytic tool for characterizing wavefront of focusing optics. Here, we present recent characterization works of focused wavefronts at NSLS-II on a variety of X-ray optics, including zone plate, K-B mirrors [1], flat 1D[2]/2D[3] and wedged [4] multilayer Laue lenses.

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Chemical and Morphological Heterogeneity in Zinc Oxide Thin Film under Humidity Treatment

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Zinc oxide (ZnO) has a wide range of applications including electronics, solar cells, and humidity sensors. It has been observed that when zinc oxide thin film is subject to humidity treatment, the surface of the film exhibits morphological heterogeneity. However, the nature of this heterogeneity, such as chemical and elemental compositions, remains unclear. Here, we focus on studying the chemical and morphological heterogeneity of ZnO thin film when the materials are subject to humidity treatment and compare it with the untreated pristine samples.

We will utilize the Sub-micron Resolution X-ray Spectroscopy (SRX) beamline at National Synchrotron Light Source II to carry out x-ray fluorescence and absorption spectroscopy measurement on the ZnO thin film. Other characterization techniques including scanning electron microscopy and Energy Dispersive X-ray Spectroscopy were also used to provide supplemental information. The materials under investigation include ZnO and Al doped ZnO on Si substrate. By comparing the results from humidified samples and pristine ones we aim to study the environment induced heterogeneity in coating materials.

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Soft X-ray Ptychography Sheds Light on Electrochemical Processes

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The presentation reports on novel Soft X-ray Fresnel CDI ptychography results, demonstrating the potential of this method for dynamic studies. Our in-situ ptychography experiments explored the electrodeposition process of Mn and Co/polypyrrole (PPy) nanocomposites, Pt-free candidates for fuel cell catalysts [1]. The measurements were performed using a custom-made three-electrode microcell, sketched in Figure 1. The cell has been developed at the TwinMic beamline of Elettra during a series of experiments that were continued at the SXRI beamline of the Australian Synchrotron [2].

The ptychography-based investigation of the electrodeposition dynamics was executed during the in-situ electrical biasing of the electrochemical cell. Besides the observation of morphological changes, we retrieved the spectroscopic information, provided by multiple ptychographic energy scans across Mn and Co edges. The most important issues related to the computational aspects of such experiments will be outlined and potential improvements of the methodology will also be discussed.

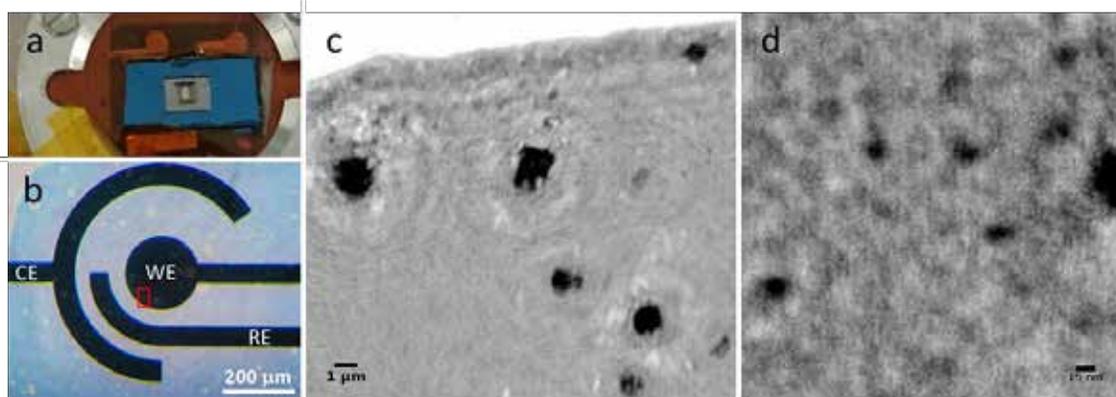


Figure 1. Visible light images of the electrochemical cell (a,b) and high resolution (<20nm) ptychographic reconstruction of an electrode area close to the electrolyte interface where the dark regions correspond to Mn-Co nanodeposits (c,d).

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X-ray Nanodiffraction meets Materials Science

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The Nanofocus Endstation of beamline P03 (PETRA III, Hamburg) is operated jointly by Helmholtz-Zentrum Geesthacht and the University of Kiel, and is one of the very few synchrotron endstations providing the experimental conditions for scanning X-ray nanodiffraction. This technique, in turn, is an excellent tool for materials science. It readily serves structural information with sub-micrometer spatial resolution from crystalline and semi-crystalline materials (metals, biomaterials, synthetic compounds) for the retrieval of e.g. grain orientation, residual stress profiles, crystal structure or texture. Because of the long focal distance focusing, the wide energy range of the P03 beamline and the highly adaptive sample positioning system, high resolution nanodiffraction experiments can be performed even in extended sample environments. A beam size of typically (250 nm)² is used for these experiments and is generated using a long focal distance KB-mirror focusing system.

The strong focus on materials science at P03 is demonstrated by the wide range of experiments already performed with in situ sample environments: pressure, indentation force, tensile stress, fluid shear, magnetic fields - all of these parameters were successfully modified in situ and combined with the high spatial resolution provided by nanofocused beam [1-6].

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Ptychographic Imaging at DLS Coherence Beamline I13-1

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Introduction

Coherent diffractive imaging techniques exploit oversampling to recover the phase information lost in the measurement of far-field diffraction patterns. In the case of ptychography, the sample is scanned with a coherent x-ray beam taking diffraction measurements while ensuring some degree of overlap between neighbouring areas of illumination [1, 2]. From these diffraction frames applying adequate constraints the projected transmission function of the object can be retrieved simultaneously with the probe, i.e. the function describing the radiation incident on the sample. Ptychographic methods can be extended to three-dimensional objects simply by recording projections for several sample orientations and applying conventional tomographic algorithms to the set of 2D images obtained with ptychographic algorithms thus finally rebuilding the whole volume [3]. This approach already allows to get high-resolution 3D images and nonetheless further improvements could be developed, for instance in order to decrease the dose and time required for each experiment.

Results and objectives

We have implemented and successfully performed ptychography experiments at I13-1, the coherence beamline at Diamond Light Source [4]. We succeeded in imaging technologically interesting materials such as nanoporous gold in both 2D [Fig. 1] and 3D. We currently aim to achieve faster scan rates and further improve 3D data acquisition and reconstruction techniques. Other possible developments include multi-wavelength imaging and on-the-fly scanning ptychography.



Fig. 1. Phase image of a nanoporous gold sample

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Towards A Cross-Platform Open Source Tomography Toolbox*

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Introduction and Objectives

We report on an ongoing project to create a portable tomography toolbox to enable the processing of user data on various computing platforms. Our goal is to create a collection of open source software applications in a single integrated package that is cross-platform and user friendly, to facilitate analysis and interpretation of tomographic data emerging from the Brookhaven National Synchrotron Light Source (NSLS) II and Center for Functional Nanomaterials (CFN) facilities as well as other locations. We aim to increase scientific productivity by providing a platform that is suitable for quick introduction of improvements in reconstruction and artifact-removal algorithms as well as advancements in computing hardware, such as the GPU accelerators and many core architectures.

Results and Discussion

While there are already a few software packages available for tomography, a universal environment is lacking. Aside from the complex library dependencies that sometimes come with the software packages, they may require different input file formats, putting a burden on the users to handle the file conversion and negatively impacting scientific productivity. The emerging virtualization technology Docker [1] allows for isolation of runtime environment in separate containers, making it possible to integrate independent software packages in a single location. To demonstrate, we dockerized TomoPy [2] and tomo_display [3], two popular tomographic reconstruction software packages, and used them to analyze the images obtained at the NSLS X2B beam line. We compared the performance of the containerized TomoPy and the native version and found no significant performance loss. We also explored the possibility of using TomoPy to perform tomography reconstruction for electron microscopy at CFN. At present, the package requires the users to be familiar with command line tools. Future development will incorporate a web interface to make the package more accessible to users. We will also incorporate additional modules for preprocessing of images, additional reconstruction methods, and visualization/analysis of the tomographic data based on x-ray attenuation, x-ray fluorescence, or electron scattering. We plan to make this toolbox generally available through a web-based location.

Conclusions

Docker makes it possible to provide easy access to a variety of open-source software relevant to tomography data at a single location. The toolbox will complement other more specialized software under development at BNL and elsewhere.

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A Hard X-ray Reflection Zone Plate for Femtosecond Spectroscopy

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Introduction

The dispersive and 2D-focusing properties of total external reflection zone plates (RZPs) offer a wide range of applications in soft X-ray spectroscopy and monochromatization [1,2]. Recently, in a proof-of-principle experiment at the KMC-2 beamline of BESSY II, the spectroscopic properties of a hard X-ray off-axis RZP for the Ni K-edge (8.3 keV) were successfully characterized [3]. Here, we present our newest results from a subsequent test at the 1-BM Optics beamline of the Advanced Photon Source (APS) at the Argonne National Laboratory with a flux density of 9×10^9 photons per second per mm^2 and a nearly parallel input beam. The use of the APS allowed us to approach the RZP's scanning range and spatial resolution within the same order of magnitude as the theoretical limits.

Measurements and results

The total external reflection zone plate was positioned at 11 m distance from the white beam slit of the 1-BM beamline at the APS. Without the use of any focusing mirror, the nearly undistorted wave front provides an effectively parallel beam, enabling a focal spot size down to $1.0 \mu\text{m}$. While varying the photon energy from 7.6 to 9.0 keV in 100 eV steps, the position and size of the focal spot at the RZP's exit arm length (100 mm) were measured in both lateral dimensions with a knife-edge scanning device. Figure 1 shows the resulting data for the energy resolving power plotted over the corresponding photon energies. Compared to our previous results [3], a 4-fold higher resolving power up to 4×10^2 at the design energy of 8.3 keV was achieved. Over an energy range of 460 eV, the resolving power can be kept as high as 2×10^2 or better.

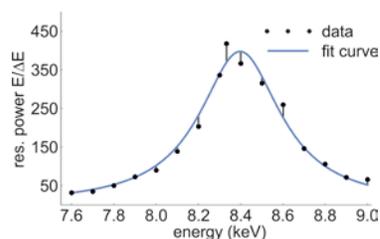


Figure 1: Resolving power $E/\Delta E$ of the RZP varying with the photon energy E

Conclusions

Due to the excellent beam properties, the RZP spectrometer showed a resolving power up to 4×10^2 with a spectral range of 460 eV around 8.3 keV. Together with the time elongation of ~ 0.35 fs and the high efficiency (17.8%) of the RZP we paved the way to hard X-ray wide-range and high-resolution femtosecond spectroscopy.

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Development of Grating-Based Phase-Sensitive X-ray Imaging Microscopy under JST-ERATO Project

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X-ray phase imaging based on transmission gratings has recently attracted attention [1] because weakly absorbing objects can be imaged with a comparably simple configuration. Its spatial resolution is normally limited by the period of gratings. One characteristic of this method is that a spherical X-ray beam is available while some other phase-contrast methods require the use of a parallel beam with crystal optics. Therefore, we can find an approach to overcome the resolution limit by combining the grating optics with X-ray imaging microscopy with a Fresnel zone plate (FZP). The alignment of gratings is designed by considering the focal point of FZP as an X-ray source. This also implies that this combination can append a phase-contrast mode to a conventional X-ray imaging microscope. X-ray phase imaging does not mean simple phase-contrast imaging but an imaging technique with quantitative phase measurement. In grating interferometry, the fringe-scanning method (or phase-stepping technique) is normally employed by displacing a grating. Therefore, tomographic image reconstruction mapping the refractive index is feasible. This property is advantageous over Zernike phase-contrast microscopes, which is especially problematic in quantitativity for non-weak phase objects.

The combination of an X-ray Talbot interferometer with a microscope employing a FZP was successfully demonstrated at SPring-8 [2]. X-ray phase tomography based on this configuration is applied to the study of bone remodeling mechanism [3]. Other combination configurations with a single phase grating [4] and a Lau interferometer [5,6] were also studied.

Recently, we have launched a five-year project for promoting phase imaging with not only X-rays but also neutrons, electrons, and others ('ERATO Momose quantum-beam phase-imaging project' supported by Japan Science and Technology Agency (JST)). As one of subjects in this project, the development of grating-based nano-phase imaging system at SPring-8, Japan. While the demonstrative experiments mentioned above [2-5] were performed with the magnification of 20 and the spatial resolution of resultant phase tomograms was about 1.5 μm , we are designing a combination of a Talbot interferometer and a 100-fold microscope with a 200 nm resolution to measure the bone mineralization degree locally around bone lacunae and canaliculi.

We will present the optical concept of grating-based micro-phase imaging with the design of grating optics in addition to the overview of the JST-ERATO project.

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XRF Imaging and Spatially Resolved XAFS in the Tender (1-5 keV) Energy Range: A New Microprobe at NSLS and NSLS-II (initial results, future plans)

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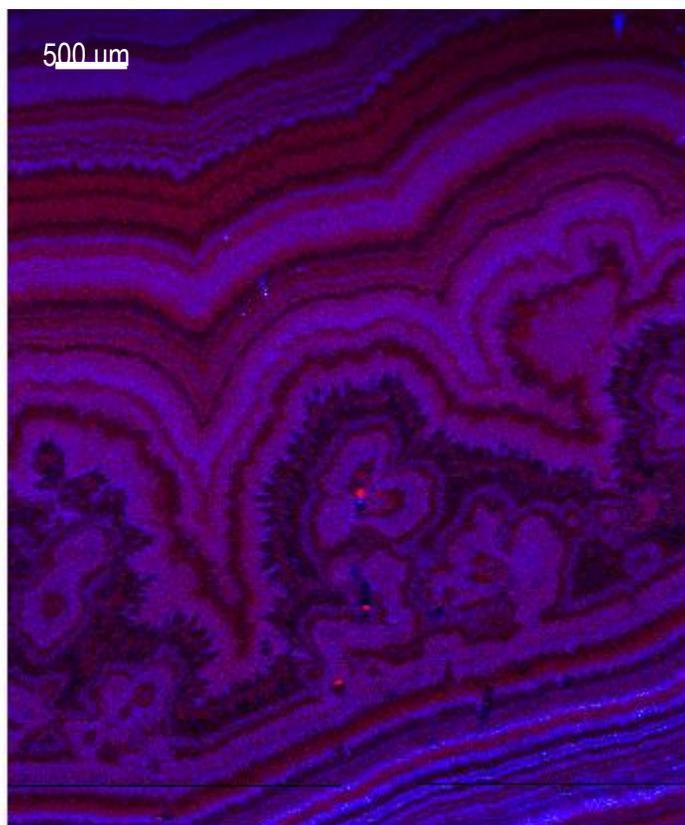
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A new tender-energy (1-5 keV) microspectroscopy endstation was recently commissioned and user-tested at NSLS Beamline X15B, in preparation for its installation at NSLS-II. Development of this helium-atmosphere microprobe was jointly funded by NSF, DOE:BES, and NASA. This unique facility enables fast on-the-fly X-ray Fluorescence (XRF) imaging and energy scanning (microbeam quick EX-AFS) of diverse samples in a non-vacuum environment, including *in-situ* cells for energy storage materials and catalysis.

Initial results will be presented. These include sulfur (K-edge 2.5 keV) in geologic materials, plant tissue, protein crystals, and battery electrodes; Phosphorus (K-edge 2.1 keV) in extraterrestrial materials, soils and minerals; Calcium (4 keV) in biominerals and geomaterials; Uranium (M₅-edge 3.5 keV) in geologic and environmental samples; as well as Si (1.8 keV), Al (1.6 keV) and Mg (1.3 keV) in various samples. *In-situ* measurements of a Li-S battery system – during operation -- will also be presented. Successful testing of the compound-focusing optics and prototype controls/data-acquisition system at NSLS Beamline X15B were significant accomplishments toward rapid implementation of this endstation at NSLS-II.

The NSLS-II TES, “Tender Energy and Spatially Resolved X-ray Absorption Spectroscopy and Imaging,” beamline will be the first beamline at NSLS-II to use solely the dipole Bend-Magnet source, will be situated at port 8-BM, and is scheduled to be completed by late Summer of 2016. The optical design incorporates a collimating/harmonic-rejection mirror pair with fixed offset over a range of pitch from 6.8 to 20 mrad, a fixed-exit monochromator, and a toroidal macrofocusing mirror focusing to a secondary source aperture (SSA). Beam diverging from the SSA is refocused by a custom KB mirror pair to the sample position. Total acceptance is up to 2.5 x 0.4 mrad, and flux delivered to the sample is projected to be up to 10¹² photons/second. Spot size will be user-tunable by adjusting the SSA at the expense of flux, from roughly 1 to 30 μm.

Shown at right is an XRF image of cave-deposited calcite, with U represented by blue colors and S represented by red.



The Soft Scanning Transmission X-ray Microscope at SOLEIL

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In spring 2015, a new zone plate based scanning transmission x-ray microscope (STXM) has been installed at the HERMES soft x-ray beamline [1] at the SOLEIL synchrotron. The beamline covers an energy range from 70 – 2500 eV, providing access to a large variety of K and L absorption edges. With special emphasis on material science, various spectro-microscopic methods, like XANES, XAS and XMCD-XMLD can be applied to investigate the sample's bulk and surface properties, at high spatial resolution down to 20 nm. The design of the STXM allows combining different sample environments and detectors in order to provide a highly flexible experimental setup. This presentation gives an overview of the STXM assembly and first performance measurements are presented.

The STXM optics assembly is mounted in an aluminum vacuum chamber which rests on a heavy-duty polymer granite with passive vibration damping. The granite can be precisely aligned using a motorized 5-axis positioning system [2]. With an in-vacuum differential interferometer that measures the lateral displacement between the zone plate and the sample [3], high position stability of less than 2 nm RMS is achieved. The modular sample environment allows imaging under versatile sample conditions: temperature controlled cryogenic sample mount with temperatures down to -150 °C, magnetic system with 2D vectorial adjustable field in magnitude and direction within ± 200 mT, electrical insulated sample mount for photo current read-out capability and rotatable sample mount, e.g. for in-plane and out-of-plane magnetization experiments. A photomultiplier and optional a quadrant diode detector are being used to acquire the transmission signal, while a photoelectron multiplier serves as surface sensitive detection device.

Commissioning of the STXM is scheduled for fall 2015 and initial user operation shall take place in early 2016.

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Development of Fast Scanning X-ray Fluorescence Microscopy at the LNLS D09B-XRF Beamline

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A large refurbishment started in 2013 with the aim of upgrading the focusing optics of the LNLS D09B-XRF beamline. Today, the beamline comprises a new X-ray microfocusing optic based on a pair of bendable mirrors in the Kirkpatrick-Baez arrangement [1]. The optical system can produce an X-ray microbeam of around 12 μm (vertical) x 22 μm (horizontal) in size with a flux density between 10^{11} and 10^{12} photons/sec at the focal spot when white beam is used. The upgrading strategy also involved the migration of the control system to a NI PXI system (National Instruments Corporation), integrated with an open source EPICS/Linux platform [2]. The control of the scanning for (2D/3D) experiments has been achieved via connection to EPICS server and to the macro language SPEC (Certified Scientific Software; <http://www.certif.com/>), being able in this way to read XRF data from the 4-channel DXP-XMAP module (XIA LLC). Point-to-point data acquisition can be performed however at the cost of a significant timing overhead, having a great impact on the overall time for each scan. Aiming to produce faster XRF data acquisition, a new approach using a PXI system as a hardware triggering interface was developed in order to provide a continuous scanning mode of operation ("on-the fly" scans) at the LNLS D09B XRF beamline. In this work, the main features of the LNLS XRF microprobe station as well as a description for setting up a general approach for fast scanning mode operation will be presented.

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Hard X-ray Bimorph KB-system for the LIX beamline at NSLS2

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Introduction and Objectives

A pair of X-ray bimorph mirrors [1-4] composing an adjustable-focus KB-system have been realized and tested for the LIX project at NSLS2. The LIX project is requiring mirror optics that can be operated as focusing device over an extremely large range of focal distances (from $q_{\min}=12$ m to $q_{\max}=23$ m). Once integrated in their mechanics the mirrors are expected to achieve sub-micrometer spot sizes and for this purpose the slope errors are required to be below 200 nrad in the whole focusing range.

Results and Discussion

The mirrors (2nd generation technology [4-5]) have been equipped with piezos bonded to the side faces of a monolithic substrate and have been polished and shaped prior installation at the beamline. The mirrors have dimension 800x60x52 mm³ and 500x60x52 mm² respectively and have been pre-polished to cylindrical shapes with radius of curvature exceeding in both cases 6 km. The necessary elliptical shape has been generated by determining the voltage distribution leading to the required elliptical shape on the base of full 2D interferometry profile data. In contrast to the oldest technology [3], this bimorph design makes it easier to polish the substrates using deterministic polishing. To optimize the polishing process and to achieve the best figuring errors data, a metrology feedback is realized measuring the mirror in its frame in the range of focusing distance required.

Conclusions

The optimization of two elliptical bimorph mirrors based on the 2nd generation technology is presented. The voltage mirrors setting are obtained from inspection of interferometry data profile allowing obtaining ultimate mirror figure errors within fews shaping iteration. The possibility to further improve the mirror shape improving the beamline performance by in-situ optimisation will be discussed.

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Synchrotron micro-scale measurement of metal localization in wetland plant root system

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Introduction and Objectives

Localization of metals in root tissue reflects the process of root metal uptake and transportation. In the rhizosphere, Fe plaque plays a role in controlling metal uptake and transportation. The objective of this study is to investigate the possible biogeochemical processes that control the mobility of metals (e.g., Cu, Fe, Pb, Mn and Zn) in *T. latifolia* L. and *S. alterniflora* root tissues using synchrotron μ -XRF technique.

Results and Discussion

We found that metal spatial distribution patterns in the root epidermis and vascular tissue were metal dependent. The results show that Fe plaque is mainly distributed in the root epidermis. Other metals (e.g., Cu, Mn, Pb and Zn) are associated with Fe in the epidermis possibly due to the metal scavenge by Fe plaque (Fig. 1). Factor analysis showed strong association between Pb and Fe, Cu and Zn in the dermal tissue, while Zn, Mn, and Cu shared strong association in the vascular bundles. This investigation provides insights of metal uptake and transport processes in the root system.

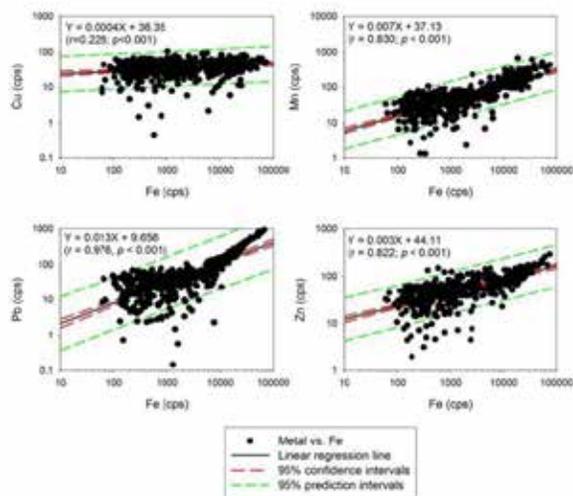


Fig. 1 Significant correlation ($p < 0.001$) of Cu, Mn, Pb and Zn with Fe in the epidermis of *Spartina alterniflora* root.

Conclusions

The synchrotron XRF measurement shows that Fe plaque was found to accumulate in the root epidermis, where Cu, Mn, Pb and Zn showed significant ($p < 0.001$) correlations with Fe possibly as a consequence of metal adsorption by Fe plaque. The results suggest that Fe plaque possibly acts as a barrier for Pb and a buffer for Zn, Mn and Cu in the plant roots. This study provides useful information on metal transportation and distribution in the wetland plant root system.

Fluorescence Tomography with Absorptive Samples at the Australian Synchrotron

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Introduction and Objectives

The X-ray Fluorescence Microscopy (XFM) beamline at the Australian Synchrotron is host to a Maia detector array, jointly developed by CSIRO and BNL [1]. The Maia's large collection area and optimised photon event processing combine to achieve high sensitivity, allowing scanning of bigger imaging regions at higher rates and higher resolution than current alternatives [2]. This makes the beamline well-suited to performing X-ray Fluorescence Computed Tomography (XFCT) of large specimens at high resolution, resulting in chemical-elemental maps of 2d slices and 3d volumes.

When acquiring XFCT data, many samples are strongly absorbing at the fluorescence energies of interest. This, along with absorption at the incoming beam energy, breaks the assumptions of conventional tomographic approaches, leading to errors in naive reconstructions [3]. We aim to develop absorption-corrected XFCT at the XFM beamline, leveraging the unique properties and abilities of the Maia detector system. We report on progress toward this goal.

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Hard X-ray Single-shot Holographic Diffraction Imaging at I13 DLS

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X-ray Fourier transform holography (FTH) is a coherent lensless imaging technique that encodes the complex wavefield resulting from the interference between a scattered object wave and a reference wave generated by an off-axis point scatterer [1, 2]. The amplitude and phase of the sample is recovered from the far-field diffraction pattern by non-iterative inversion.

We have successfully implemented hard X-ray holographic diffraction imaging at Diamond I13 [3] using nano-fabricated phase-shifting objects with nanometer-sharp edges as the reference source, which provide amplified image contrast while preserving resolution compared to an absorbing mask. The flexibility of the setup allows the separate translation and rotation of the sample and the reference, opening possibilities for ptycho-holography [4] and tomo-holography. We obtained reconstructions of the amplitude and phase of strongly and weakly scattering inorganic samples with a resolution in the 150nm range by single-shot parallel-beam illumination at $E=9.1\text{keV}$. We have also applied the method to image biological samples such as plankton and bacterial cells. As a lensless single-shot measurement, hard X-ray holographic diffraction imaging holds potential for 3-dimensional structural imaging and is well-suited for ultra-fast imaging [5] with X-ray free electron lasers.

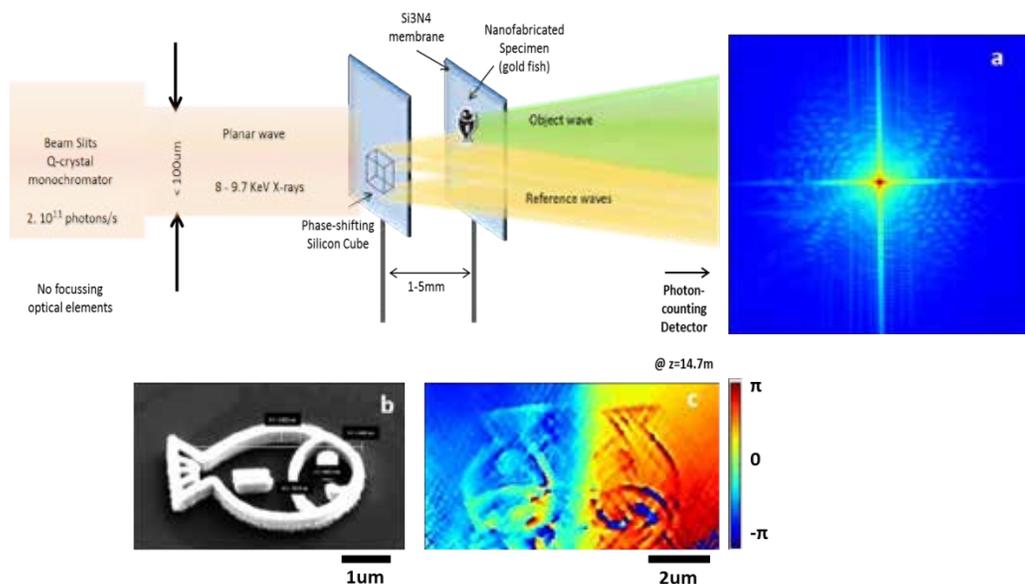


Figure 1 (a) Diffraction pattern recorded with a photon counting detector. (b) SEM image of gold fish test sample of 350nm thickness and 4μm length. (c) Phase reconstruction of (b) by linear differential of the inverse FFT of (a).

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Fully depleted pnCCDs with adaptable dynamic ranges

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Introduction and Objectives

pnCCDs have been used in different fields, notably in X-ray astronomy, synchrotron and free electron laser (FEL) science. At FELs, pnCCDs are used as imaging X-ray spectrometers because of their high readout speed, high and homogeneous quantum efficiency, low readout noise and high radiation hardness [1]. The high brilliance of the FEL X-ray flashes enables scientists to decipher the atomic structure of molecules with only one X-ray flash. Depending on the sample, the resulting diffraction peaks can be very intense, with up to 10^7 X-rays per reflection. Once the X-rays saturate the pixel, any further charge spills over into the adjacent pixels. The intensity information is still correct, but the spatial distribution broadens. For X-ray diffraction experiments, the structure of the sample is calculated from the position of the reflection peak, and the degradation of the spatial intensity distribution results in a loss of the structural information. Meanwhile, in other areas, only a few photons or no photon hit the detector. Detector systems for FELs must be able to process these extreme differences in intensity without sacrificing spatial resolution.

Results and Discussion

Through advancements in detector design, the pnCCD can be used as single photon counting detector for X-ray spectroscopy with a high spatial and energy resolution, and as integrating detector for X-ray imaging, with count rates up to 10^3 photons with 1 keV per pixel and frame. We present how the internal potential distribution of the pnCCD can be adjusted by the operation voltages according to the needs of the experiment.

Specifically, we describe how the charge handling capacity of a fully depleted, back illuminated pnCCD, with a pixel size of $48\ \mu\text{m} \times 48\ \mu\text{m}$, has been increased from approximately 80,000 signal electrons to 400,000 signal electrons. The electric potential conditions during charge collection, storage and transfer were studied with two-dimensional numerical device simulations. Relating the charge handling capacity to an electric capacitance, the pixel full well capacity depends on the height of the potential barrier and the storage depth. The experimental confirmation of the simulation results was performed with a laser setup that allows for the injection of different amounts of charge in individual pixels. Moreover, a new internal anti-blooming mechanism was developed. In this mode excess charge is drained to the inner substrate via existing MOS contacts which are used as anti-blooming gates.

Conclusions

Because of the advanced design of the pnCCDs, their properties can be adapted to the requirements of the individual experiments. Thus, high energy and spatial resolution modes are available as well as high charge handling capacity and anti-blooming modes for handling extremely high amounts of signal charges.

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Effects of different fixation methods in the study of *Rhodnius prolixus* head using 3D microCT imaging

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Introduction and Objectives

Microcomputed tomography (microCT), has become an important tool in studies of insects, mainly *Rhodnius prolixus* [1], the insect vector of Chagas' disease. Studies have shown [2] that with an appropriate fixative, microCT can produce high-quality images of soft tissues.

In this work, fixative methods were used to improve 3D imaging visualization of internal structures of *Rhodnius prolixus*. All specimens were imaged using microtomography of IMX beamline at Brazilian Synchrotron Light Laboratory. The fixatives utilized were formalin, gluteraldehyde, Bouin's Fluid and osmium.

Results and Discussion

All images allowed the visualization of many structures of the *Rhodnius prolixus* head, and the results showed relevant differences in the images. Some fixatives highlighted the internal structures better than others and allowed the visualization of important organs in the study of Chagas' disease (protocerebrum, trachea and pharynx), as can be seen in Figure 1.

Conclusions

Using simple fixatives, microCT can provide high-contrast images of insect's tissues. The results revealed that it is possible to improve visibility of details in *Rhodnius prolixus* 3D images by the appropriate use of fixative and staining methods.

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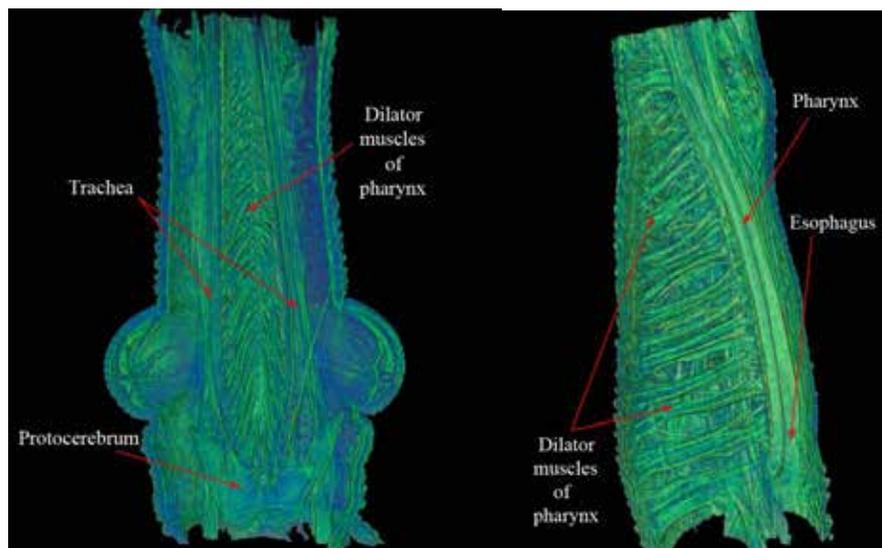


Figure 1: Longitudinal and sagittal section of *Rhodnius prolixus* head.

Multi-scale 3D Structural Characterization with Dark-field X-ray Microscopy

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The physical properties of materials derive from their internal structure, which is typically organized hierarchically across several length scales. Facilitating the direct study of multi-scale structures and their interactions therefore provides valuable insight into a wide range of materials. Here we describe dark-field x-ray microscopy; a new non-destructive technique for the three-dimensional mapping of orientation and strain in embedded volumes of dense matter on length scales from 100 nm to 1 mm (Fig. 1a) [1]. Two recent applications of the new technique are presented: the mapping of sub-grain evolution during the annealing of plastically deformed aluminum, and the quantitative mapping of domains and strain fields within functional ferroelectric ceramics (Fig. 1b). Crucially, the ability to directly characterize these complex, multi-scale phenomena *in situ* constitutes a key step towards the formulation and validation of multi-scale models that account for the entire heterogeneity of a material.

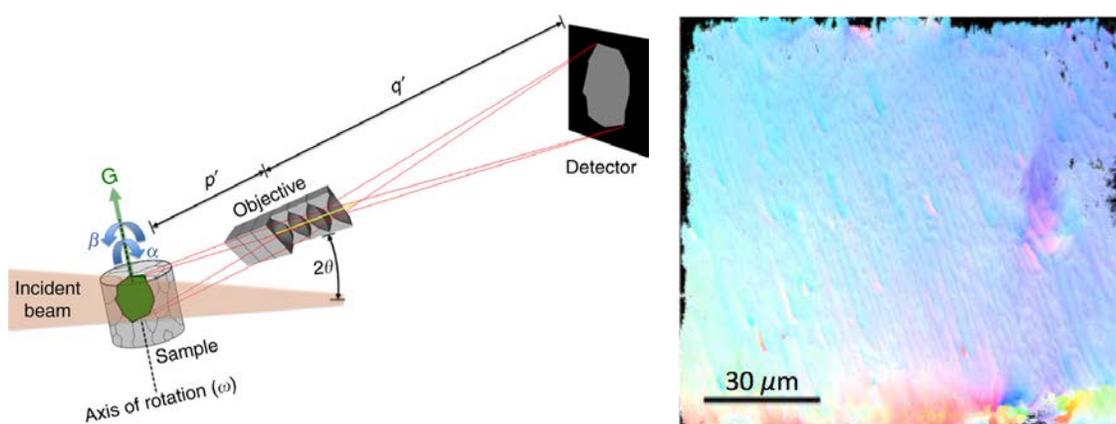


Fig 1. (a) Schematic of the dark-field x-ray microscope, where the Bragg-diffracted radiation is magnified by the x-ray objective. (b) Spatial map of orientation distribution of ferroelectric domains in a KNbO_3 crystal obtained using dark-field x-ray microscopy.

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Development of Tikhonov Regularization Algorithms with Descriptive and other *a priori* Limitations for Solving Deconvolution Problems Arising in Micro XFA

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X-ray fluorescence analysis on SR beams with polycapillary lenses (micro XFA) enables elemental analysis in volumes with linear dimension within the diameter of the focal spot. The use of two lenses in the confocal scheme confines the area under study to a "pixel" about 15x15x15 microns [1].

The spatial resolution can be improved up to several microns using computational methods for solving inverse problems. The micro XFA problem is based on solving a three-dimensional deconvolution equation [1] and is reduced to a system of linear equations of a specific "block" structure [2]. A lot of solution methods are known that were developed for solving such problems arising in image processing ("image deblurring"). For example, iterative regularization methods are convenient and widespread [3]; variational regularization algorithms are also applied [4]. In any case, good-quality solutions require application of algorithms with good regularization properties such as stability and high convergence. The micro XFA problem is very ill-posed because of the large width of the instrument function ($\text{FWHM} > 6$ for image reconstruction with accuracy of 1-2 microns). In other words, the influence of signal errors on the solution is substantially higher, and this imposes high requirements on the algorithms. Therefore, a lot of methods are not applicable at all at the actual noise levels (0.1-10%). One can try to improve the result quality with due account of *a priori* information.

We took two promising solution methods based on the Tikhonov regularization method and taking into account *a priori* information and adapted them to the micro XFA deconvolution problem, simultaneously examining their effectiveness. The first method uses a technique of confinement of the solution space with "conical" sets. The method allows one to take into account monotony, convexity, non-negativeness and so on without the use of projection operators. It is known that in some cases [4] the use of this method of due account of *a priori* information gives solution of high quality. The second regularization method is based on the selection of a suitable boundedly-sourcewise solution subspace [4, 5]. This is a very strong *a priori* limitation, and various techniques based on it were originally intended for solving inverse problems of mathematical physics. Currently, we are adapting these two methods to the needs of inverse deconvolution problems. For the first method, the matrixes of "descriptive limitations" were already composed for a number of typical instances of *a priori* information.

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Ultrasmooth reflective layer for hard X-ray single bounce capillaries

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Determination of the spatial distribution of low-Z elements based on their X-ray fluorescence requires certain design of X-ray optics, used for both synchrotron radiation micro-fluorescence or stand-alone micro-XRF spectrometers. The solution described in the literature [1,2] is based on the two glass polycapillary optics: the first one is used to focus the primary beam on the sample and the other is used in front of the EDX detector, to restrict the field of view (confocal geometry). This system allows improving the depth resolution between 100 μm at 1 keV and 30 μm for 17.5 keV.

The goal of our investigation was to replace the primary polycapillary optics with a newly-designed single bounce metallic capillaries, in order to improve resolution at energies below 9 keV. The new, ultrasmooth metallic capillaries were invented and manufactured in-house. The research on our single bounce gold capillaries with elliptic internal shape have been carried out for a few years [3,4]. However, it has been recently redesigned and redeveloped, which resulted in significant improvement of its characteristics. Surface roughness was reduced up to 0.5 nm and slope error to 0.3 mrad. For these capillaries, an expected depth resolution varies from 3 μm for 1 keV and 10 μm for 9 keV.

The developed method of manufacturing our ultrasmooth reflective layer is to be used for manufacturing for X-ray metallic capillaries which are to be tested in our in-house system (micro-XRF spectrometer). Ultimately, we hope the newly designed capillaries will also be used as optical elements in scientific devices for X-ray nanotomography, X-ray microdiffraction, X-ray microfluorescence, in synchrotron sources as well as for X-rays from laboratory-scale sources.

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Microfabrication and Testing of Refractive Hard X-ray Optics

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Introduction and Objectives

Refractive lenses are versatile optical components and act e.g. as condensers or objectives in hard ($E > 10$ keV) X-ray microscopes [1]. One-dimensional focusing lenses may be realized by microfabrication techniques, whereas the main challenge is the transfer of the lithographically defined two-dimensional pattern into the substrate. Deficiencies in the fabrication result in non-uniform lenses. Likewise, X-ray absorption in the lenses limits flux gains and resolution. By optimizing the manufacture and introducing new lens materials we seek to realize more uniform and more efficient X-ray optics.

Results and Discussion

- With respect to Si lens design, we included sacrificial structures surrounding the lens target structures. This effectively improved the sidewall verticality upon deep reactive ion etching. Process control was facilitated by a characterization procedure based on replica molding and atomic force microscopy [2].
- An absorption-minimizing (kinoform) lens comprising 60 ‘adiabatically arranged’ single lens elements was realized and tested at ESRF ID06 (Figure 1). We measured a 180 μm long line beam with a waist of 300 nm (FWHM), corresponding to an aspect ratio of 600. A flux gain of 75 at 17 keV was achieved.
- We explored a new route for X-ray lens manufacture: injection molding. A preliminary test of a thermoplastic lens showed a 60 μm long line beam with a waist of 700 nm and a gain of 50 at 17 keV.
- We addressed the challenge of making objectives in silicon by the interdigitation of lenslets alternately focusing in the vertical and horizontal directions. With a silicon objective in a bright-field hard X-ray microscope we demonstrated a resolution of 300 nm, close to theoretical expectations [3].

Conclusions

Including sacrificial structures in the lens manufacture facilitates obtaining uniform silicon X-ray lenses with etch depths beyond 100 μm without sacrificing optical quality. Polymer lenses produced by injection molding are promising in respect to high efficiency optics at low cost. The silicon objective presents a viable alternative for imaging with hard X-rays.

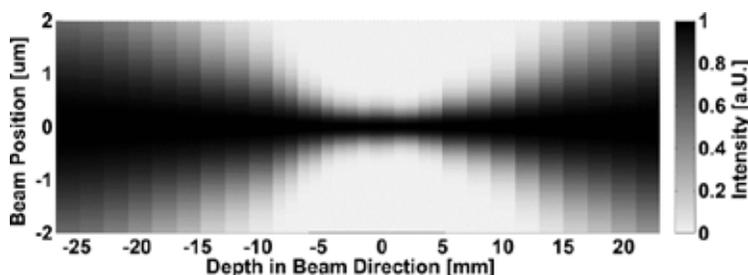


Figure 1. Reconstructed vertical profile of a 180 μm long line beam based on absorptive knife-edge measurements around the focus of a silicon lens with focal length of 230 mm at 17 keV.

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Can cryo-cooling mitigate chemical changes for hydrated samples?

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High intensity ionizing radiation such as beam from an X-ray beamline can cause radiation damage (chemical changes and/or physical damage) to the sample. The amount of beam damage depends on flux density, total dose, temperature, concentration, hydration and chemical species, among others.

For hydrated samples, beam-induced radiolysis of water is of great concern. X-rays dissociate water in the sample creating radicals of hydrogen and hydroxide ($H\cdot$, $OH\cdot$), hydrogen peroxide (H_2O_2), ozone (O_3), hydrated free electrons (e^-_{aq}) and other species which can reduce or oxidize elements of interest within the sample. Another concern is mass loss- X-rays can ablate components of the sample and change the absolute mass, concentration and hence the signal strength measured over time. These beam-induced changes make it difficult to determine the unaltered chemical state of redox-active elements in hydrated samples.

Lowering the sample temperature is presumed to retard beam-induced chemical changes by slowing chemical reactions rates and diffusion. An ambient pressure cryogenic sample chamber was used to explore low temperature beam damage mitigation of hydrated samples.

In the first experiment, chemical speciation was known (100% Cr(VI), 10 mM) and known to be homogenous. Sample thickness was uniform (sample cell comprised of Si_3N_4 windows). Transformation of Cr(VI) to Cr(III) was evaluated as a function of temperature and beam intensity by monitoring changes in the pre-edge of Cr μ -XANES spectra. Chromium reduction occurred immediately at all flux densities tested, but was lessened with lower flux density and lower temperature. Kinetics of Cr reduction was similar for the different flux densities tested. Transformations were not observed with a dried sample.

In a separate experiment with frond tissue cross-section from an As hyperaccumulating fern, the chemical speciation was known (100% As(III)-S complexation) and known to be homogenous in ground tissue. At room temperature, however, the first scan indicated a mixture of As(V)-O and As(III)-O, and successive scans showed increasing amounts of As(V)-O complexation with time (Fig. 1); this was an obvious example of beam damage that provides clear motivation for sample cooling. By cooling to $-150^\circ C$, one is able to collect a single scan that is indicative of the known As(III)-S speciation, while successive scans indicate As(III)-O. After scanning >1 hr on the cryostage at $-150^\circ C$, there was no indication of As(V)-O species. A cryogenic sample environment only slowed the transformation from As(III)-S to As(III)-O, although it mitigated the transformation from As(III)-O to As(V)-O.

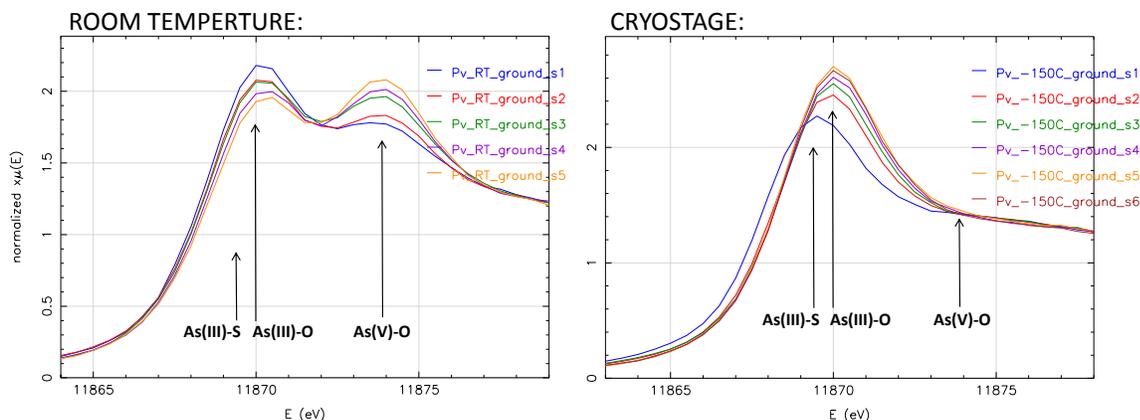


Figure 1. X-ray microanalysis (μ -XANES) of As in ground tissue of frond from hyperaccumulating fern performed at room temperature ($25^\circ C$) and cryogenic temperature ($-150^\circ C$).

***In vivo* optical tweezers-based X-ray elemental imaging of single cellular model organisms**

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Introduction and Objectives

We report on a radically new elemental imaging approach for the analysis of biological model organisms and single cells in their natural, *in vivo* state. The methodology combines optical tweezers (OT) technology for non-contact, laser-based sample manipulation with synchrotron radiation confocal X-ray fluorescence (XRF) microimaging *for the first time*. The main objective of this work is to establish a new method for *in situ* elemental imaging of free-standing living biological microorganisms and single cells in their aqueous environment.

Results and Discussion

Using the microalgae model organism *Scrippsiella trochoidea*, several successful test experiments focussing on applications in environmental toxicology have been performed at ESRF-ID13, demonstrating the feasibility, repeatability and high throughput potential of the OT XRF methodology (Fig. 1-2).

Conclusions

The novel high-throughput OT micro-XRF methodology is very well suited for the *in vivo* elemental micro-XRF analysis of biological organisms and is expected to significantly contribute to the new trend of investigating microorganisms at the cellular level [1,2].

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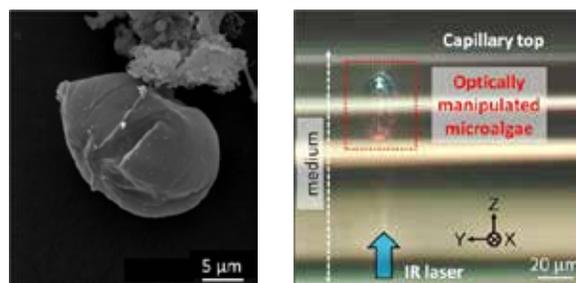


Figure 1: (a) SEM image of a *S. trochoidea* microalgae. (b) Optically levitated microalgae in aqueous environment.

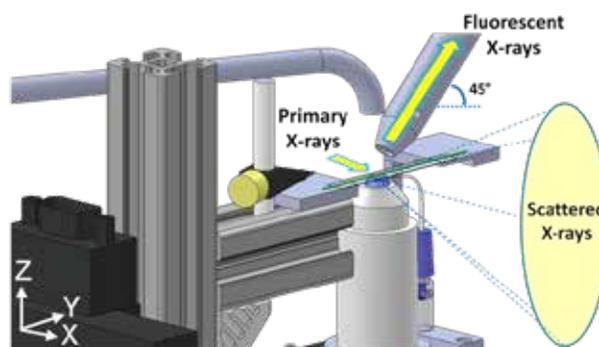


Figure 2: Detail of the OT setup integrated at ESRF-ID13.

Nano-machining for Advanced X-ray Crystal Optics

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We present our recent technological achievements in development of the nano-machining of active surfaces of the X-ray crystal optics [1]. This technique uses a single crystal diamond tool with extremely precise and temperature stabilized positioning system. It has been used for preparation of laser optics elements, however, its application for X-rays with inherently more severe requirements for surface quality is challenging as an alternative to traditional chemical and chemo-mechanical surface finishing. Various simple (flat, spherical, cylindrical) as well as so called free-form surfaces (including aspherical ones) can be prepared. The objective is to prepare high-quality surfaces of the desired shape with sub-nanometer surface roughness and low sub-surface damage of the crystal lattice. The prepared optical elements are to be used for the hard X-ray radiation with the aim to increase diffraction efficiency and intensity throughput either in Laue transmission or Bragg reflection geometry.

The surface parameters of selected nano-machined Ge crystals were evaluated in terms of flatness, local surface roughness and sub-surface damage by means of stylus profilometry, atomic force microscopy, and micro Raman spectroscopy, respectively. The functional diffraction properties of the analyzed crystals were tested using them as optics elements for the X-ray reciprocal space mapping of selected samples by high-resolution X-ray diffractometry. A compromise had to be found between the surface shape precision, local roughness and sub-surface damage at reasonable economy of the whole process.

The final surface roughness of a flat Ge surface down to 0.3 nm (RMS) was achieved for a very slow regime of the processing using feed rates ≤ 0.5 mm/min. At such a slow feed rate, a parasitic surface grating formed due to rastering the diamond tool along the surface was suppressed, the sub-surface damage being in the form of ≈ 30 nm thick amorphous layer. However, slow long-time temperature drifts are crucial in the regime of small feed rates and special measures have to be adopted. We can conclude from our pilot experiments that the diamond tool nano-machining is a promising technology for high-quality surface treatment to replace less homogeneous chemical methods used in the X-ray crystal optics.

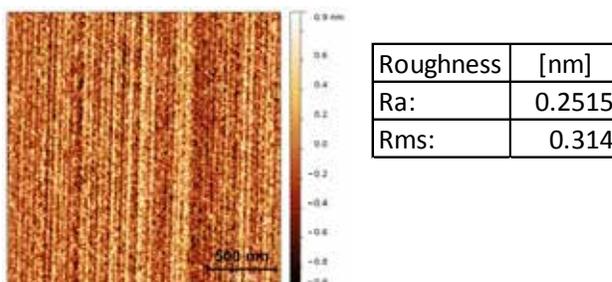


Fig. 1 AFM image of Ge(220) crystal processed by single point diamond turning with 3000 rpm spindle rotation speed, 0.125 mm/min feed rate and 2 μ m depth of cut. (Ra, Rms - linear and areal roughnesses).

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X-ray phase-contrast imaging at Diamond-Manchester I13 Branchline

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Introduction and Objectives

The x-ray microtomography station of Diamond-Manchester I13 Beamline [1] (Diamond Light Source, UK) is located at about 225 m from the undulator source, providing excellent lateral coherence of the x-ray beam. Imaging and tomography can be performed in the 8–30 keV range with a beam of size 15×8 mm². Typical spatial resolutions of 1–5 μm are achievable, depending on the optical magnification of the detector system.

Up until now, inline phase-contrast imaging has been available for users at the I13 Diamond-Manchester Imaging Branchline. Here, we present the first results from this beamline using various other phase-contrast imaging techniques such as grating interferometry [2] and x-ray speckle tracking [3].

Results and Discussion

X-ray grating-based and speckle-based techniques have been successfully implemented at I13. The grating and speckle interference patterns used as a reference for phase retrieval are of high visibility and the acquired images of various soft-tissue samples show excellent contrast at a high resolution.

Conclusions

The results of x-ray grating interferometry and speckle-based imaging at I13 presented here show high potential for these methods to be available for users to perform high-contrast, high-resolution imaging of samples from various fields such as life and materials sciences.

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Correlation between Processing Conditions, Chemical Heterogeneity, and Morphology in Nanofoams for Energy Applications

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Nanoporous materials, fabricated from dealloying method, exhibit unique properties such as high surface-to-volume ratio and a continuous network which enable transport of fuels or electrolyte as desired. A new dealloying method utilizing metallic melt as the dealloying agent leads to the first time successful fabrication of less-noble nanoporous metal such as stainless steels. This largely reduces the cost of the materials while preserving the unique morphological factors that are desirable for energy applications.

Specifically porous air electrode of lithium-air battery and porous supporting structure for gas diffusion layer of fuel cells are two promising applications. We utilized the fluorescence microscopy at Sub-micron Resolution X-ray Spectroscopy beamline and Transmission X-ray Microscope to shed the light on the relationship between underlying kinetics, processing conditions, chemistry and morphology. We also applied scanning electron microscopy and Energy Dispersive X-ray Spectroscopy to study the compositional gradient due to diffusion limit in the dealloying process. Based on the result, we found the relation between morphological parameters such as porosity, tortuosity, surface area, with dealloying reaction temperature and time. Via modeling with these parameters, the influence of morphology, chemistry and processing conditions to battery or fuel cell performance can be established.

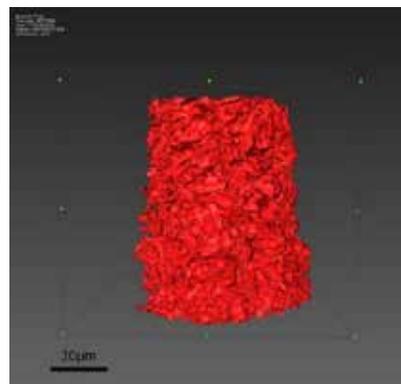


Figure 1. 3D reconstruction of stainless steel

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Imaging properties of 2D-parabolic x-ray compound refractive diamond lens, testing on laboratory setup

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In laboratory condition we tested the x-ray imaging properties of two dimensional parabolic compound refractive diamond lens. The compound refractive lens (CRL) was manufactured using high quality type IIa single crystal grown by high pressure, high temperature methods in TISNCM. The lens consisting of 24 single parabolic plano-concave lenses with apertures of 1 mm and parabola apex radii of 200 μm is arranged in a metallic holder. 2D lens paraboloid profile was processed by laser micro-machining. The surface roughness of paraboloid measured with the atomic force microscopy is about 1 μm . The diagnostic system consisted of the Rigaku 9 kW rotating anode x-ray generator (CuK_α – radiation), the x-ray area detector PIXel^{3D}, 6m optical table, the tungsten mask with the different shape holes, PANalytical parallel beam X-ray mirror for illumination of the mask, the high resolution x-ray emulsion film Fuji IX50. We establish that the CRL transmits the x-ray images with little blurring at the edges at the one-to-one x-ray lens imaging mode. The experimental value of the CRL focal length of 690 mm is slightly different from the calculated (738 mm). A possible reason of this may be related to the surface roughness and different radii of curvature of paraboloids and misalignment from the centre axis of the paraboloids during the assembly. The intensity gain of CRL measured using the 50 μm pinhole is about 2. Unique optical properties of the diamond single-crystal lenses coupled with its excellent thermal properties allows them to be applied as focusing, imaging and beam-conditioning elements at high-heat flux beams of today and future X-ray sources.