MICRO TOMOGRAPHY AT THE IMAGING BEAMLINE P05 AT PETRA III

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Abstract

The Imaging Beamline P05 at the PETRA III storage ring consists of two experimental hutches – a micro CT hutch and a nano CT hutch [1-3]. Both end stations are designed for full field CT with spatial resolutions ranging from 1µm (micro CT) to below 100nm (nano CT) at energies between 5-50 keV. Here, we report on the current status and future plans of the micro CT experiment (figure 1) of the imaging beamline.

Since its start of regular user operation in 2013 the micro tomography end station of the Imaging Beamline P05 focused on delivering high quality absorption contrast tomograms with very high density resolution [4-6]. Generous space around the sample position at the experimental setup of the micro CT experiment allows for the installation of sample environments like furnaces, stress rigs, cryo streams and fluid cells. For many users providing superior density-resolution in absorption-contrast continues to be the most important technique requested at the micro CT hutch. Additionally phase contrast imaging techniques (grating based and propagation based) are implemented and offered to users. In order to address users in need of measuring larger sample series and high-speed tomography a sample-changing robot has been implemented and a fast CMOS detector was installed at P05. Both robot and camera are currently in commissioning and will be available to users soon making P05 a vital analytic tool for a wide range of sciences.

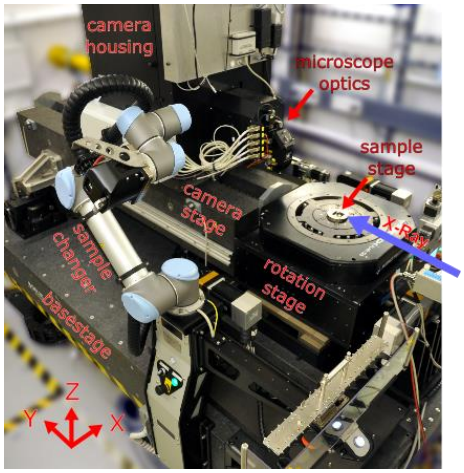

Figure 1: Micro CT setup of the Imaging Beamline P05.

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E-BEAM WRITTEN SOFT X-RAY OBJECTIVE AND CONDENSER OPTICS FOR TXM AND HOLOGRAPHY APPLICATIONS

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Abstract

The optical key components of the microscopes are the zone plates used for imaging the sample. In case of the transmission X-ray microscope (TXM), the condenser should homogeneously illuminate the object and match the N.A. of the objective lens for highest resolution. Today zone plates, capillaries and mirrors are commonly used as condenser optics. We will present the latest status of high N.A. zone plate objectives fabricated for the HZB TXM. Our 100 keV high resolution e-beam lithography system is essential for this work. The high resolution of this system is demonstrated in figure 2.

For table top laboratory TXM´s operating at a plasma source a high N.A. is advantageous not only to match the aperture of the zone plate objective but also to collect the emitted photons from a large solid angle. We manufactured a gold condenser zone plate (CZP) with 4.5 mm diameter, an outermost zone width of 30 nm and 150 nm zone height. Compared to previously reported condenser zone plate optics with an outermost zone width of 30 nm [1], we doubled the gold zone height for improving the diffraction efficiency of the CZP.

Beam-shaping condenser lenses are interesting candidates to generate a square-shaped flat-top object illumination [2] for TXM´s and holography setups for potential FEL single shot applications with coherent beams. We fabricated an x-ray hologram [3] with a smallest period of 100 nm to generate a flat-top illumination. The holographic optic was used in the HZB TXM setup at the U41 undulator source at the BESSY II storage ring. Experimental results of the illumination pattern will be shown.

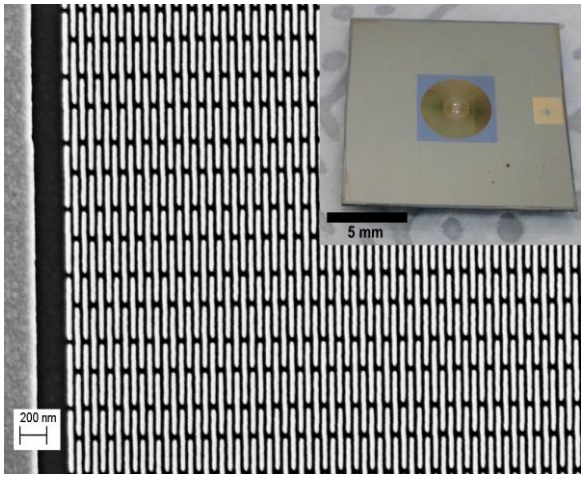

Figure 1: Condenser zone plate with 4.5 mm diameter and 30 nm outermost zone width.

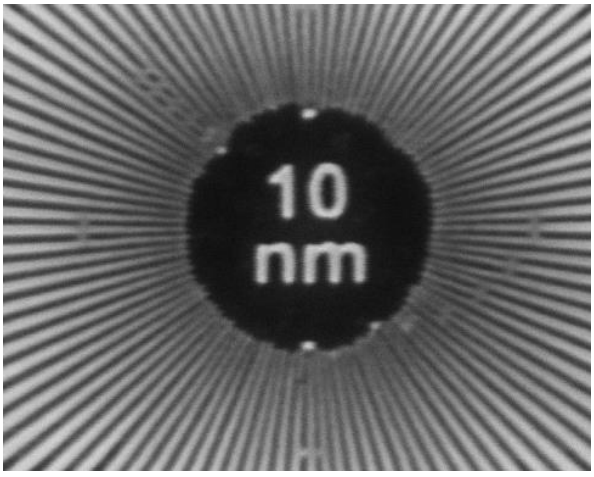

Figure 2: E-beam written Siemens-star pattern. Developed HSQ layer with 10 nm minimum line width.

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SUB-20 NM RESOLUTION IMGING WITH MLL NANOFOCUSING OPTICS: CHALLENGES AND OPPORTUNITIES

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Abstract

The Hard X-ray Nanoprobe (HXN) beamline at NSLS-II has been designed and constructed to enable imaging experiments with unprecedented spatial resolution and detection sensitivity [1]. The HXN X-ray Microscope is a key instrument for the beamline. It provides a suite of experimental techniques which includes scanning fluorescence, diffraction, differential phase contrast and ptychography utilizing Multilayer Laue Lenses (MLL) and zoneplates (ZP) as nanofocusing optics [2,3]. During this presentation, the development and commissioning phases of the instrument will be reviewed; requirements to routinely achieve sub-20 nm imaging resolution will be summarized. We will discuss our approach to fabrication of compound 2D MLL optics, and present the first results demonstrating sub-20 nm resolution.

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NANOTOMOGRAPHY ENDSTATION AT THE P05 BEAMLINE: STATUS AND PERSPECTIVES

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Abstract
The Imaging Beamline IBL/P05 at the DESY storage ring PETRA III, operated by the Helmholtz-Zentrum Geesthacht, has two dedicated endstations optimized for micro- and nanotomography experiments [1-3]. Here we present the status of the nanotomography endstation, highlight the latest instrumentation upgrades and present first experimental results.

In particular in materials science, where structures with ceramics or metallic materials are of interest, X-ray energies of 15 keV and above are required. Even for sample sizes of several 10 µm in diameter lower energies are often not feasible. The P05 nanotomography instrument is dedicated to materials science and is designed to allow for imaging applications with X-ray energies of 10 to 50 keV. In addition to the commissioned full field X-ray microscopy setup the highly flexible layout of the endstation allows switching to cone-beam configuration. Kinematics for X-ray optics like CRLs, Fresnel zone plates, beam-shaping optics are implemented and a KB-mirror system can also be installed into the optics hutch. Altogether this leads to a high flexibility of the nanotomography setup such that the instrument can be tailored to the specific requirements of the sample system.

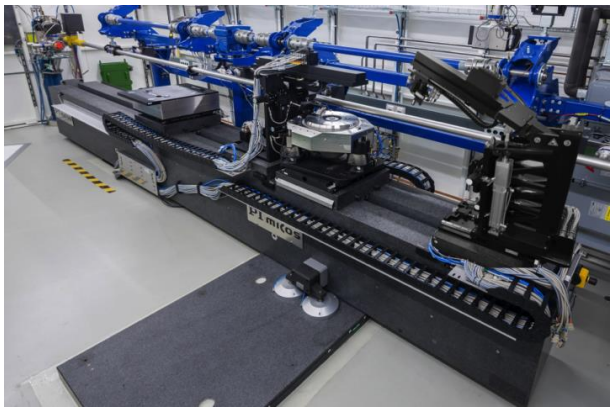

Figure 1: Nano tomography setup at the Imaging beamline IBL/P05 at PETRA III.

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FOCUSED ION BEAM MICROMACHINING OF X-RAY OPTICS

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Abstract

The Ion Beam Lithography (IBL) has been considered for X-ray optics fabrication at least for a couple of decades [1, 2], but only recently, IBL gained more attention and success [3-6]. This is in part due to the developments and now wide-spread use of the ion beam instrumentation technology [7]. Improvements in the processes and the material systems further contribute to this new found interest in the X-ray optics community. Here, we report on the latest improvements we have achieved in terms of reduction in fabrication time, high yield manufacturing and improvements in the imaging performance.

We will also discuss the applications of the direct-write rapid IBL, such as fabrication of large arrays of high resolution micro-FZPs. In Figure 1, a 9×9 array of 81 FZPs, fabricated directly in gold/Si_3N_4 bilayer membrane, is shown. Each FZP has an effective Δr of 30 nm and the whole array is directly written on the membrane region in an overnight process. The outermost zones of the last FZP, that was dubbed the FZP_{99} (81^{th}), is shown in Figure 2. The overall quality remains high until the end of the milling process, demonstrating the beam stability. Such arrays can be used for one-shot focusing in free electron lasers, synthetic aperture imaging or for instance, in zone plate array lithography applications.

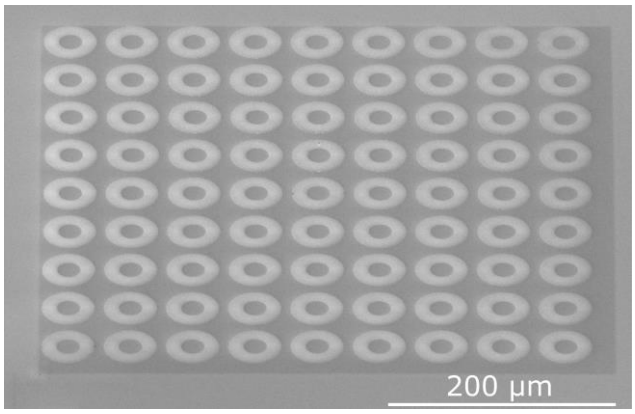

Figure 1: An SEM image of a high resolution FZP array composed of 81 individual lenses. Viewed under 52˚ tilt.

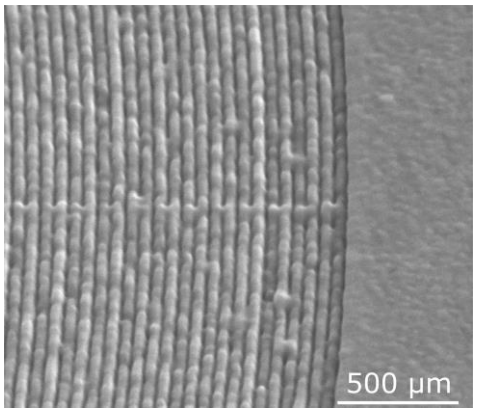

Figure 2: An SEM image shows the outermost zones of the FZP_{99}, the 81^{th} FZP to be written. Viewed under 52˚ tilt.

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PREPARATION OF HIGH QUALITY MULTILAYER ZONE PLATES FOR SUCCESSFUL HARD X-RAY MICROSCOPY

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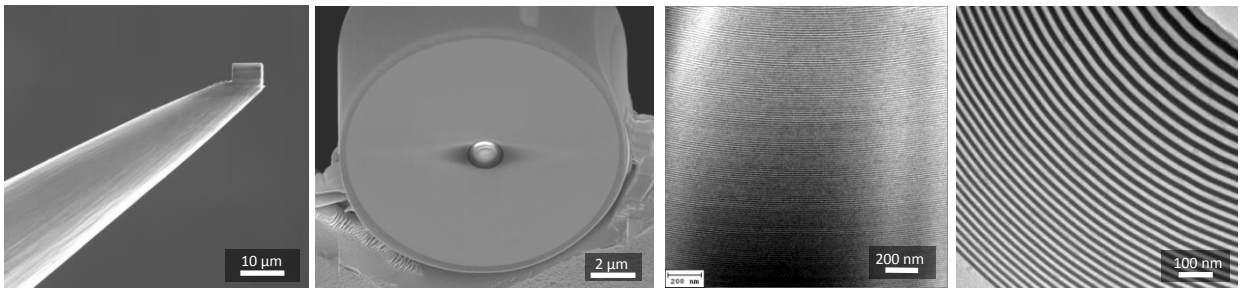
Abstract
In order to realize high resolution microscopy in the regime of hard x-rays, zone plates with both thin outermost zone widths and large optical thicknesses are essential. In this regard multilayer zone plates (MZP) present a promising alternative to conventional Fresnel zone plates as extraordinary aspect ratios are possible. For that multilayers are deposited onto a rotating wire and from this a lens with the desired optical thickness is cut out.

In 2013, we demonstrated unprecedented sub-5 nm point focusing of hard x-rays (at 7.9 keV) by the combination of a high gain Kirkpatrick-Baez (KB) mirror system and a high resolution W/Si MZP [1]. This design study proofed the enormous potential of the combination of pulsed laser deposition (PLD) and focused ion beam (FIB) for MZP preparation [2].

Despite cumulative smoothing effects during deposition and the non-destructive lens cutting process, the W/Si-MZP's quality suffered from sufficient but comparatively low efficiency (2%). By improving the MZP's material system we are now using Ta2O5/ZrO2 MZP which have been already proven to be a system of high potential in the first experiments at 18 keV [3]. By establishing a tilting geometry applying pulled glass wires the efficiency could be increased to 8,4 % (at 7,9 keV) while using an outermost zone width of only 5 nm. In the very first transmission x-ray microscopic experiments clearly 50 nm features could be resolved (evidently only limited by vibrations and drift in the experimental setup so far).

Here we present the underlying considerations and mechanisms concerning the MZP preparation on our way towards high quality MZP as well as the latest results proving the capability of our MZP for hard x-ray microscopy in the sub 10-nm regime.



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ZONE PLATE DEVELOPMENT: PARTNERSHIP BETWEEN APPLIED NANOTOOLS AND THE CANADIAN LIGHT SOURCE

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Abstract

This presentation describes the results of a collaboration between Applied Nanotools Inc. (ANT, www.appliednt.com) and the Spectromicroscopy (SM) beamline [1] at the Canadian Light Source (CLS), for the development of high resolution and efficiency zone plates, for soft and hard X-ray imaging. Scientific programs that use the ambient and cryo- Scanning Transmission X-ray Microscopes (STXM) at the SM beamline require high resolution and high efficiency zone plates, for spectromicroscopy of radiation sensitive materials; for studies at higher photon energies (up to 2.5 keV, and eventually 4 keV) and for ptychography. Figure 1 presents an SEM image of an ANT zone plate optimized for 1 keV, with 17 nm outer zones.

The SM beamline has developed a process for testing zone plates (ZP), including microscope assisted ZP mounting, determination of ZP parameters, and performance tests with a focus on measuring absolute ZP efficiencies as well as spatial resolution checked with an ANT calibration standard. These measurements provide feedback and a direct comparison to theory on process method and design parameters such as ZP diameter, resolution, zone metal and thickness, and silicon nitride membrane thickness, etc. Figure 2 presents representative data for the efficiency of an ANT zone plate (17 nm outer zone width, 270 μm diameter, 130 nm Au zone thickness), with and without central stop, in comparison to theoretical predictions of the efficiency [2]. These results show a very good consistency between measured and theoretical predictions of efficiency.

At the time of this submission, soft X-ray zone plates with 17 nm outer zones have been demonstrated with efficiencies of 8% and high spatial resolution.

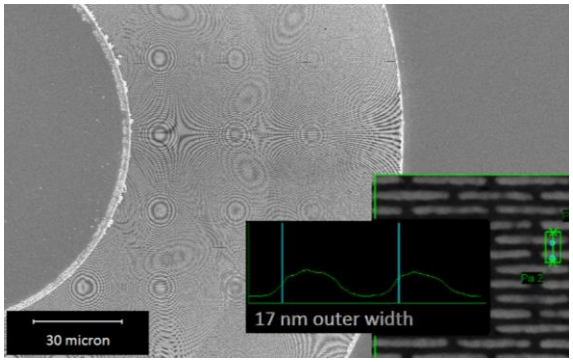

Figure 1: SEM image of a zone plate with 17 nm outer zones for soft X-ray focusing and a 2 μm thick gold central and outer stops.

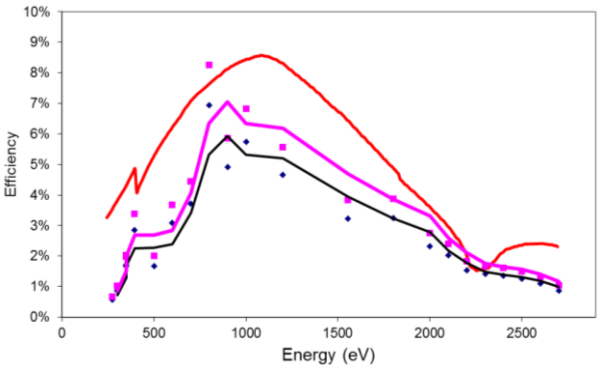

Figure 2: Comparison of the theoretical (red) and measured zone plate efficiencies with a central stop (black) and without a central stop (purple). Theoretical efficiency includes absorption from the substrate (50 nm SiN and seed layer).

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FULL FIELD X-RAY NANO-CT AT SSRF

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Abstract

Full-field X-ray nano-CT is one of the most powerful tools for in-situ, non-destructive observation of the inner structure of objects with high resolution. Many X-ray nano-CT systems have been constructed in the world and have realized 30nm spatial resolution with 20 microns field of view(FOV). Based on the user operation experiences of years at SSRF X-ray imaging beamline, we get to know that lots of user experiments will rely on X-ray full-field nano-CT with big FOV and 100nm scale resolution. A full-field X-ray nano-imaging system has been designed and constructed at the Shanghai Synchrotron Radiation Facility (SSRF)[1]. This microscope is based on a beam shaper and a zone plate using both absorption contrast and Zernike phase contrast, with the optimized energy set to 10keV. The first experimental results of the full-field X-ray nano-CT system based Equally Sloped Tomography(EST) developed at SSRF X-ray imaging beamline (BL13W1) are reported. 3D imaging of tantalum particles was reconstructed by EST with 128 projections. Spatial resolution of 100 nm has been achieved calibrated by a Siemens star pattern in absorption mode. The developed system has a FOV of 50 microns and is ready to be opened to users.

And a dedicated full field X-ray nano-imaging beamline based on bending magnet will be built in the SSRF phase-II project. The beamline aims at the 3D imaging of the nano-scale inner structures. The photon energy range is of 5-14keV. The design goals with the FOV of 20 microns and a spatial resolution of 20nm are proposed at 8 keV.

Figure 1: Nano -imaging system at SSRF BL13W1 experimental station

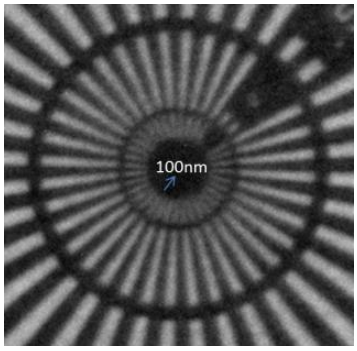

Figure 2: The imaging of resolution target

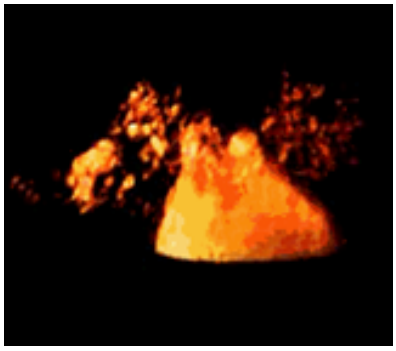

Figure 3: 3D imaging of particles based EST

Reference

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PREPARING FOR HARD X-RAY MICROSCOPY WITH MZPS

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Abstract

Thanks to high quality Fresnel Zone Plate (FZP) optics suitable for soft x-rays, microscopy in the water window has emerged as a powerful imaging technique. In comparison, hard x-ray microscopy cannot truly benefit from its promising features: long penetration depth, even smaller wavelength, and core shell sensitivity for high-Z atoms. The other side of the coin: focusing of hard x-rays was limited for a long time to many thousands wavelengths.

Novel fabrication techniques enlarged the numerical aperture by one order of magnitude over the last two decades. One promising kind of optic are Multilayer Zone Plates (MZPs), which are the hard x-ray analogue to FZPs: diffractive optics with outermost zones well below 10 nm, but with an optical thickness which is inaccessible with e-beam lithography.

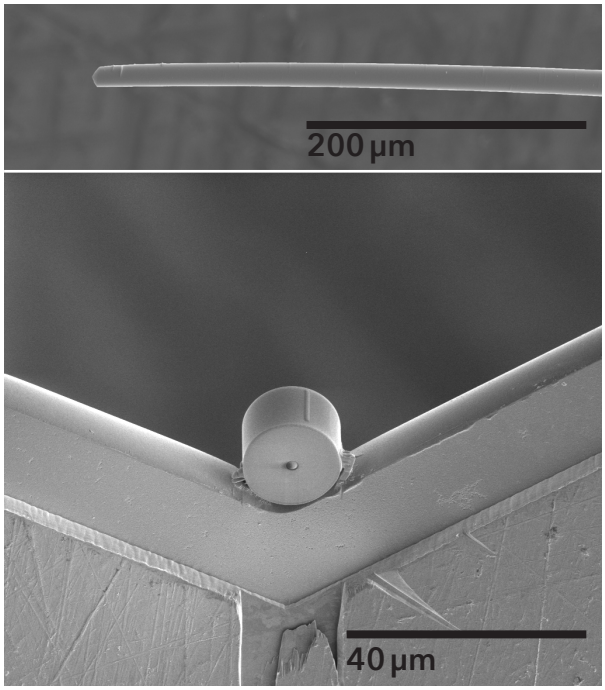

Figure 1: Top view and front view of large Multilayer Zone Plate for hard x-ray imaging.

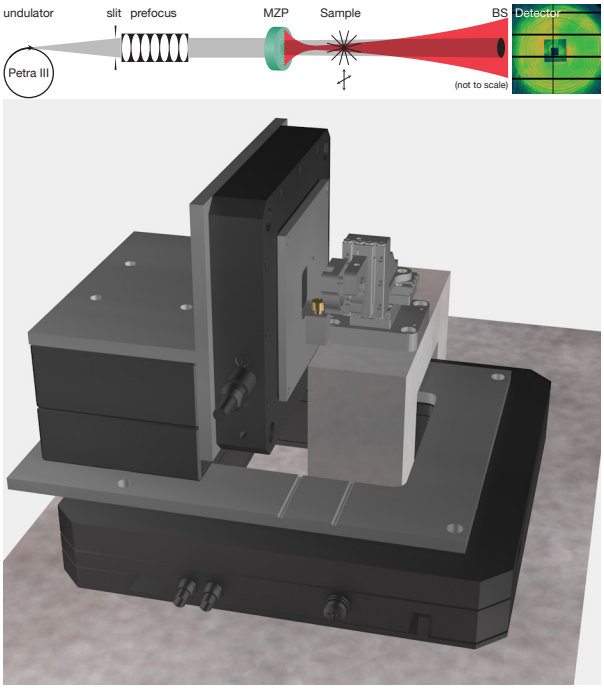

Figure 2: Experimental scheme and new optics–sample tower for the GINIX set-up.

Pulsed Laser Deposition, for example, allows for high-resolution MZPs with outermost layers of 5 nm and smaller, but now for optical thicknesses of 10 µm and more. These lenses are thus promising candidates for high resolution imaging optics on the 5 nm scale, even at high x-ray energies of 12 keV and beyond.

Here we show first experiments using MZP optics for conceptual imaging experiments at 8 to 18 keV in different geometries, including scanning and holographic experiments. With recent improvements in the fabrication, we also obtained transmission x-ray microscopy at 8 keV, fully resolving 50 nm features in nano-porous silicon. Furthermore, we present commissioning results of a new combined optics–sample tower to reduce vibrations and drift.

References

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HARD X-RAY SCANNING NANOPROBE WITH NM RESOLUTION

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Abstract

NANOPROBE, a joint technology research center (JTRC) project between SOLEIL and MAXIV Synchrotrons, serves to equip the NANOSCOPIUM [1] and NANOMAX [2] beamlines, respectively. An ensemble of a Fresnel zone plate (FZP) stage and sample scanning stage, built to specification and employing a modular design is presented. State-of-the-art techniques and nm-focused x-ray beams necessitate both nm-level long term stability and dynamic scanning accuracy. The FZP stage is designed to host two independent zone plates with full control of 5 degrees of freedom and exceptional long term stability (~8h). This was achieved by ensuring ultimate passive stability characteristics as well as active interferometry-based feedback for slow drift corrections.

The sample stage performs continuous scans (flyscan [3]) along the horizontal axis combined with step scans along the vertical axis with 20 nm accuracy at speeds of 5 µm/s over several hundreds of µm^2. Overview scans (2x2 mm) are performed at 100 µm/s with 100 nm position accuracy. This was achieved using dynamic position corrections by taking into account known repeatable positioning errors as well as interferometric feedback.

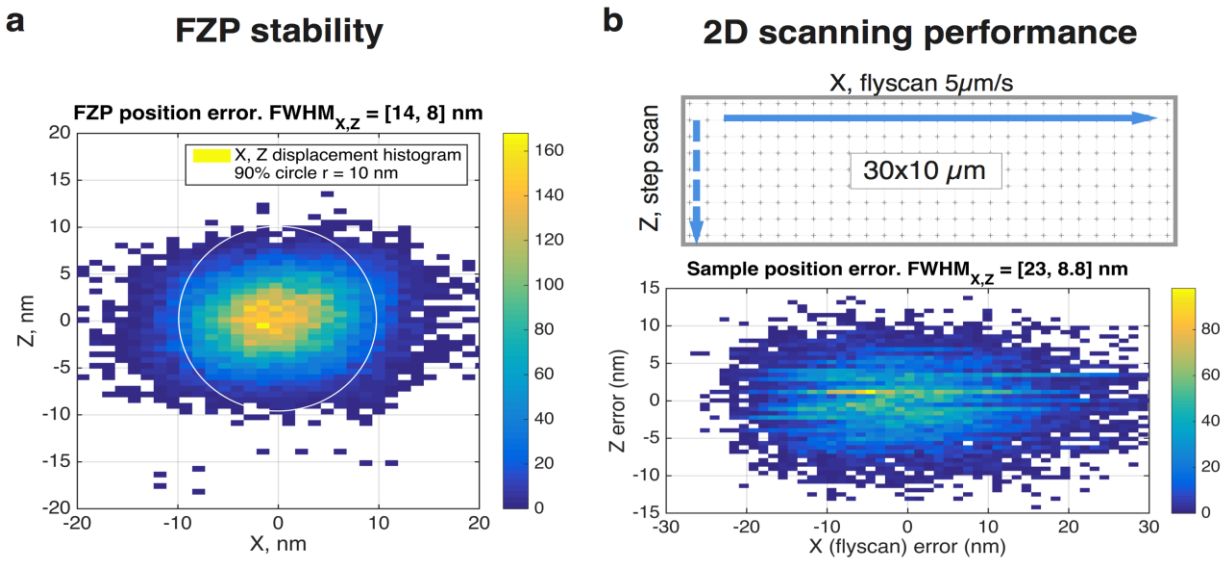

Figure 1: (a) 2D histogram, showing the displacement of the FZP stage with respect to the reference frame during 11 hours. 90% of time the position error is within +/- 10 nm. (b) 2D histogram showing sample position errors during the 2D scan. The scan range was 30x10 µm and the horizontal axis (X) was in continuous motion within each line (flyscan) [3]. The position errors were measured using laser interferometry.

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A THREE-MATERIAL MULTILAYER LAUE LENS WITH REDUCED INTERNAL STRESS

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Abstract

Multilayer Laue lenses (MLL) are promising optics for focusing hard x-rays to a few nanometers. [1] Although spot sizes and resolutions in the region of 16 nm (1D) and 30 nm (2D) have already been demonstrated using MLL, smaller spot sizes and significantly larger efficiency at high photon energies are important and feasible. This is especially true for lenses with a small focus and reasonable working distances, the distance from the order sorting aperture to the focal plane. Smaller spot sizes can be achieved with higher numerical apertures; reasonable working distances of several millimeters make it necessary to manufacture lenses with an aperture of several tens of micrometers. For an MLL the aperture is the deposited multilayer stack height.

It is therefore necessary to manufacture MLL stacks with thicknesses ranging from several tens to about a hundred micrometers, corresponding to depositing a total of several thousand to more than ten thousand. Inherent stress can cause delamination and cracking of the substrate during or after the deposition process for thicknesses of more than 50 micrometers. We have therefore developed a multilayer system that exhibits low stress allowing us to achieve significantly thicker multilayers without delamination. Molybdenum and Silicon are used as absorber and spacer, respectively. Additionally a transition layer is deposited (see Figure 1 (a)). The thicknesses of the layers can be chosen to reduce stress significantly, and a lowest stress design depends on the specific design of the stack [2]. Figure 1 (b) and (c) show a lamella made from such a multilayer stack from two different views.

Calculations have shown, that the expected efficiency is very similar to a Mo/Si bilayer design and significantly exceeds the efficiency of tungsten based material combinations for x-ray energies between 10.5 keV and 20 keV [3]. For 12 keV an efficiency of ~ 40% was measured at beamline APS/1-BM with a flat MLL of the present design.

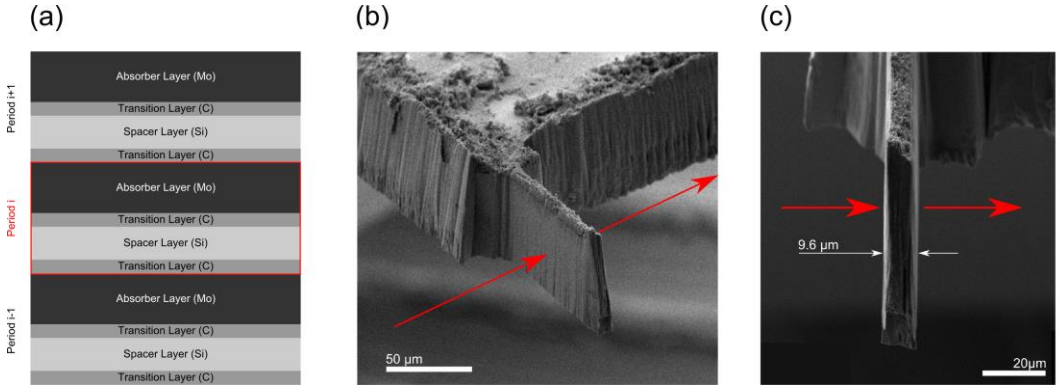

Figure 1: (a) Scheme of the three-material system. (b) MLL lamella section connected MLL bulk material (c) MLL lamella side view with section thickness.
The red arrows indicate the possible x-ray direction in (b) and (c).

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NANOSURVEYOR 2: A COMPACT INSTRUMENT FOR NANO-TOMOGRAPHY AND SPECTROSCOPY AT THE ADVANCED LIGHT SOURCE

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Abstract

The Advanced Light Source is developing a compact tomographic microscope based on soft x-ray ptychography for the study of nanoscale materials [1-3]. The microscope utilizes the sample manipulator mechanism from a commercial TEM coupled with laser interferometric feedback for zone plate positioning and a fast frame rate charge-coupled device detector for soft x-ray diffraction measurements [4]. Utilizing a unique mechanism for simultaneous scanning of the zone plate and order sorting aperture, the microscope can achieve point-scan rates around 100 Hz, including motor move, data readout and x-ray exposure, with a positioning accuracy of better than 2 nm RMS. The instrument will enable the use of commercially available sample holders compatible with standard transmission electron microscopes thus allowing easy sample exchange between x-ray or electron based instruments. This instrument is a refinement of a currently commissioned instrument called The Nanosurveyor, which has demonstrated a resolution of 5 nm in two dimensions and 11 nm in three. Once moved to the new Coherent Scattering and Microscopy beamline it will enable spectro-microscopy and tomography of energy materials with wavelength limited spatial resolution.

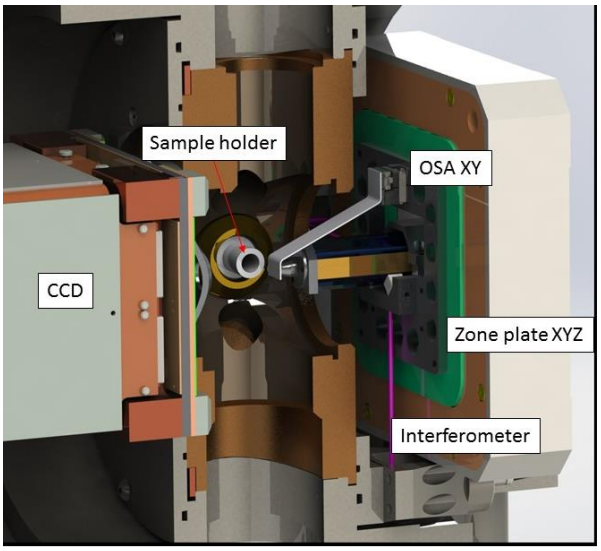

Figure 1: Nanosurveyor 2 ptychographic microscope showing the sample stage, zone plate scanning mechanism, order sorting aperture, interferometer and ALS fastCCD.

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GOING ROUND IN CIRCLES, IN SEARCH OF THE PERFECT ZONEPLATE

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Abstract

Zone Plate fabrication is a multifaceted and complex process, broadly of two distinct stages. The first stage is the creation of the pattern in some kind of resist, and the second stage, is the transfer of the pattern in the desired material. Although a Zone Plate pattern can be generated holographically, most people use Electron Beam (E-beam) lithography, which is generally more controllable, and possibly more accurate depending on pattern dimensions. There are also two different methods of pattern transfer; *Additive*, and *Subtractive*. The main Additive method is *Electroplating* whereby the resist is used as the mould to create the pattern in a metal such as Nickel or Gold. In the Subtractive method, the resist is used as the first in a multilevel mask arrangement, which is used to create grooves in the desired material, already pre-coated on a substrate, (usually SiN). This is achieved with carefully controlled Reactive Ion Etching techniques (RIE).

At ZonePlates.com, we specialize in the use of *Tungsten* as the absorber material. Tungsten is by far the best metal for this task, with superior thermal and elastic properties, perfect for the creation of efficient and stable structures. However, it is a well known fact that Tungsten is a very difficult metal to deposit in a controlled way, and thick stress free Tungsten films are very difficult to achieve!

We also create our patterns in a different way to most fabricators. This is in order to reduce the effect of field distortions (static or dynamic), which determine the accuracy of the pattern, and ultimately the quality of the focal spot. We use a "sector at a time" approach, which has many advantages to list here, and in addition allows for the easy formation of n-focal optics, where each of the 10-200 sectors can be created to focus anywhere in the X/Y/Z plane. Fig.1 shows as schematic of this approach, and fig.2 shows a Partial ZP created in this way.

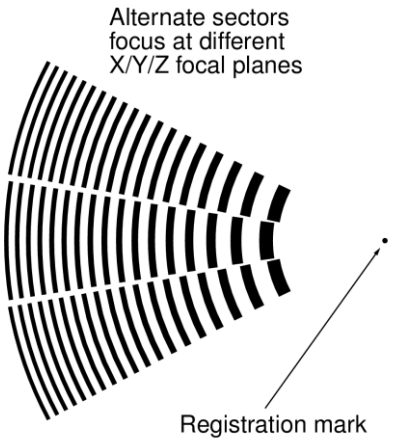

Figure 1: The principle of "sector at a time" approach for the creation of ZonePlate patterns. In a normal ZP, all sectors focus at exactly the same X/Y/Z space, but could easily be made to focus at different positions if necessary.

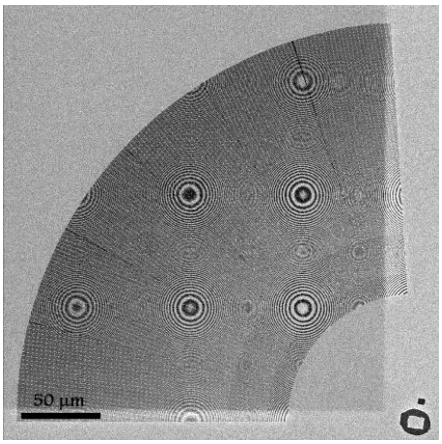

Figure 2: Quarter ZP (6 sectors), D=500 µm. drn=50 nm. The location of the focal spot is off window at low right of the image, eliminating the need for a Central Stop (soft X-Rays).

Despite the fact that we have been involved in ZP fabrication for many years, we are constantly re-visiting and updating our techniques in all aspects of the fabrication process. This is usually driven by new "evidence" that occasionally come to light, and helps in the solution of long standing "mysteries" that have been awaiting attention for many years.

MEDIUM ENERGY MICROPROBE ENDSTATION AT CANADIAN LIGHT SOURCE

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Abstract

Chemical analysis at the microscopic scale (1-100 μm) is often required for many fundamental and industrial applications. Microprobe XAFS is a popular method to meet these requirements. Kirkpatrick-Baez (K-B) mirrors are normally used to realize the micro-focusing, commonly for energies above 5 keV. The soft X-ray microcharacterization beamline (SXRMB) at the Canadian Light Source uses a bending magnet source and covers an energy range of 1.7 – 10.0 keV. It is designed to have a resolving power of 10^4 and a flux of 10^{11} photons/100mA/Sec for bulk XAFS and high energy XPS. The microprobe endstation at the SXRMB has been designed to operate both under vacuum and under inert gas environment for the usually difficult medium-energy region (2-5 keV). A high resolution and large area CCD camera is equipped to obtain optical image of sample. A 4-element Si drift detector is used for mapping in fluorescence mode and TEY mapping is also possible. The initial commissioning result shows that the beam spot can be focused down to approximately 10 μm × 10 μm and the flux is 10^8 - 10^9 photons/100mA/Sec at beam energy of 2200 – 8500 eV.

The microprobe capability at the SXRMB was commissioned and the general user application started in 2014. In this presentation, the initial applications of both the mapping and micro-XAFS in focusing on the elements in the medium energy range, such as Si, P, S and Ca will be presented.

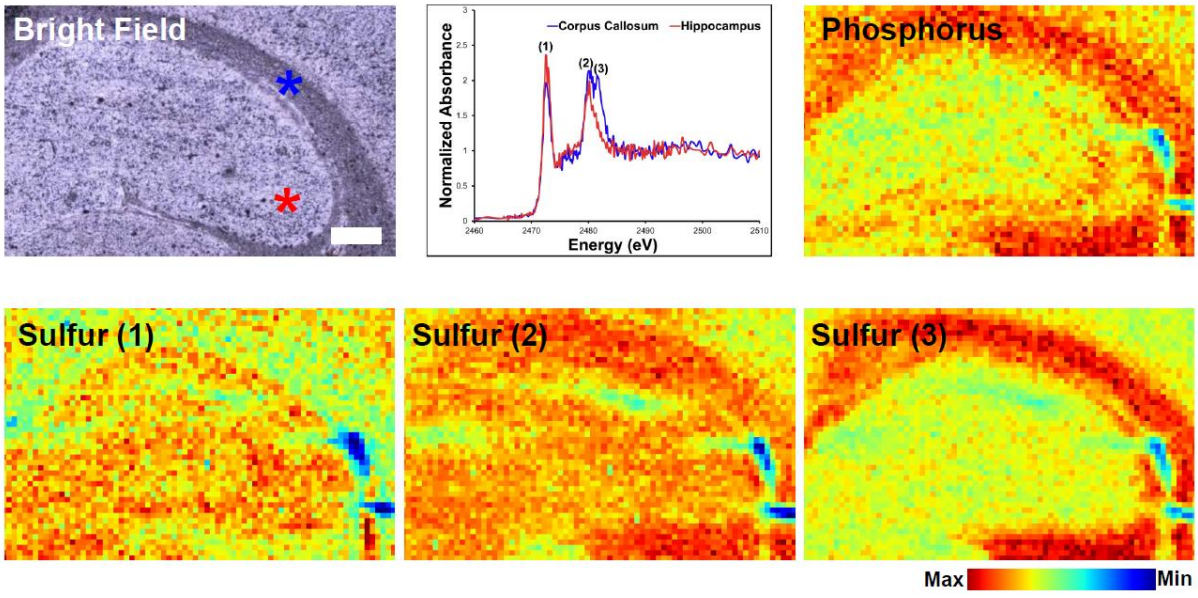

Figure 1: Chemical specific imaging of the anatomical differences in sulfur speciation within the rat brain (hippocampus) (Courtesy of Mark Hackett).

COATED YAG:Ce SCINTILLATOR FOR EFFICIENCY IMPROVEMENT IN INDIRECT X-RAY DETECTOR

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Abstract

Scintillator YAG:Ce is grown and manufactured to convert X-ray to visible light in indirect X-ray detectors. To improve the efficiency of the detector, the scintillator whose thickness is 30 μm and diameter is 25mm, was coated with Ag of 100nm thick at the X-ray incidence side and SiO2 for anti-reflection coatings at the back side. The efficiency and the contrast of the detector was evaluated at SSRF BL13W beamline. Compared to the case of the uncoated scintillator, the count of the detector with coated YAG:Ce scintillator is increased by 40% at 20keV, while the resolution and contrast remains undegraded.

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FULL-FIELD HARD X-RAY MICROSCOPY – AN APPROACH FOR PHOTON ENERGIES ABOVE 8 keV

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Abstract
Only few studies have been published regarding full-field X-ray microscopy at comparatively high photon energies above Cu Kα radiation (8.04 keV).There are some challenges that arise at higher photon energies: the reduced brilliance of laboratory X-ray sources, potential technological issues to manufacture X-ray capillary condensers, the decrease of diffraction efficiency of Fresnel zone plates used as objective lenses, and the decrease of numerical aperture, and therefore, of the collected flux using same optics. On the other hand, an advantage will be the possibility to study iron-based and other high-Z materials. Specific applications such as analysis of microchips will benefit from reduced sample preparation, since the silicon wafer becomes more transparent.

We report on the current experimental status of a full-field hard X-ray microscopy approach, which takes advantage of X-ray multilayer mirrors as condenser optics [1] and multilayer Laue lenses as objective lens [2] solving two of the issues listed above. This setup was combined with several laboratory X-ray sources using Cu Kα, Ga Kα and Mo Kα radiation. Future application with synchrotron radiation will be discussed.

This work is partially supported by the German Ministry for Education and Research, BMBF, under the Program "IKT 2020 - Research for Innovations", Project No. 16ES0070, within the frame of the projects „Master 3D“ and „3D-Innopro“.

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HELICAL SCANNING X-RAY CT IN MATERIALS SCIENCE

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Abstract

Helical scanning is an implementation of X-ray computed tomography (XCT) that is particularly attractive for the imaging of samples which are extended in one dimension. This lends itself to a range of non-trivial samples and materials not least including the human body. In materials science, the scanning of fibre-reinforced composite samples is especially important and in geology the standard sample consists of cores which are long cylinders of material. With circular cone-beam scanning - as is standard in most laboratory systems - long samples can be scanned in sections and stitched together afterwards; but this is time-consuming and has the potential to introduce a number of errors. More importantly is the fact that reconstruction of the cone-beam scanners through the use of filtered back projection algorithms, such as the FDK algorithm [1], means that Tuy's condition [2] is only true for the central horizontal plane and the reconstructed data becomes unusable for any quantification close to the extremes of the volume. This is not the case for helical scanning and the reconstructed data is faithful to the original. Cone-beam artefacts are not introduced during the reconstruction as the effects of the cone-beam geometry are avoided as the sample is scanned with a helical trajectory. Tuy's condition is satisfied for the entire sample volume allowing rigorous quantification of the whole reconstructed dataset.

We will show the latest helical XCT results from our FEI Heliscan system [3] with application to materials science. This will include data from fibre-reinforced composite materials and geological core samples. We will show the relative advantages and disadvantages of helical scanning and demonstrate the types of materials and structures that can be scanned. Finally, we will show the implementation of a helical trajectory iterative reconstruction algorithm which shows some benefits for reducing beam hardening artefacts.

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HYBRID PHOTON COUNTING DETECTORS FOR ADVANCED X-RAY IMAGING

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Abstract

PILATUS was the first hybrid pixel counting (HPC) detector available for X-ray data collection at the synchrotron. Its advanced capabilities, noise-free detection with high dynamic range and excellent stability at high frame rates, are essential for superior data quality in all scattering and diffraction experiments. HPC detectors are favored for measurements of the weak scattering signals at high q in SAXS as well as for precisely measuring diffraction patterns around the direct synchrotron beam in coherent diffractive imaging [1]. A world record in 3D resolution of 16 nm has been achieved in X-ray ptychographic tomography using PILATUS [2].

The latest PILATUS3 detectors offer pixel count rates of up to 10 Mcps and high spatial resolution. Point-spread functions of theses detectors have been measured at the PTB laboratory at BESSY II, which show that their spatial resolution essentially corresponds to the pixel size, with negligible cross talk between neighboring pixels [3].

The new EIGER detector series has dramatically extended the opportunities enabled by these HPC detectors in X-ray imaging applications. EIGER features a pixel size of only 75 µm, in comparison to PILATUS3 with 172 µm, more than doubling the q-resolution. The EIGER X 1M detector (Figure 1) allows data acquisition at up to 3'000 frames per second. This significantly increases the speed of scanning SAXS/WAXS and coherent diffractive imaging applications, allowing images to be recorded faster or with higher spatial resolution. The design of the EIGER detector is compatible with medium-vacuum.

In-vacuum detectors enable measurements with ultra-soft x-rays and thus high q-resolution, also giving access to the lowest q data near the beam stop. Moreover they optimize the data quality by removing absorption and scatter caused by air and windows. An in-vacuum PILATUS 1M detector (Figure 2) has been installed at the FCM beamline of PTB at BESSY II for GI-SAXS measurements at energies from 1.75 to 10 keV [4].

These latest detector developments are presented along with experimental results.

Figure 1: New compact EIGER X 1M detector operating at up to 3,000 fps with 75 µm pixel size.

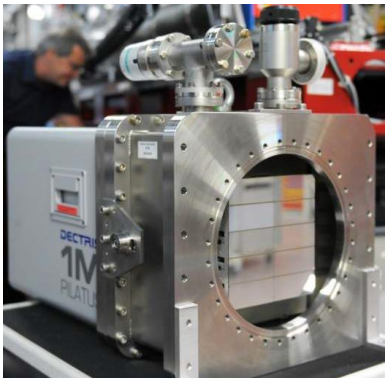

Figure 2: In-vacuum PILATUS 1M detector at the FCM beamline of PTB at BESSY II. Image courtesy of M. Krumrey (PTB).

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HARD X-RAY ZONE PLATES: SIMULATIONS AND FABRICATION FOR HIGH ASPECT RATIOS

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Abstract

Metal-assisted chemical etching (MACE) [1] combined with zone doubling [2] has been shown to be a promising method for the fabrication of Fresnel zone plates for imaging and nanofocusing with multi-keV x-ray beams. We have used a multi-step process which first involves high resolution e-beam lithography, metal deposition and lift-off to produce the metal precursor pattern for MACE. With this process, we have fabricated Si zones with 28 nm width and 2 μm thickness. We then followed this process with deep reactive ion etching (RIE) in order to thin the wafer substrate under the zone plate region to about 10 μm prior to atomic layer deposition for zone doubling.

The optical performance of thick zone plates and multilayer Laue lenses is usually calculated by using coupled wave theory (or related methods) for a fixed grating period, extrapolating the results to the range of grating periods present in an actual optic [3]. These methods serve as the correct reference for tests of an alternative approach. In order to understand arbitrary optical structures, we have explored the use of multislice propagation [4]. We show that the multislice method is able to reproduce the results of coupled wave theory for regular structures, and describe its application to optics with irregular flaws such as the actual fabrication errors that can be present in thick zone plates.

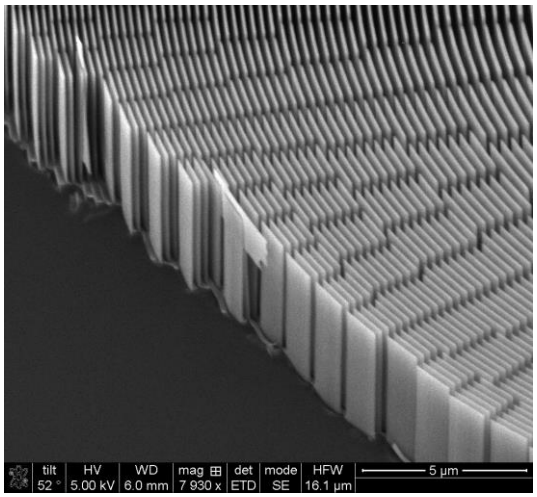

Figure 1: Silicon zone plate fabricated using Metal-assisted Chemical Etching (MACE). This SEM image is of a zone plate with 50 nm zone width on 200 nm period (as required for zone doubling) with a zone height of 3 μm.

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FRESNEL ZONE PLATES FOR NANO-ARPES

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Abstract

Conventional angle-resolved photoelectron spectroscopy (ARPES) instruments usually work with spot sizes well above ten micrometres. However, the local electronic structure becomes of crucial importance when investigating properties of complex materials, size effects or boundary effects with dimensions in the nanometre regime. A way to overcome this issue is to focus the X-ray probe down to dimensions well below one micrometre [1], by using Fresnel zone plates, which are generally capable of resolving structures close to ten nanometres [2].

We present the fabrication of dedicated Fresnel zone plates optimized for photon energies between 50 and 200 eV for use at the nano-ARPES endstation of I05-ARPES beamline at Diamond Light Source. The endstation has been recently commissioned and is taking the first users now. The major challenge is the high absorption of X-rays in this energy regime by many support materials. For photon energies between 50-100 eV, we use extremely thin silicon nitride membranes of down to 28 nm thickness, see Fig.1. The outer zone width of 132 nm and the diameter of 1.5 mm are chosen to provide a suitable focal length for the nano-ARPES end station (8-16 mm at 50-100 eV). The zone structures consist of 200 nm thick HSQ resist, providing a good efficiency over the relevant energy range. Using a layout with interruptions for e-beam writing, we are able to minimize the stress within the material, and avoid mechanical rupture of the thin and fragile support membranes.

First tests show that the probe size of our zone plates is well below one micrometre, see Fig. 2. The first order derivatives of both the transmitted radiation and the electron signal show full width at half maximum (FWHM) values of 0.9 and 0.6 µm at 50 and 100 eV, respectively, when scanned across the edge of a window in a silicon wafer.

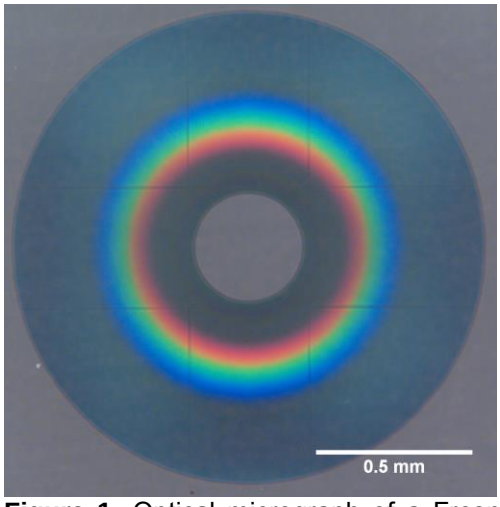

Figure 1: Optical micrograph of a Fresnel zone plate with 132 nm outer zone width and a diameter of 1.5 mm, supported by a 28 nm thick silicon nitride membrane.

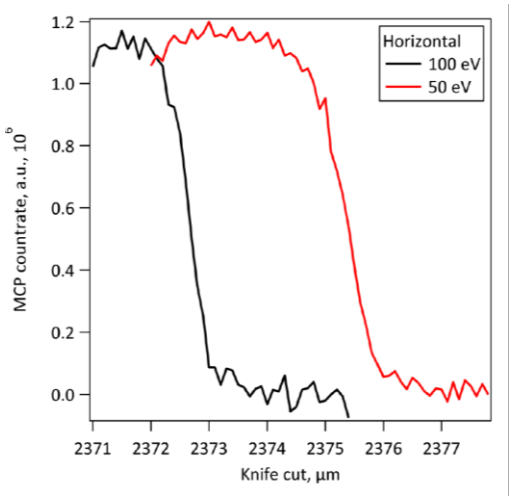

Figure 2: Scanning profiles across a membrane. Gaussian fits of the first order derivative yield FWHM values of 0.6 µm (100 eV) and 0.9 µm (50 eV).

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A HARD X-RAY NANOPROBE AT DIAMOND LIGHT SOURCE

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Diamond Light Source, Diamond House, Harwell Science and Innovation Campus, Didcot, OX11 0DE, UK

Abstract

Beamline I14 is the hard X-ray nanoprobe beamline currently under construction at Diamond Light Source, UK, and scheduled to come into operation in 2017. The beamline will be a dedicated facility for nanoscale microscopy and micro-nano SAXS serving two endstations housed in a new external building approximately 175m from the main synchrotron ring.

The nanoprobe endstation aims to achieve the smallest possible focus (initial aim 50nm) with the capability to exploit future optics developments. The optical design will be optimised for scanning X-ray fluorescence, X-ray spectroscopy and diffraction. The mesoprobe endstation will be optimised to carry out small and wide angle X-ray scattering studies as well as scanning fluorescence mapping with a variable focus beam in the range 5µm – 100 nm.

The beamline will complement electron and optical microscopy and enable new science in a number of areas spanning materials science, biology, engineering and earth science. The I14 beamline facility has also been merged with a new national electron microscopy facility providing 4 electron microscopy suites covering the physical and life sciences. This facility combines staff and expertise from a number of different areas which we believe we will allow us to make exciting progress in sample preparation techniques and correlative x-ray and electron microscopy studies. Here we present the design and key specifications of Beamline I14, and highlight potential applications.

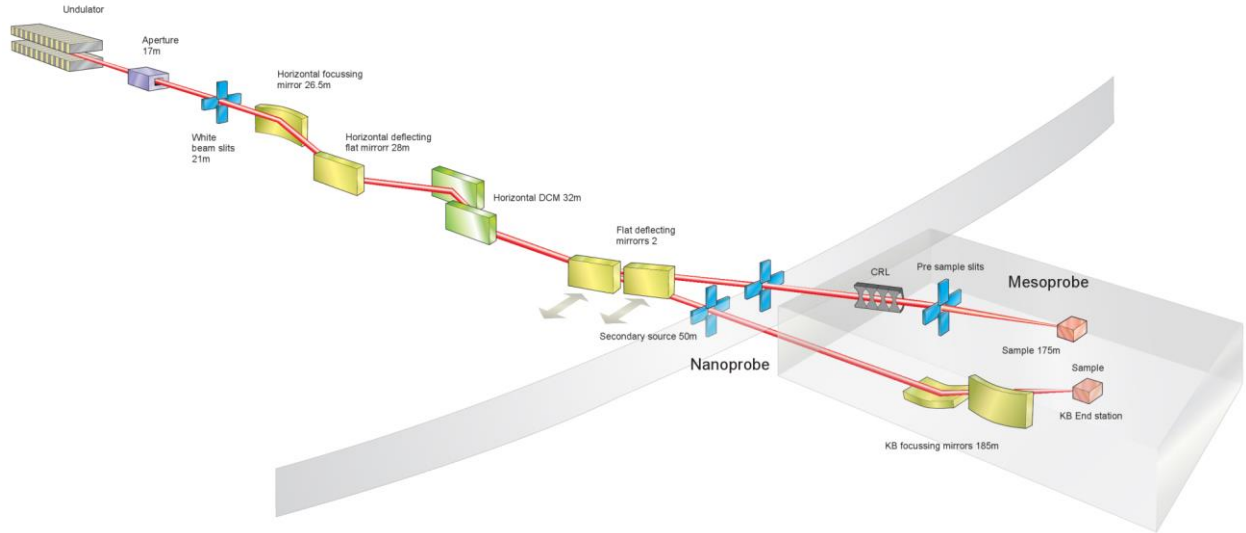

Figure 1: I14 Beamline Schematic

X-RAY IMAGING WITH STRUCTURED ILLUMINATION

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Abstract
We have adapted to x-ray microscopy, the methods of structured illumination microscopy (SIM) that were developed for use in visible-light microscopy (VLM) [1]. In principle this makes it possible to carry out optical sectioning of the image data, where only the in-focus features of the sample are transferred through the optical system, and provides an alternative approach to 3D imaging by combining a through-focal series of 2D image slices.

The optical system of the transmission x-ray microscope (TXM) is analogous to that of a conventional VLM, consisting of a condenser lens to illuminate the sample and an objective lens to image it. The general idea of SIM is that the condenser lens projects a patterned illumination on to the sample and, since the overall resolution of the SIM image is affected by both the condenser and objective, an x-ray SIM requires a matched pair of condenser and objective zone plates, ideally operating at their diffraction-limited resolutions using fully incoherent illumination.

This presentation describes how the TwinMic x-ray microscope at Elettra [2] has been modified to provide a SIM mode of operation for the TXM. The SIM procedure is to illuminate the sample with an intensity stripe pattern as shown in Figure 1. These stripes can be thought of as a carrier wave that is modulated by the conventional-image data. The illumination pattern is then shifted laterally across the sample by a fraction of a stripe period and another similar image is recorded. A SIM dataset consists of a series of such images recorded with the stripes displaced by different shifts. A 2D image slice can then be reconstructed via a straightforward algorithm [1], and a through-focal series of these yields the 3D image.

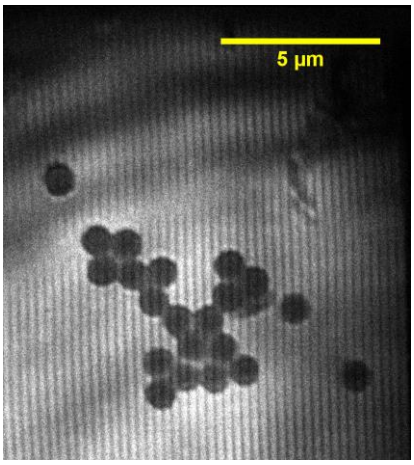

Figure 1: TXM image of 1.09 µm diameter polystyrene spheres taken at a photon energy around 500 eV using the SIM mode of operation on the TwinMic beamline at Elettra.

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CHARACTERISATION OF THE IMAGING AND COHERENCE BEAMLINE I13 AT THE DIAMOND LIGHT SOURCE

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Abstract

I13 is a 250 m long hard x-ray imaging and coherence beamline (6 keV to 35 keV) at the Diamond Light Source. The beamline comprises of two independent experimental endstations: one for imaging in direct space using x-ray microscopy and one for imaging in reciprocal space using coherent diffraction based imaging techniques [1], implementing several novel design ideas, like the Double Mini-Beta Scheme [2], horizontally deflecting super-polished beamline mirrors and horizontally deflecting monochromators [3] to preserve the coherence.

In particular coherent diffraction based imaging techniques are very demanding on the beamline performance and it requires several iterations until a beamline reaches its full potential. Though the final prove of performance is a working experiments, in general this does not identify the components with the largest scope for improvement. To obtain such detailed insight into the beamline characteristics, each component has to be tested and optimized on its own, making use of dedicated optical and physical metrology facilities during the assembly phase of the instrumentation as well as in-situ x-ray based metrology techniques. In addition to that, all aspects potentially impacting on the performance have to be covered. Of particular importance are: the quality of optical components, the mechanical design concept, vibrations, drifts, thermal influences and the performance of motion systems. In this paper we will detail the optimization process at I13, how the different components were characterised and how this contributed to improving the beamline performance in terms of beam-stability and coherence length, which are key-parameters for successful user experiments.

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NEW DEVELOPMENTS IN X-RAY OPTICS TOWARD HIGH EFFICIENCY, SUBMICRON ACHROMATIC FOCUSING

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Sigray, Inc. Concord, CA USA

Abstract

The increasing importance of x-ray microanalytical and nanoanalytical techniques to a broad range of research applications is driving demand for major improvements to x-ray optics used in both synchrotron [1-3] and laboratory [4] settings. Sigray has developed several new paraboloidal x-ray mirror lens designs aimed at substantially improving flux at the synchrotron for submicron focusing and providing unprecedented capabilities in laboratory instrumentation performance in terms of resolution, sensitivity, throughput, and working distance. Applications for these x-ray mirror lenses include straightforward upgrades of existing, lower resolution synchrotron beamlines for microanalytical capabilities and development of new, high throughput laboratory/industrial x-ray techniques such as microXRF, microXRD, and CD- SAXS when coupled to ultrahigh brightness x-ray sources (e.g. Sigray's FAAST™ source, transmission nanofocus sources, laser plasma sources, and liquid metal jet anode x-ray sources).

In this presentation, we will discuss several major advances that have been made to the paraboloidal x-ray mirror lens in terms of figure error and reflective surface coatings. The optics performance will be reviewed, particularly in regard to key optics performance attributes needed for optimization in microanalytical applications, including: transmission efficiency, numerical aperture (NA), FWHM of point spread function, working distance, focus chromaticity, energy bandpass, energy transmission, percent of source brightness preservation, and phase space acceptance.

Such developments will provide tremendous benefits. In terms of synchrotrons, advantages include a 4X flux improvement over KB mirrors and enabling straightforward development of highly flexible beamlines with multiple, interchangeable optics. For laboratory instrumentation, a double paraboloidal x-ray mirror lens design can be used to achieve <8 μm resolution with excellent source brightness preservation, which is far beyond the resolutions of ~25-300 μm of polycapillary and Montel mirror optics. The x-ray mirror lens may furthermore be configured for collimation with extremely small divergences for applications requiring parallel or line-focus beams.

Examples of the unique advantages of the paraboloidal x-ray mirror lenses include that they produce a single, fixed focal spot over a wide band of x-ray energies, unlike non-imaging x-ray optics such as polycapillary x-ray optics (which produce concentric "focal spots" of sizes inversely proportional to x-ray energy). This achromatic nature is essential for enabling high accuracy in microanalytical measurements using polychromatic laboratory x-ray sources, as many well-developed algorithms assume a single focal spot at the sample. In addition, another key benefit of the double paraboloidal x-ray mirror lens is its constant magnification, even for off-axis x-rays, providing significant advantages over optics such as ellipsoidal single-bounce imaging optics.

Future potential developments of the x-ray source and the optics designs will be discussed, including further resolution improvements and low x-ray energy optics capabilities, such as focusing of low keV x-rays for excitation of low Z elements and the L&M lines of higher Z elements.

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IMAGING WITH THE LIQUID-METAL-JET SOURCE: MICRO-CT AND FULL-FIELD MICROSCOPY

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Abstract

Laboratory-based full-field X-ray microscopy is still challenging, as both - the effective aperture of optical elements and the brightness of X-ray tubes - is typically low. With the goal to establish X-ray microscopy for a higher number of users, we have built up a modular setup based on a highly brilliant liquid-metal-jet source, exploiting the gallium K_a-Line at 9 keV [1]. In order to cover a broad range of different samples, the microscope can be operated in two modes.

In the “micro-CT mode”, small pixels are realized with a crystal-scintillator and an optical microscope via shadow projection geometry. Here, samples in the millimeter range can be scanned routinely with low exposure times. Additionally, this mode is optimized with respect to in-line phase contrast.

In the second “nano-CT mode”, a higher resolution can be reached using X-ray optics. By using Fresnel zone plates or compound refractive lenses (made from SU-8 resist by deep X-ray lithography [2]), structures down to 150 nm are resolved at moderate exposure times (several minutes per image).

At this conference, we present the current status of the instrument.

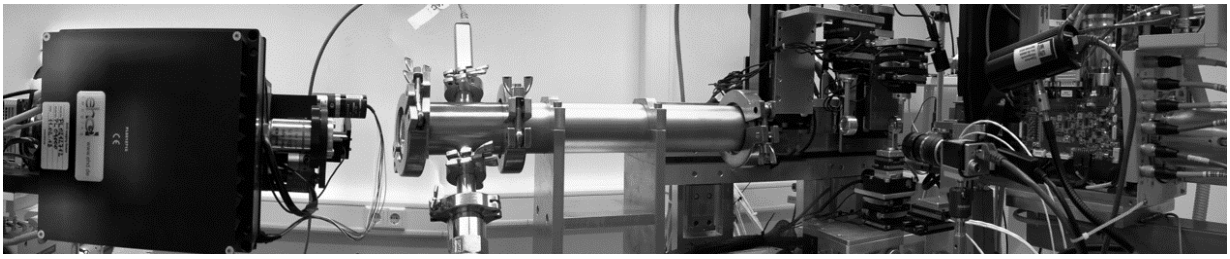

Figure 1: Photography of the setup: detector, vacuum tube, lens, sample, pinhole, beam stop, condenser, and source (from left to right).

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X-RAY FULL FIELD MICROSCOPY AT 30 KEV

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Abstract
X-ray full field microscopy allows transmission imaging of opaque samples, which is often required in material science and microstructure technology. An X-ray full field microscope for 30 keV consisting of illumination optics, imaging lens and detector, has been realised with a length below 5 m. We achieved a resolution better than 200 nm in our experiments at PETRA III, P05 [1]. Figure 1 shows a detail of an X-ray micrograph of a 500 nm thick gold Siemen-star divided by its flat field. The X-ray optical magnification was 26.

As imaging lens, we used a compound refractive lens (CLR) with a focal length of 90 mm and an entrance aperture of 71 µm. The imaging lens was fabricated by deep X-ray lithography out of mr-L negative photo resist [2] at KIT/IMT. Deep X-ray lithography allows fabricating submicron structures with very high precision. In order to reduce vignetting and unequal resolution, we adapted the local apertures of the single lens elements [3].

We choose a rolled X-ray prism lens (RXPL) [4] as illumination optics. The condenser had a working distance of 2.5 m and an aperture of 2 mm. Rolled X-ray prism lenses feature a high transmission and are easily adaptable to their application during the fabrication process. Therefore, this lens was specifically designed for illuminating the entire field of view with a divergence tailored to the angular acceptance of the objective lens. Thus, we achieved a high brightness of the image and consequently a short exposure time.

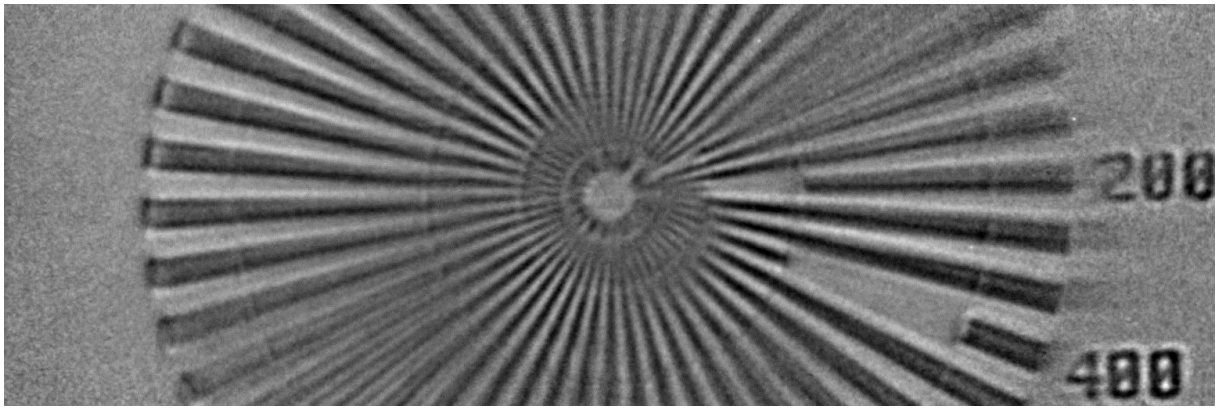

Figure 2: X-ray micrograph of a Siemens-star at 30 keV divided by its flat field (30 µm outer diameter of Siemens star).

Our concept allows building up fast and compact high energy microscopes with high resolution at synchrotron beamlines for ambient conditions. Our theoretical calculations suggest resolutions below 60 nm are achievable after optimizing the fabrication process of our lenses.

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PHASE RANDOMISING SCREENS FOR SOFT X-RAY IMAGING

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Abstract
Increasing the phase diversity of the illumination has been found to improve the quality of image reconstructions in various forms of coherent diffraction imaging (CDI) [1, 2] and ptychography [3], while speckle-based imaging techniques also make use of phase-diverse illumination that is still spatially coherent [4]. When using hard x-ray illumination it is possible to make a random phase screen (RPS) from materials with low atomic number Z, since the complex refractive index $n = 1 - \delta - i\,\beta$ for these materials is such that the ratio $\delta/\beta \gg 1$, and the phase shift imparted to the wave will be more significant than the effects of absorption. At soft x-ray energies the ratio $\delta/\beta \sim 1$ even for low-Z materials, and an RPS produced in a similar way is less useful, since a much larger fraction of the incident illumination is absorbed by the RPS.

A random array of apertures in a uniform, partially transmitting film provides an alternative form of diffuser at soft x-ray energies [5]. If the apertures in the array are identical, and are illuminated by a coherent plane wave, then the diffracted amplitude from each aperture will be the same, except for a phase shift determined by the position of the aperture in the screen plane. For a screen with a large number of randomly positioned apertures the resultant phase of the waves diffracted by the aperture array will vary randomly across the wavefront, so a random pinhole array is a form of RPS. Random pinhole arrays designed for use with soft x rays have been used successfully as test patterns when measuring the modulation transfer function of the optical system [6], and as a diffuser to enhance the diversity of the probe used for ptychography [7].

In this paper we outline how the design of a random pinhole array for use at soft x-ray energies can be optimised to match the angular acceptance of downstream optical elements, and to minimise the contribution from the zero-order diffracted component so that the strength of the phase-diverse contribution to the illumination is maximised.

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MONOCHROMATIC HIGH RESOLUTION X-RAY IMAGING OF PLASMA-LASER PRODUCED USING FRESNEL ZONE PLATE

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Abstract
Monochromatic X-ray imaging at micron scale is a convenient tool for studying the dense plasma produced by laser facilities.We use a microscope made of a gold transmission Fresnel Phase Zone plate (FPZP) who have high spatial resolution capability (1-5 µm) and high efficiency. We explore different zone plates optimized at different photon energies. We present experimental imaging studies of plasma X-ray sources with FPZP's. We show the interest to combine FPZP with a multilayer mirror (ML) which selects a narrow bandwidth. This device allows to choose the imaging wavelength by modifying the focal length and the angle of ML.

We compare the performances of two gold FPZPs with (Figure 1) and without central beam stopper (Figure 2). These lenses have a focal length of 250 mm for 1.835 keV. A complete characterization of ML and FPZP were made at the synchrotron radiation facilities: Physikalisch Technische Bundesanstalt (PTB) laboratory [1] and SOLEIL metrology beamline [2]. Spatial resolution close to the theoretical value of the FPZP is achieved. We explain the difference between experimental results obtained with monochromatic and quasi-chromatic X-ray plasma sources.

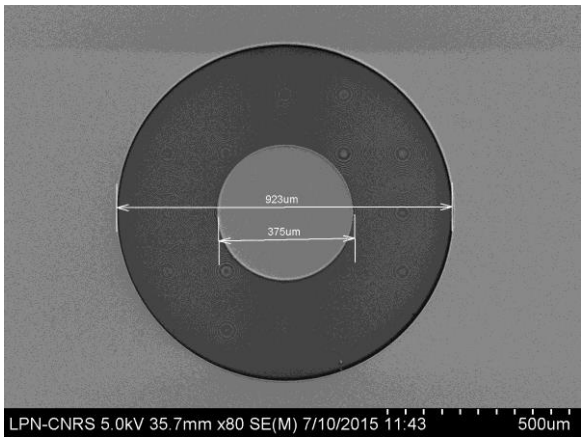

Figure 1: Scanning electron micrograph of the Au zone plate with a center beam stopper.

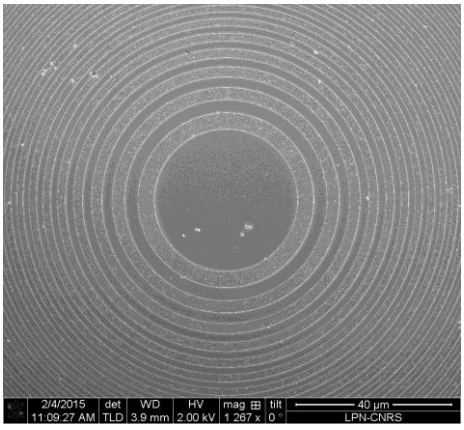

Figure 2: Scanning electron micrograph of the Au zone plate without center beam stopper.

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FIXED TARGET SINGLE-SHOT IMAGING OF NANOSTRUCTURES USING THIN SOLID MEMBRANES AT SACLA

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Abstract

Single shot imaging using femtosecond X-ray pulses from X-ray Free Electron Lasers has unveiled high-resolution structures of nanoparticles and biological specimens [1]. The X-ray pulse power is enough to vaporize specimens when being focused into a few microns area [2]. This makes it essential to have a single particle loader providing fresh samples to each X-ray pulses. Here, we introduce single shot imaging at SACLA to investigate various types of specimens, from metallic nanoparticles to biological macromolecular complexes, prepared on Si3N4 membrane using the MAXIC [3, 4]. Significant reduction in sample consumption is achieved while maintaining the data acquisition rate at 30Hz compatible to the SACLA operation rate at present.

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STRUCTURAL ANALYSIS OF ANCIENT CASTING MOLD FROM SHANG IN CHINA ANALYSED USING SYNCHROTRON X-RAYS

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Abstract

Casting is of symbolic significance and strictly graded in the Bronze Age in China. Fabrication and use of bronze vessels prevailed in Shang dynasty and casting activities mainly distributed in the center of Shang, which indicating a centralization of authority [1, 2]. While it is rare but important for the excavation of a casting mold in the east of Shang. Study of the excavated remains is of great significance to understand the eastward development of Shang culture [3]. Here, we report a unique casting mold with fine decorative patterns excavated from east Shang for the first time. Combining high performance of Synchrotron X-rays and advanced physicochemical analysis methods, nondestructive three-dimensional imaging and high-precision in situ trace-elemental analysis were performed. Considering related casting activity, copper and iron element were particularly analyzed with different methods. The result indicates that the mold had been used for copper casting. Promisingly, using a combination of different X-ray analytical techniques in the study of archaeological molds may contribute to obtain a thorough understanding for archaeologists.

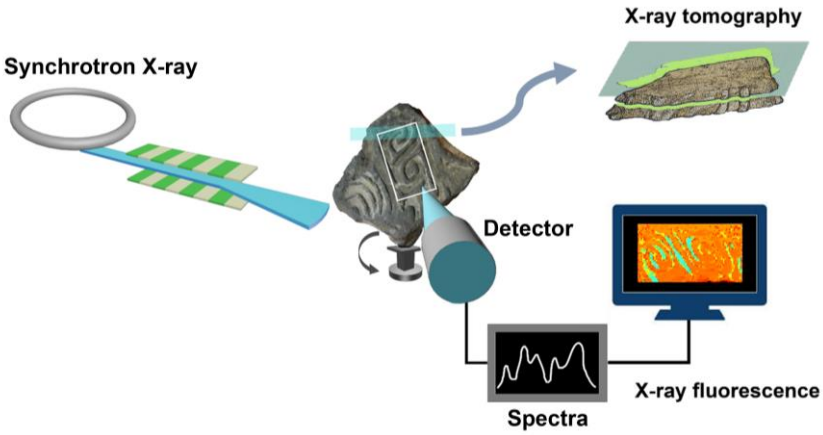

Figure 1: Synchrotron X-ray microtomography and fluorescence imaging of the pottery mold.

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XRF, TEY AND PTYCHOGRAPHIC IMAGING IN STXM CHARACTERIZATION OF BATTERY MATERIALS

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Abstract

Lithium-ion batteries (LIBs) have been drastically growing in applications from portable consumer electronics to large battery electric vehicles. Their performance is critically related to the electrode materials, especially the cathode materials. A deep structural and chemical understanding of the crystalline electrode materials and the interaction between the materials and the electrolyte using advanced analytical tools is not only important to LIB performance enhancement but also crucial to the development of new LIBs for future highly demanding applications.

The scanning transmission X-ray microscopy (STXM) at the Canadian Light Source (CLS) has demonstrated powerful capabilities in characterization of the LIB materials through high spatial resolution X-ray absorption images and the spatially-resolved X-ray Absorption Near Edge Structures (XANES) spectroscopy.[1] However the conventional STXM requires thin sections or fine particles of the sample with thickness/size of tens to hundreds of nanometers, which appears to be substantially challenging for large LIB crystalline materials of a few micrometer size. In addition direct and non-destructive imaging of the LIB electrode assembly is not feasible due to non-transparent in the soft X-ray energy range. To overcome these issues, imaging through energy dispersive X-ray fluorescence (XRF) and total electron yield (TEY) through sample current were implemented in STXM for thick LIB crystalline materials and electrodes, and promising result has been obtained. Furthermore, the newly developed STXM-Ptychography technique has also been applied to characterize LIB electrode materials with unprecedented spatial resolution. These capabilities and applications have significantly enhanced the existing CLS STXM.

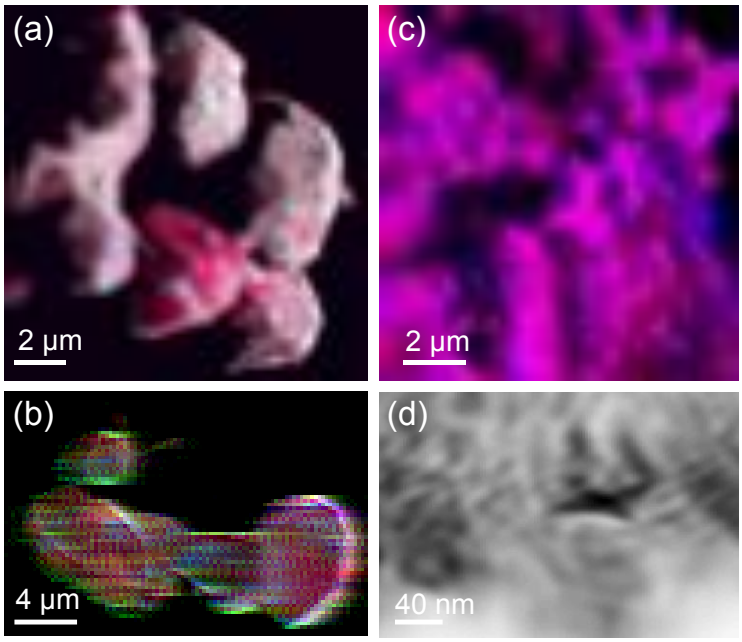

Figure 1: (a) XRF-STXM and (b) TEY-STXM imaging of $LiNi_{0.33}Fe_{0.33}Mn_{1.33}O_4$ battery electrode crystals, Red: Mn(IV), Green: Ni(II), Blue: Fe(III); (c) direct XRF-STXM imaging of $LiCoO_2$ battery electrode, Red: Co(III), Blue: O; (d) STXM-Ptychography of Li_2FeSiO_4 battery electrode nanocrystals.

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IN SITU PTYCHOGRAPHY AND ETEM STUDY: ACTIVATION OF A CU-ZNO@ZSM-5 CORE-SHELL CATALYST

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Abstract

Hard X-ray ptychography now offers a resolution below 10 nm [1] and is therefore regarded as one of the most promising tools to image materials and catalysts in the meso scale (20-500 nm), which is important for heat and mass transfer. It allows probing the catalyst inside a reactor under *in situ* conditions (elevated temperature, gas flow, pressure) and is thus complementary to electron microscopy [2]. This enables hierarchical characterization of catalysts on the different length scales, i.e. the atomic scale by TEM, the meso scale by X-ray ptychography and the macro scale with conventional microscopic techniques [3].

The present study reports on the development of *in situ* cells for both hard X-ray microscopy and its combination with ETEM to study structural changes both from the nm scale to the 500 nm scale on a bifunctional core-shell catalyst. The core consists of a Cu-ZnO/Al_2O_3 methanol catalyst, surrounded by a ZSM-5 shell used for methanol dehydration to dimethyl ether [4]. During activation, CuO in the core is reduced and thereby shrinks [5]. This might affect the stability and bifunctional operation of the catalyst. Hence, we studied the core-shell interface during redox cycles with respect to volume changes of the Cu containing crystallites.

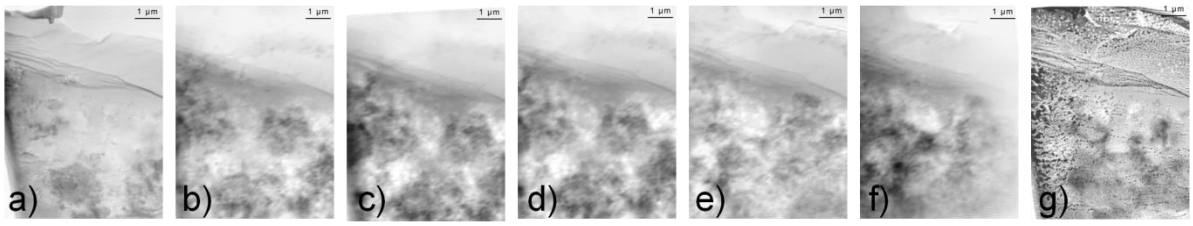

Figure 1: a) Inverted *ex situ* BSE-SEM image (vacuum), b-f) *in situ* ptychograms (phase contrast) b) room temperature in He, c) 250°C in H_2, d) 250°C in O_2, e) 350°C in H_2, f) 350°C in O_2, g) inverted *ex situ* BSE-SEM image after the *in situ* treatment at PetraIII, P06. The scale bar relates to 1 µm.

Figure 1 shows a comparison of SEM images before and after the redox treatment, as well as *in situ* hard X-ray ptychograms during the treatment. Comparable series were performed under model conditions using ETEM. Both techniques indicated changes on the nm-scale but a stable core-shell interface region, which was not affected by processes within the core. Despite the lower resolution of the *in situ* ptychography (approx. 20 nm vs. sub-nm in TEM), the study demonstrates the complementarity and potential for *in situ* ptychography as a tool in catalyst characterization, as realistic conditions can be applied during imaging.

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RUNOUT ERROR CORRECTION IN TOMOGRAPHIC IMAGE RECONSTRUCTION BY INTENSITY SUMMATION METHOD

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Abstract

The spatial resolution of a 3D X-ray phase-contrast computed tomography (XPCT) image is highly dependent on rotational-motion errors (axial runout error, radial runout error, and wobble) incurred in the rotation stage. As an alternative means of error correction and alignment, feature tracking of fiducial markers (IMOD and SPIDER) has been developed [1.2]. More recently, a method of feature-tracking alignment without fiducial markers also has been presented [3]. In this study we focused on undesirable artifacts, known as halos, that normally appear in phase-contrast microscopy with capillary. Halos result in spurious bright areas around the phase sample or reverse contrast in images. In fact, in the line profile across a sample, there is noticeably steep intensity gradient. Also, using a capillary is handier than fiducial markers in terms of fixing and containing solution samples such as live cells. In this paper, we present an image alignment method that utilizes halos to measure axial and radial runout errors incurred in the rotation stage. We used only intensity information, without extra hardware or complicated calculation (Fig. 1). Notably, the method, as demonstrated herein, can utilize the halo artifact to determine displacement. Compared with the non-aligned image, the shape of the specimen and the capillary are clearly visible (Fig. 2). This method is based only on intensity information; it entails no complicated calculations. We herein propose how to use the halo artifact for alignment.

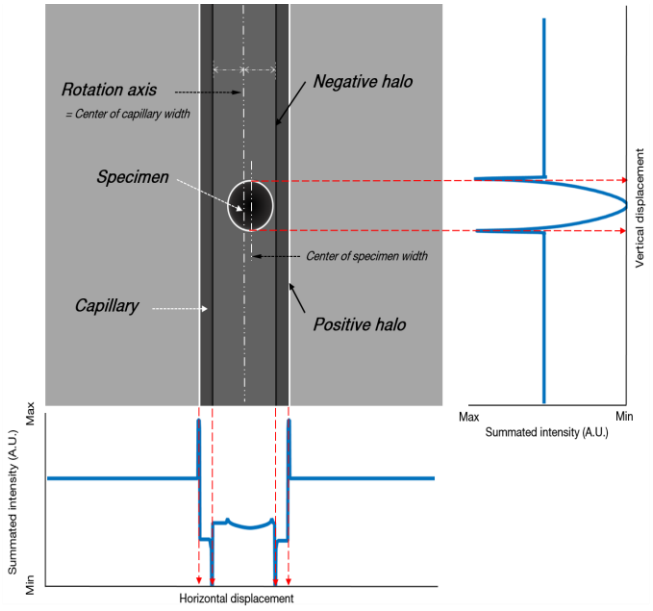

Figure 1: Virtual image of X-ray phase-contrast microscopy with summated intensity graph.

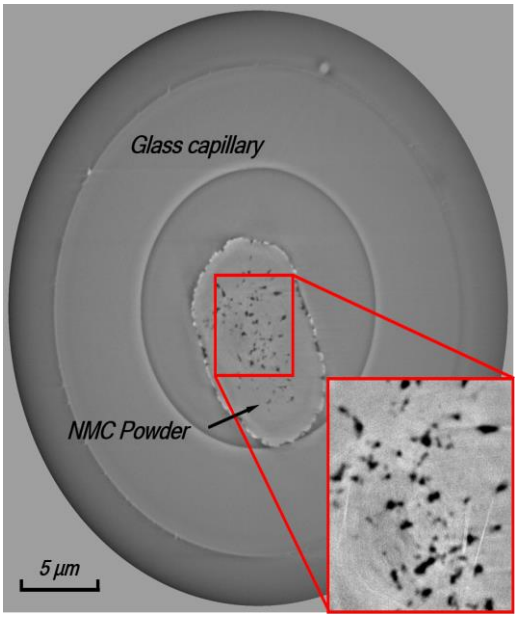

Figure 2: Reconstructed slide images of NMC powder after correction.

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ORGANELLE DISTRIBUTION IN A HYDRATED BIO-CELL BY CORRELATION BETWEEN SOFT X-RAY AND FLUORESCENCE IMAGES

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Abstract
Soft X-ray (SX) imaging in Water-window wavelength region (□=2.3□4.4 nm) can be used to image biological cells in a solution and shows a higher spatial resolution than images obtained in the visible wavelength region. Organelles of bio-cells obtained by SX imaging are clearer compared with those obtained by visible imaging owing to the difference in extinction coefficients between the two wavelength regions. However, it is difficult to identify the organelles imaged in the water-window wavelength region by its appearance alone. In this study, we obtain and compare SX and fluorescence images of Leydig cells of a mouse testis loaded with fluorophores. Identification of the organelles in the SX image is carried out by comparison with the fluorescence images with the use of principal component analysis. The result shows that the common structures between the SX images and fluorescence images obtained by staining with phalloidin or DAPI coincide well with the fine structures observed in the SX image (Fig. 1).

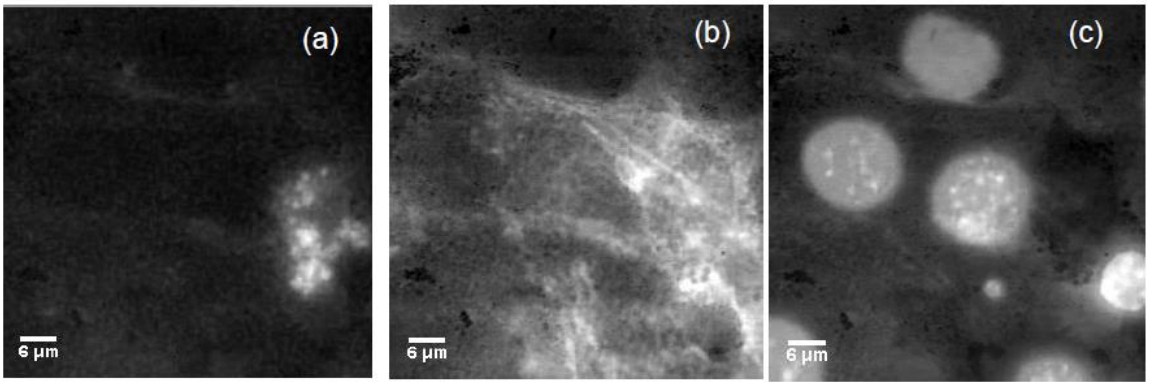

Figure 1: Eigen images between SX and fluorescence images with MitoTracker staining (a), with phalloidin staining (b), with DAPI staining (c). These eigenimages show the common structures between the SX image and the fluorescence images.

NOISE ANALYSIS OF SPECKLE-BASED DIFFERIENTIAL PHASE-CONTRAST IMAGING

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Abstract

Speckle-based x-ray phase-contrast imaging has been developed in recent years [1]. As it is readily implemented with polychromatic sources [2], it has drawn increasing interest. A near-field speckle image, used as a wavefront marker, is generated using a static diffuser such as a sandpaper or membrane. By tracking the changes to the speckle image caused by the disturbance of the wavefront introduced by an object, we get information on absorption, phase shift and scattering in the object.

As the transverse shift of the speckle image is proportional to the refraction angle, the result appears as differential phase shift. The goal of this study is to analyze the noise property of the differential phase-contrast (DPC) image, as well as other parameters that affect the final image quality, e.g., choice of subset size for correlation analysis. This quantitative approach can be used for future optimization and proper choice of methods.

An analytical derivation shows how the variance of the final DPC image depends both on the variance (and hence exposure time) of the original images, and on the visibility of the speckle pattern. This expression has distinct similarities to the variance of grating-based DPC images [3]. We can also show, using analytical derivation, how the object shape affects the signal-to-noise ratio of the images. Experiments and simulations support the analytical derivations. Figure 1 shows speckle images with different exposure times taken at Diamond Light Source's I13-1 Coherence Branchline. Figure 2 shows the analysis of the images in Figure 1, to support the theoretically derived dependence of the DPC variance on exposure time.

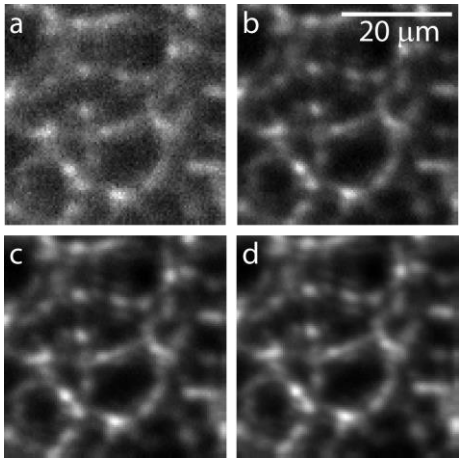

Figure 1: Speckle image taken with exposure time 0.5, 2, 8, 15 s, a - d respectively.

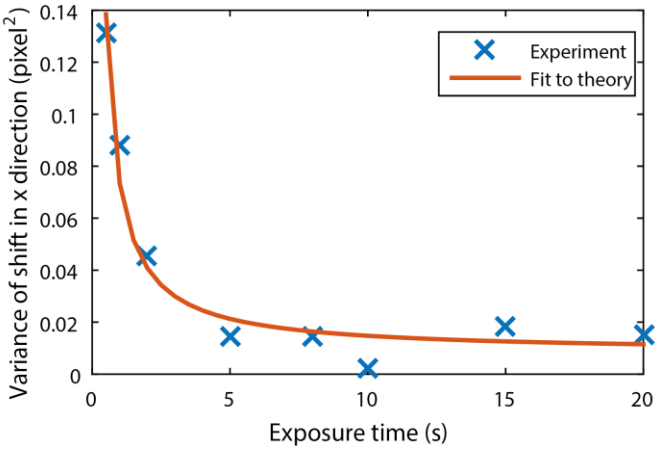

Figure 2: Plot showing the relation between the variance of DPC and the exposure time.

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MULTIPLE LENGTH-SCALE & DIMENSIONAL X-RAY MICROSCOPY AT SSRL AND BEYOND

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Abstract

Studies of complex heterogeneous systems, e.g. research in functional materials and geoscience, usually require a suite of analytical tools that are capable of providing complementary information at different length scales with different contrast mechanisms. This is due to the fact that the heterogeneity of the complex system usually exists across a wide range of length scales.

In this presentation, we demonstrate the strength of correlative multiple-length-scale X-ray microscopy (at Stanford Synchrotron Radiation Lightsource and beyond) by presenting three scientific case studies including 1) study of underground formation for CO_2 sequestration [1]; 2) study of complex heterogeneous catalysis material for petroleum refining [2]; and 3) study of the morphological and compositional changes of Fe melt at high pressure and temperature, which is relevant to the earth formation [3]. These scientific cases serve as good examples to show the link between the macroscopic behavior and the microscopic properties at multiple-length-scales. The developed correlative approach is also generally applicable to a wide range of applications.

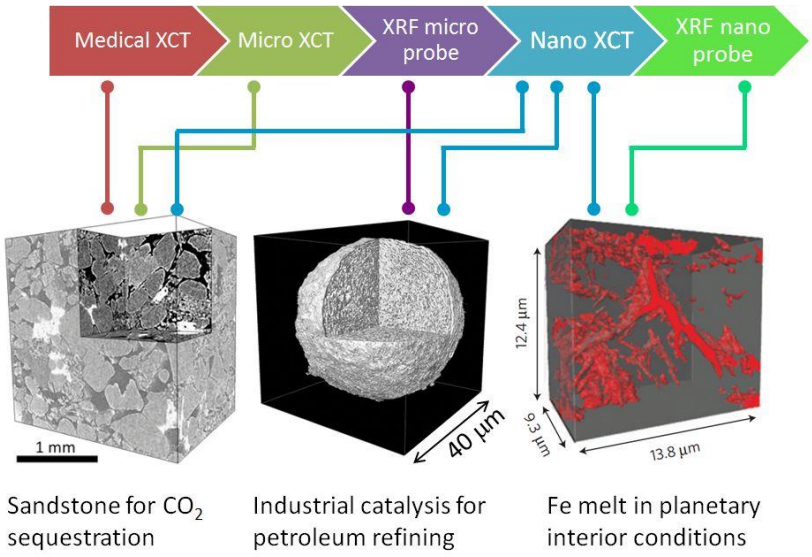

Figure 1: Three scientific case studies that utilized correlative multiple-lenth-scale X-ray microscopy at SSRL and beyond.

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SCANNING X-RAY MICROSCOPY AT SUB-20 NM RESOLUTIONS FOR SCIENTIFIC EXPERIMENTS BY GENERAL USERS

Y. S. Chu, H. Yan, E. Nazaretski, X. Huang, S. Kalbfleisch, K. Lauer,
W. Hu, L. Li, M. Ge, N. Bouet, J. Zhou, W. Xu, P. Ilinski

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Abstract

The Hard X-ray Nanoprobe (HXN) beamline at the NSLS-II has been commissioned [1]. Its initial microscopy capabilities are now available for scientific experiments by general users, including scanning fluorescence and differential phase contrast imaging [2] using either multilayer Laue lenses (MLL) or a Fresnel zone plate (ZP) as focusing optics in separate microscope modules. Presently, the MLL-based microscope module is capable of producing a beam size down to ~11 x 13 nm^2, ideal for high-resolution fluorescence imaging experiments at a fixed photon energy. The ZP-based microscope module can focus to a minim spot size of ~60 nm with relatively larger working distances, ideal for XANES microscopy and in-situ experiments requiring such a working distance. The HXN x-ray microscope with a compact size and high mechanical stiffness demonstrates excellent positioning stability with the vibrational amplitudes below 2 nm at the resonance frequencies. Operating in a He environment within a UHV enclosure, thermal stability better than 0.05° C/day is achieved. The HXN fluorescence imaging capability has been used for investigating hard materials as well as biological systems. Within a year, fluorescence tomography, nanodiffraction, and ptychography capabilities will also be offered to general users. The presentation will focus on the commissioning data, scientific results, and the near-future scientific direction of the HXN beamline.

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THE HERAKLES SCANNER: INTEGRATED ABSORPTION TOMOGRAPHY/X-RAY FLUORESCENCE SCANNER FOR NON-DESTRUCTIVE 3D ANALYSIS ON THE MICRO-SCALE

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Abstract
The Herakles 3D X-ray scanner is a radically new instrument, integrating three different X-ray based microanalysis methodologies, being absorption computed tomography (absCT), XRF tomography (XRF-CT) and confocal XRF analysis (cXRF). These techniques yield complementary three dimensional information in a basically non-destructive way.

By combining these methodologies in the same instrument, the user is able to acquire both morphological and elemental 3D information on the micro-scale with a single experimental run. The experimental sequence starts with an absCT scan, providing a detailed visualization of the internal structures of the sample with a resolution of approximately 1 micron. The reconstructed CT images are then used to select the regions of interest for detailed elemental analysis using either the XRF-CT or cXRF stage. Due to the ultra-accurate motor movements, the sample can be transferred from the absCT station in front of the XRF source with micrometer precision, enabling analysis of even the smallest heterogeneities discovered in the sample during the absCT measurement. Furthermore, the complementary information gathered with these techniques opens a wide range of new possibilities for integrated data analysis and online data correction algorithms.

Data demonstrating the precision and scientific value of the new experimental procedure are represented, including measurements on both test samples and “real life” samples.

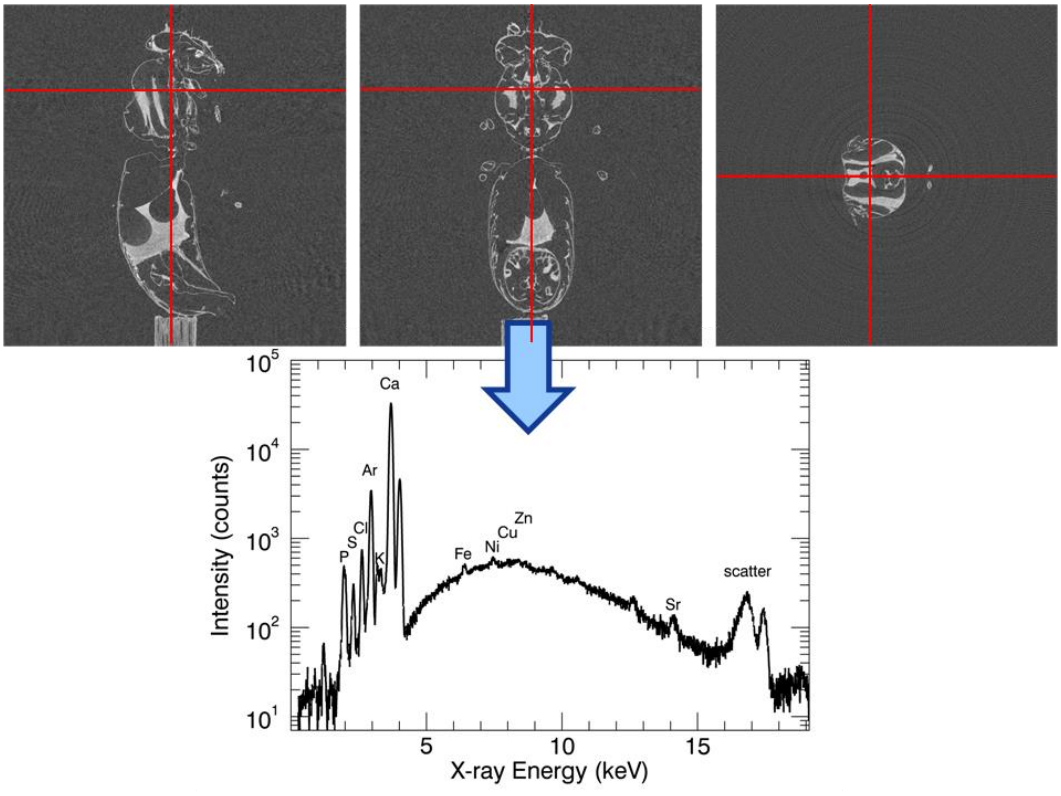

Figure 1: Using the absCT data of the sample (wasp test sample) a point for XRF analysis is selected, yielding the elemental composition.

AGGREGATION OF GAS AT MICROSCOPIC SCALE INVESTIGATED BY SYNCHROTRON RADIATION TECHNIQUE

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Abstract

The non-intrusive measurement, nanoscopic spatial resolution and the combined with a chemical sensitivity makes STXM an ideal technique to study the properties and behavior of individual nanobubbles in a complex environmental interface. Here, a synchrotron-based STXM with nanometer resolution was used to investigate the existence and behavior of interfacial gas nanobubbles confined between two silicon nitride windows. The observed nanobubbles of SF6 and Ne were quite stable with the diameters smaller than 2.5 micrometers. However, the larger bubbles became unstable and grew up during the soft X-ray imaging, indicating that stable nanoscale gas bubbles may have a length scale, which was in consistent with the previous report by atomic force microscopy (AFM)[1]. More importantly, the chemical information from X-ray absorption showed that there might be a new state of water inside of nanobubbles[2]. Also, X-ray fluorescence absorption and mapping were used to investigate the absorption of Krypton on porous materials or surfactant containing solution. Our study showed that synchrotron radiation techniques were promising techniques to study the aggregation of gases near the solid/water interfaces at the nanometer scale.

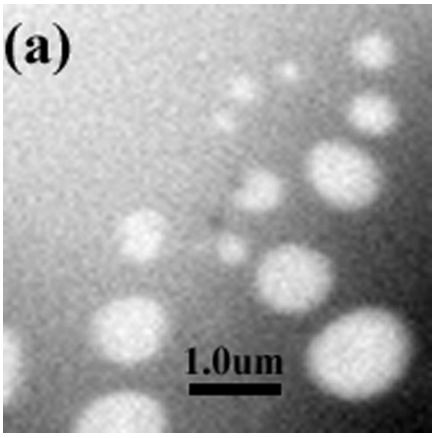

Figure 1: STXM images of nanobubbles at 890eV.

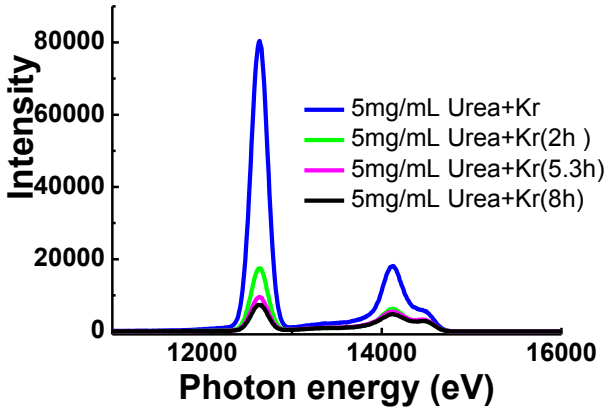

Figure 2: This is the caption for Figure 2 of this paper.

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THREE-DIMENSIONAL CORRELATIVE IMAGING: A COMPARISON BETWEEN X-RAY COMPUTED TOMOGRAPHY AND FIB-SEM SLICE-AND-VIEW IN THE STUDY OF SOFC ANODES

Abstract

The study of functional porous materials, for uses spanning electrochemical devices to complex geological samples, has received increasing interest in the research community over the last decades. This is due in part to the advent and development of diverse imaging capabilities provided by such techniques as X-ray micro- and nano- computed tomography (CT) [1]. These allow for the three-dimensional reconstruction of heterogeneous materials whose intricate microstructural properties are otherwise difficult to gauge. Information can be gathered either at a synchrotron or, more recently, by means of lab-based sources equipped with specialized optics.

These techniques have been leveraged in investigations of many energy materials, including solid oxide fuel cells (SOFCs), which present an electrochemical alternative to fossil fuel consumption, with high efficiencies and vast fuel versatility [2]. Recent work has focused on imaging SOFCs to better understand structure-property relationships and the dynamics involved in various degradation pathways. However, only few studies have emphasized the importance of corroborative evidence [3, 4]. Reliable identification and segmentation of the three phases in SOFC anodes, by means of X-ray CT, has thus far been restricted to X-Ray Absorption Near-Edge Spectroscopy (XANES) which requires the use of less readily available synchrotron sources.

This work focuses on the use of *lab-based* X-ray nano-CT to gain access to properties such as volume fractions, volume-specific surface areas, tortuosity and triple-phase boundaries (TPBs). Importantly, the focus is on providing corroborative evidence of the segmentation by comparing X-ray derive datasets with those furnished by focused-ion beam scanning electron microscopy slice-and-view. Sample preparation techniques, including laser micromachining methods, are also discussed with a focus on the fabrication of samples of appropriate geometry.

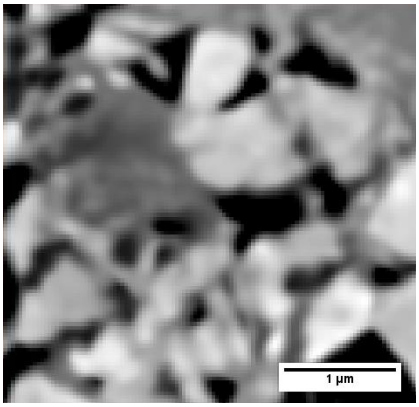

Figure 1: Filtered orthoslice of a representative Ni-YSZ anode, imaged by X-ray nano-CT.

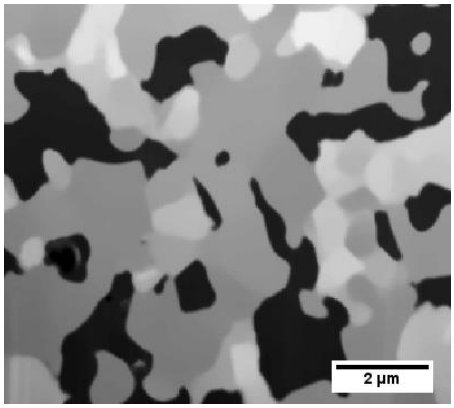

Figure 2: Filtered orthoslice of a representative Ni-YSZ anode, imaged by FIB-SEM slice-and-view.

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COMPARISON OF GRATING- AND SPECKLE-BASED X-RAY PHASE-CONTRAST IMAGING

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Abstract
X-ray phase-contrast imaging provides a promising complement to conventional attenuation-based radiography for observing weakly absorbing materials. Several different phase-contrast methods have been developed in recent years, among others imaging with a grating interferometer [1], and a relatively new method based on a sample-induced displacement of a near-field speckle pattern [2]. The speckles are generated by passing the x-rays through a random phase object, such as a piece of paper or sandpaper. Both phase-contrast methods yield multimodal images – differential phase, dark-field and absorption – and can be implemented using broadband laboratory x-ray sources.

As phase-contrast imaging is developed mainly for future medical applications, it is important to know the strengths and weaknesses of the different methods, as well as controlling the radiation dose the sample is exposed to. We therefore compare grating- and speckle-based x-ray phase-contrast imaging under the same dose conditions. Simple samples have been imaged with both methods, at equal source-object distances and under constant total exposure time, using a polychromatic liquid-metal-jet x-ray source [3] and the Coherence Branchline I13-1 at the Diamond Light Source. The quality of the resulting images is evaluated with respect to image-quality measures such as contrast-to-noise ratio and resolution.

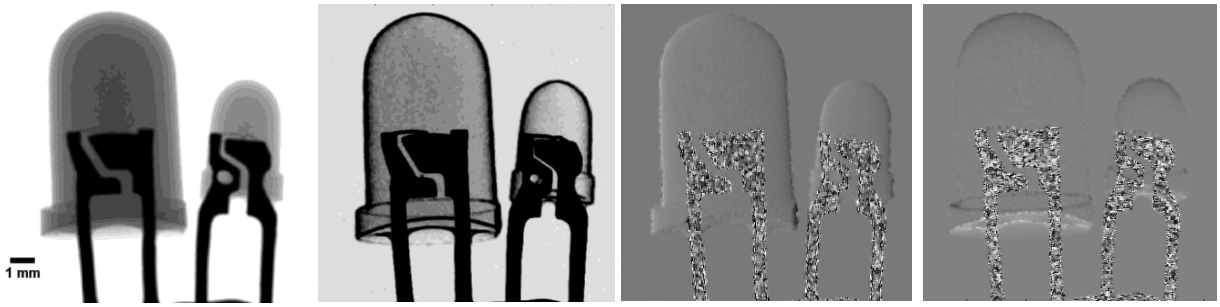

Figure 1: Two light-emitting diodes imaged with a liquid-metal-jet x-ray source using speckle-based phase contrast. From the left: absorption (a), dark-field (b) and refraction angle in horizontal (c) and vertical (d) directions.

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ANGULAR-DEPENDENT ABSORPTION SPECTROSCOPY REVEALS APATITE CRYSTAL ORIENTATION IN HUMAN TEETH

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Abstract

Background: Recently we used full-field X-ray absorption near edge structure spectroscopy (FF XANES) at the Ca K-edge with sub-micron resolution to assess alterations of hydroxyapatite (HA) originating from human bone. We compared regions of different maturation stage, different anatomical site and different states of pathology. Our main conclusion was that the spectral differences are dominated by polarization effects. We showed how it is possible to exploit this angular absorption dependency by analyzing 1s to 4p electronic transitions and showed that Ca-K-edge spectroscopy is sensitive to HA crystal orientations [1].
Human teeth, composed of an outer highly mineralized layer of enamel surrounding a bulk of bone-like material termed dentine, are prime examples of hardened, hierarchical, evolutionarily-optimized cutting tools reinforced with carbonated HA (cHA).
Thus, given known crystal orientations in teeth and due to the polarized state of the ID21 beam, peak shifts directly reveal structural inhomogeneities and anisotropy of the c-lattice parameter.
Objectives: Here we present an experimental workflow to quantify the modulations of crystal orientations in a human tooth sample by collecting XRF radiation at few specific energies of the primary X-ray beam.
Methods: µXANES and XRF mapping were performed using the scanning X-ray microscope at ID21 installed at the ESRF. The linearly polarized monochromatized beam was focused to 0.8 × 1.0 µm². Photodiodes were used to monitor the incoming beam intensity (I_0) and the XRF and scattered radiation (I). Scans were performed in continuous (zap) mode and specific energies (4053 eV, 4055 eV and 4150 eV) were selected for µXRF mapping.
Conclusions: Collecting µXRF maps at specific energies clearly reveals the modulation pattern of HA orientation in both enamel and dentin in human teeth. This technique to characterize bio HA is potentially relevant in many fields such as paleontology or material science in medical applications [2].

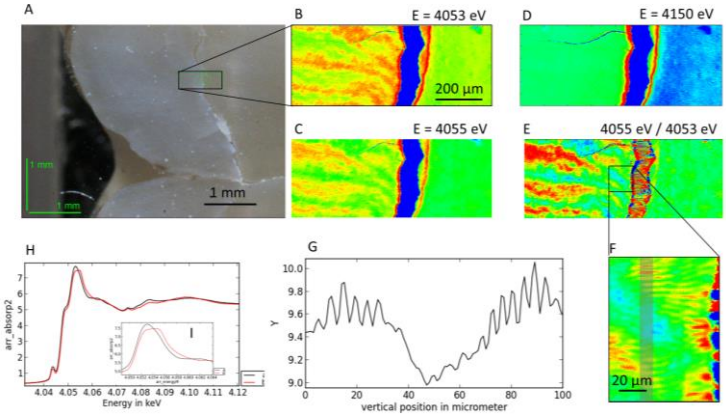

Figure 1: Preliminary results and workflow indicating the assessment of HA crystal orientation. ROIs in enamel and dentine are indicated in the microscopy image (A) of which XRF maps were collected. Modulations of crystal orientation are clearly visible in B and C and in map E showing the ratio of B and C. Map D shows that the Ca concentration does not reveal any modulations. F depicts the ratio of maps collected at higher spatial resolution at 4055 eV and 4053 eV. Color-bars are adjusted to have the highest contrast. Panel G: intensity profile of the ratios shown in F along the grey-underlined vertical bar. From subfigures E, F and G it can be seen that crystal orientation is modulating with two different frequencies periodicity of about 5 µm and of about 90 µm periodicity. Two XANES spectra originating from two different enamel regions are shown in H and I (demonstrating different ratios of the 1s to 4p transitions).

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CORRELATIVE X-RAY AND FIB-SEM TOMOGRAPHY TO ADDRESS MULTI-SCALE CHALLENGES IN MATERIALS SCIENCE

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Abstract

X-ray microscopy (XRM) is in a state of rapid development. Fueled by advancements in synchrotron facilities and newly-developed imaging techniques, laboratory XRM is steadily gaining adoption and is becoming a standard tool for material characterization on the micro- and nanoscale. The growth of XRM instrumentation is the result of an increase in research interests to solve practical challenges. Here, we present recent advancements in and application examples of laboratory imaging as well as efficient multi-scale / correlative workflows combining XRM with higher resolution, but destructive FIB-SEM tomography.

One of the motivating principles for XRM is the ability to capture many different length scales of information in 3D without sectioning the samples. This is especially useful for studying the multi-scale nature of a material, capturing its evolution over time, and revealing the meso- and micro-structures with resolution down to the tens of nanometers. Furthermore, by preserving the sample, XRM serves a unique function in a correlative microscopy workflow, providing a missing piece of 3D and 4D information that may be used to drive other techniques, such as focused ion beam coupled scanning electron microscopy (FIB-SEM).

Correlative XRM has found particular utility in the field of energy materials (e.g., Li-ion batteries), as well as in more traditional materials science applications (e.g., metals research). Here, we present the results of characterizing commercial Li-ion batteries, using multi-length scale XRM to capture the complex features over a range of times and length scales. The results were then passed into the FIB-SEM, to use the FIB to extract and image targeted regions of interest with EDS analysis. Furthermore, we present the results of applying this approach to characterizing an aluminum 7075 alloy, using the XRM technique to survey a large volume and the FIB-SEM to localize a representative region for nano-scale analysis. Finally, we present a 4D characterization study of the corrosion of a magnesium specimen, with the XRM used to characterize the evolution of microstructure during corrosion, with nano-scale XRM to identify local heterogeneities and the FIB-SEM to further clarify the structures with higher resolution.

Both an introduction to the correlative, multi-scale approach and a discussion of the application examples will be provided.

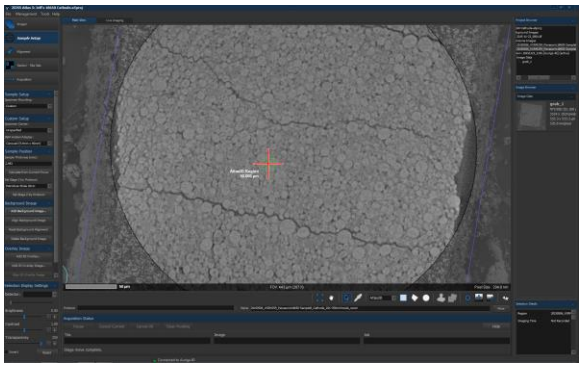

Figure 1: 3D XRM data of a Li-ion battery overlaid on an SEM micrograph for correlative microscopy.

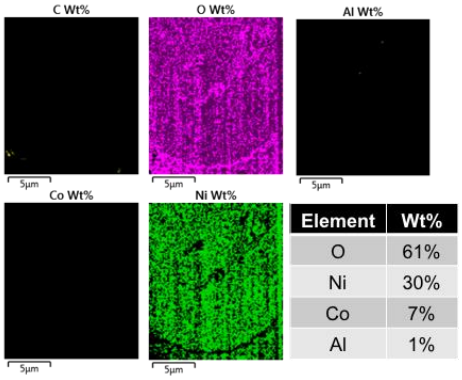

Figure 2: Corelative EDS analysis of a targeted region of interest from the Li-ion battery, identified within the XRM volume.

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MULTIMODAL IMAGING OF THE HUMAN KNEE DOWN TO THE CELLULAR LEVEL

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Abstract
Computed tomography (CT) reaches the best spatial resolution for the 3D visualization of human tissues among the available nondestructive clinical imaging techniques. Nowadays, sub-millimeter voxel sizes are regularly obtained. Regarding investigations on true micrometer level lab-based micro-CT (µCT) has become gold standard. The aim of the present study is firstly the hierarchical investigation of a human knee post mortem using hard X-ray µCT and secondly a multimodal imaging using absorption and phase contrast mode in order to investigate hard (bone) and soft (cartilage) tissues on the cellular level. After the visualization of the entire knee using a clinical CT (Siemens Somatom Emotion 16) with a lateral pixel length of 0.3 mm and a slice thickness of 0.6 mm (see Fig. 1a), a hierarchical imaging study was performed using a nanotom® m (phoenix|x-ray, GE Sensing & Inspection Technologies GmbH, Wunstorf, Germany) equipped with a 180 kV / 15 W nanofocus X-ray source. First, the acceleration voltage and the current were adjusted to 180 kV and 30 µA for the entire knee. Due to the size of the knee the pixel length could not be below 65 µm (see Fig. 1b). The two data sets were rigidly registered using a cross-correlation metric in order to directly compare the two imaging modalities. At this level, the trabecular structures of the bones can be investigated. For the reduction of the pixel length down to 25 µm the tibia and the femur were extracted and both scanned with 150 kV / 50 µA. The highest resolution could be achieved after extracting cylindrically shaped plugs from the two bones. The tibial cylinder was imaged with a pixel length of 8 µm (60 kV / 310 µA) and the femoral plug with a pixel length of 3 µm (40 kV / 350 µA). The high resolution scans revealed the mineralized cartilage zone including the tide mark line as well as individual calcified chondrocytes. For the visualization of the cartilage, grating-based phase contrast µCT (I13-2, Diamond Light Source) was performed. With an effective voxel size of 2.3 µm it was possible to visualize individual chondrocytes within the cartilage.

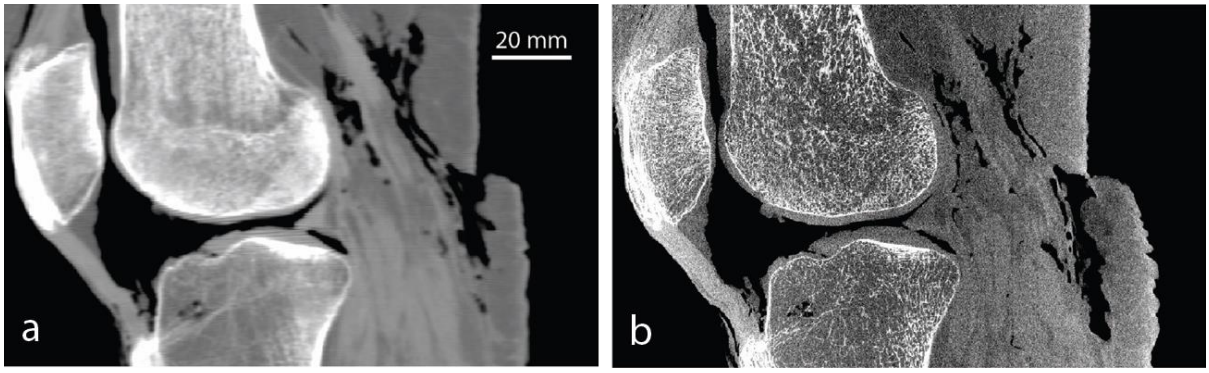

Figure 1: Sagittal slice of entire knee measured using the clinical scanner (a) and the nanotom® m (b) showing femoral condyle, tibial plateau and patella. Soft tissue includingpatella tendon, quadriceps tendon and m. gastrocnemius can be depicted.

SCANNING SOFT-X-RAY SPECTRO-NANOPROBE AT THE POHANG LIGHT SOURCE

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Abstract

A scanning transmission x-ray microscope (STXM) is operational at the Pohang Light Source. With a zone plate having 25 nm outermost zone width, STXM provides chemical state maps on the sample with space resolution of 30 nm. The usable photon energy range is 250 – 1,500 eV, with typical energy resolving power of 2,000 – 5,000. The polarization of x-rays can be changeable by adjusting the phase of the undulator. The STXM has been actively utilized for the investigation of formation of magnetic domains within thin films, oxidation states and chemical states of functionalized graphene layers, reaction mechanism of cells encapsulated and cultivated within graphene layers, and so on. In this presentation, basic functional features and some of new application examples will be provided. Besides these, there will be a report on our recent attempt to implement two techniques of Ptychography and low energy x-ray fluorescence spectroscopy into the STXM.

INVESTIGATION OF INELASTIC SCATTERING OF X-RAYS FOR THE ALKALINE EARTH OXIDES

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Abstract

The advent of synchrotron X-ray sources spurt an interest in the studies of scattering of X-rays from different atomic and molecular systems [1,2]. They provide valuable information about the two dimensional electronic momentum density contour maps analogous to the charge density maps. These investigations are carried out with a view to see directly the binding effects in the molecules [3]. There are various techniques to analyze the effect of bonding. Each one has its own special feature [4]. In this paper, such effects will be presented for alkaline earth oxides, i.e. BeO, MgO, CaO and SrO. Among them BeO and CaO are used in many high performance semiconductor parts for communication equipment. Whereas MgO and SrO are important catalysis in petrochemical products. In view of this, we will present the directional properties of internally folded density function B(r). The variation of this function (per electron) with radius of the molecules is shown in the Figure 1.

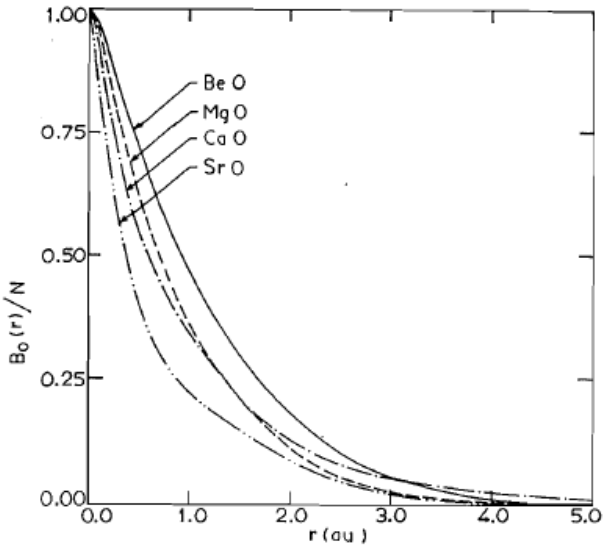

Figure1: Isotropic B(r) function (per electron) for BeO, MgO, CaO and SrO.

Further, we will also investigate how this study will reveal the changes in chemical properties and different bonding situations in this homologous series of diatomic molecules. The detailed results will be presented and discussed at the conference.

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DEVELOPMENT OF CONCAVE-CONVEX IMAGING MIRROR SYSTEM FOR COMPACT FULL-FIELD X-RAY MICROSCOPE

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Abstract

The development of full-field X-ray microscopes is highly desired because they have a potential to observe relatively thick samples with high resolution. An advanced Kirkpatrick-Baez (AKB) optical system was proposed as an achromatic and coma-free imaging optical system [1, 2], and it can reach a spatial resolution of approximately 50 nm [3]. However, the full-field X-ray microscopes with the AKB system require considerable distances of several tens of meters between the imaging mirrors and the camera to obtain the high magnification factor. Consequently, the apparatus becomes very large and its versatility decreases.

To overcome this problem, we propose a concave-convex imaging mirror system (Fig. 1 (a)) that can provide a large magnification factor even with a short total length. The large magnification benefits from a set of convex and concave mirrors that make up the principal surface near the sample (Fig. 1 (b)). This characteristic can facilitate the realization of a laboratory-scale achromatic full-field X-ray microscope with a 50 nm spatial resolution.

In this study, we investigated one-dimensional imaging characteristics of the proposed imaging optics. We confirmed that it can achieve a magnification factor of 310 with a total length of only 2 m. The point spread function (PSF) calculated using a wave-optical simulator based on the Fresnel-Kirchhoff's integral is shown in Fig.2. It was suggested that the expected minimum resolution should be less than 40 nm at an X-ray energy of 10 keV. Moreover, an investigation of the PSFs at off-axis conditions revealed that it can obtain a field of view of 10.5 μm.

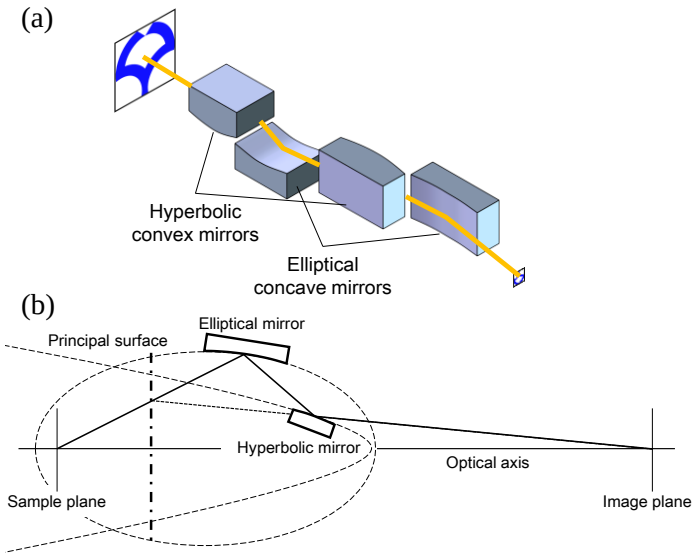

Figure 1: Schematic diagram of concave-convex imaging mirror system: (a) arrangement of mirrors, (b) cross sections in the one-dimensional imaging mirrors.

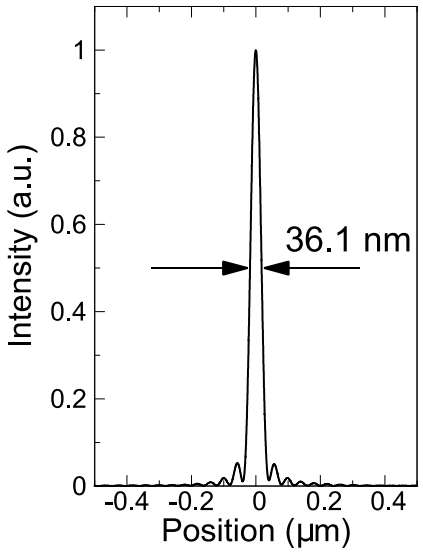

Figure 2: Calculated point spread function at an X-ray energy of 10 keV.

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MECHANISM INVOLVED IN X-RAY MICROSCOPY OF BIOLOGICAL MATERIALS

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Abstract

X-ray fluorescence (XRF), particle-induced X-ray emission (PIXE) and electron probe micro-analysis are the important techniques to examine biological samples [1]. The mechanism involved in such studies are inner shell ionization; vacancy creation; filling up of the vacancy by radiative and non radiative transition and consequently production of the X-rays. All these processes are influenced by various factors (Fluorescence yields and Coster-Kronig transition probabilities) which in turn are caused by certain effects viz. multiple ionization, united atom, relativistic and non relativistic effects. Calculated results are compared with the experimentally measured values of the L X-ray spectra for Platinum by proton impact. Numbers of workers [2-4] have studied these effects for heavier ions. But in the case of protons, investigations are still required. In this paper, we will discuss all these factors responsible for the X-ray production. The comparison of various intensity ratios is illustrated in Fig. 1. In this figure, we have presented L_γ/L_α line intensity ratio calculated for multiple ionization non relativistic (MINR), multiple ionization relativistic (MIR) and without multiple ionization (WMI) along with experimentally measured values.

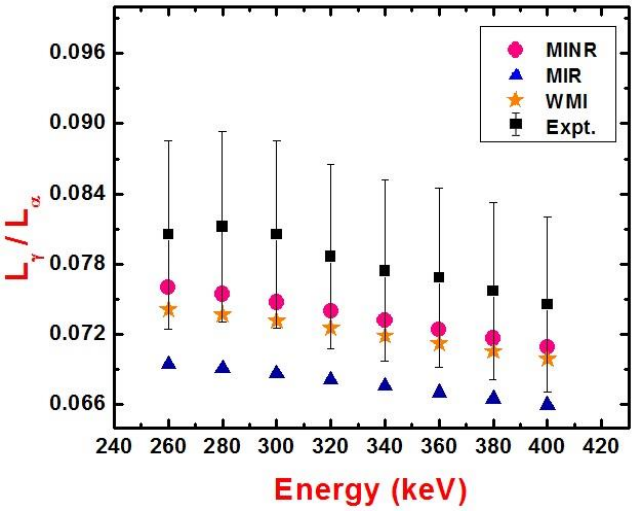

Figure1: L_γ/L_α Line intensity ratios for Platinum as a function of proton energy.

The detailed results will be presented and discussed during the conference. Conclusively, the present work provides an important application in quantitative analysis, where the data are derived from the observed fractional yield of the decay, e.g. X-ray microscopy.

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AN INNOVATIVE ON-THE-FLY SCANNING DATA ACQUISITION SYSTEM FOR X-RAY NANOPROBES AT TAIWAN PHOTON SOURCE

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Abstract

The on-the-fly scanning control system is built base-on the high speed hardware, high throughput data stream and multi-channel control interfaces. Using the FPGA with embedded processer, the input and output data are processing through the hierarchy formed by the Digital to Analog Converter (DAC), Analog to Digital Converter (ADC), Gigabit Ethernet (GbE), X-ray fluorescence (XRF) and Interferometer control interfaces, as shown in Figure (a).

During the scan, while the 2 DACs control the X-,Y- axes of the flexure stage, in parallel up to 4 ADCs acquire the data and packet it into data packets. The data is tagged with position from the laser interferometers and timing in micro-second precision. The GbE sends the data back to the computer to reconstruct the scanning images as shown in Figure (b). It is technically easy to combine two sequential scan images without any stitching process (see Figure (b)). Besides, the data also serves for information analysis, such like vibrational and temporal analysis.

To demonstrate, we build a test system consisting of an e-beam source, a nano flexure stage and laser interferometers as shown in Figure (c).The scanning speed for this image is at 5 lines/s. The speed can be higher and only limited by the stage. The data rate is up to 500K samplings/sec/channel. This system will be implemented into the X-ray Nanoprobe beamline and other scanning type microscopes at Taiwan Photon Source in the future.

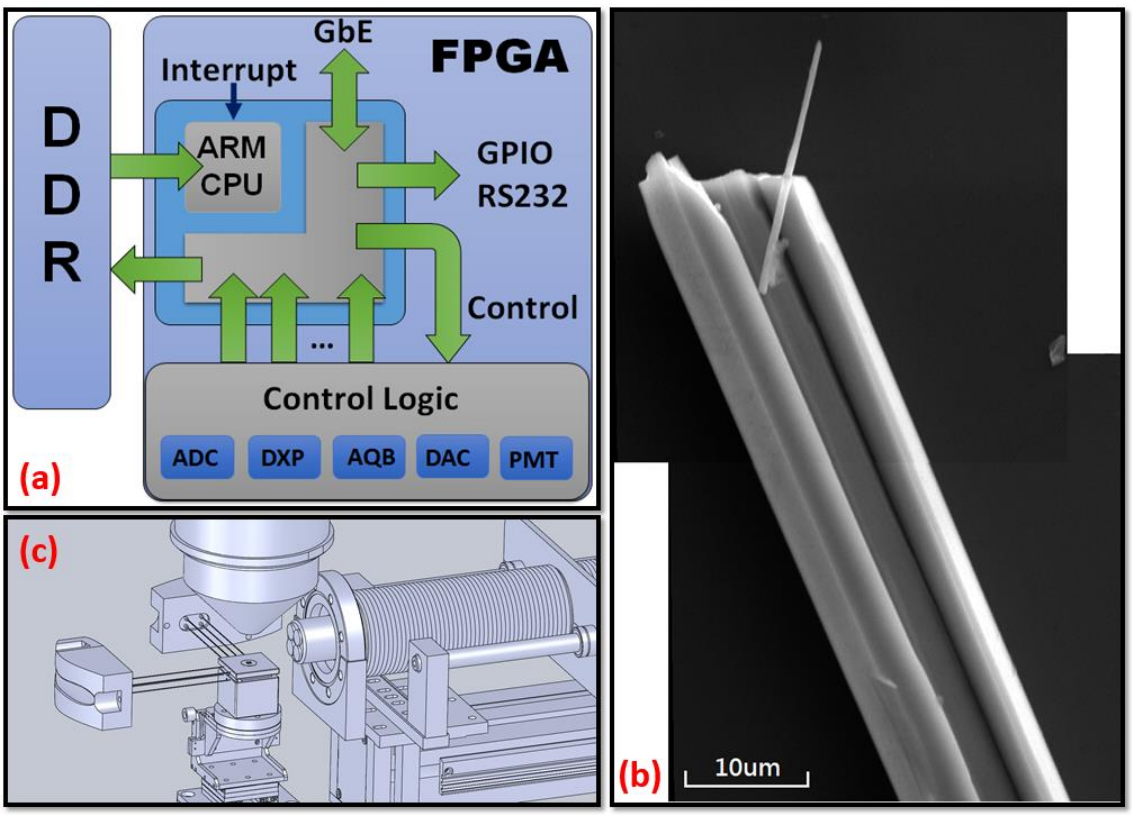

Figure (a) the block diagram for the data acquisition system **(b)** On-the-fly scanning image. It combines two fly scanning images **(c)** the demo system consists of a focused e-beam, a nano-flexure stage and laser interferometers.

A NOVEL SPIRAL TRAJECTORY SCANNING SYSTEM FOR X-RAY MICROSCOPY BASED ON FPGA

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Abstract

Scanning microscopy has long been one of the most important members in the X-ray microscopy family. The sensitivity as well as the efficiency of a scanning X-ray microscope strongly rely on the scanning strategy [1]. The conventional raster scanning systems, yet being adapted commonly, are disadvantageous due to low overlapping ratio, setting the limitations in driving frequency and signal performance [2].

This paper purposes a novel design of a equidistant trajectory scanning system, based on FPGA timing control, for generating ideal image scan trajectories, such like mesh, concentric, Fermat spiral, and novel spiral scan patterns. By contrast, the novel spiral pattern generated by the system can provide a more uniform coverage and higher overlap ratio (up to 12%) than the commonly used raster and concentric patterns, under the same trigger time (>50 ns). This equidistant trajectory scanning system will be implemented to the hard and soft X-ray scanning microscopes at the Taiwan Photon Source.

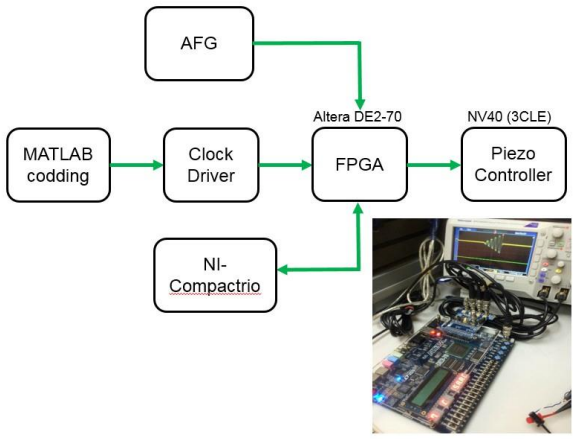

Figure 1: The schematic diagram of the equidistant trajectory scanning system.

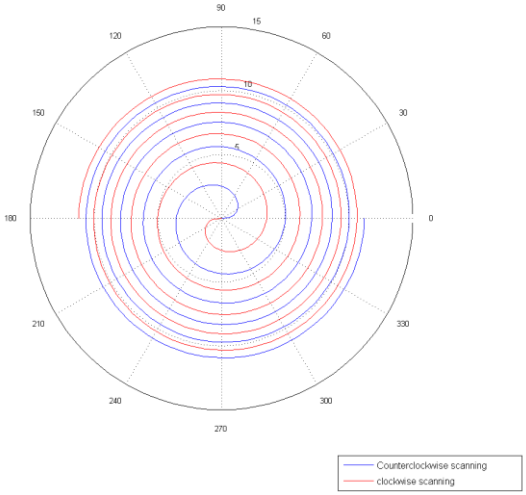

Figure 2: The scan trajectories of the novel spiral pattern.

References

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DEVELOPMENT OF X-RAY PHASE-CT MICROSCOPE BASED ON LABORATORY SOURCE

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Abstract

We are developing quantitative X-ray phase microscopy using a laboratory source by combining an X-ray microscope optics and a Lau interferometer [1]. By incorporating this concept, a novel X-ray micro-interferometer system which can measure quantitative phase-CT with high spatial resolution is achievable. The schematic of the optics is shown in Fig.1. A source grating and a phase grating are installed in full-field X-ray microscope optics. Image of the source grating formed by the microscope objective works as secondary sources. The phase grating is set so that the projection from the each source generates fringes on the image plane constructively. As a result of conventional fringe scan measurement by displacing one of the gratings, doubled image having opposite phase will be obtained like in ref. [2]. For the system, Zeiss Xradia Ultra800 is adopted as a base microscope in this study.

In the presentation, we will show detail design, preliminary experiment using visible light system, and initial X-ray result.

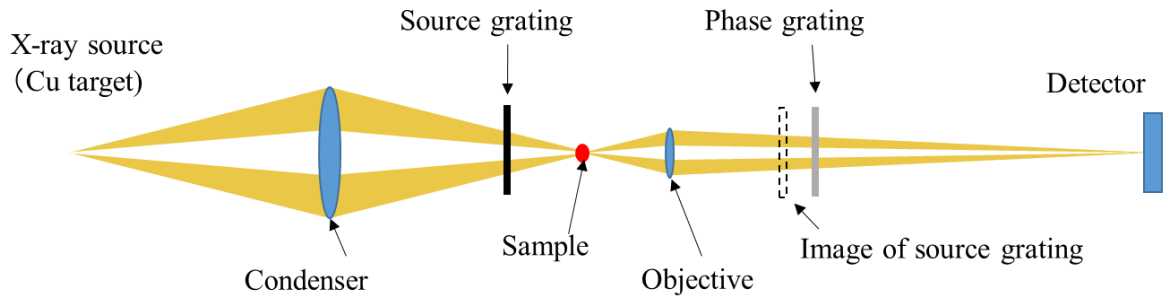

Figure 1: Schematic of the X-ray micro-interferometer

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DEFECT-ASSISTED HARD X-RAY MICROSCOPY WITH POLYCAPILLARY OPTICS

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Abstract

Polycapillary x-ray focusing devices are built from hundreds of thousands of bent glass micro-capillaries that are stacked into hexagonal arrays. Defects were identified as to deteriorate the x-ray transmission of these devices. In this presentation, we show that intrinsic point defects (missing, broken or larger capillaries) lead to the formation of multiple x-ray images of an object, which was positioned in the focal plane (Fig. 1) . These multiple images can be analyzed using the so called coded aperture approach [1-3]. The resulting spatial resolution is limited by the defect size and not by the focal spot size, which has typically size of 10-100 μm. In a proof-of-principle experiment [4], using commercially available optics with natural defects, we obtained sub-micron resolution (Fig. 2), that has not been achieved with focusing polycapillary optics until now. Tailored optics with a controlled distribution of "defects'" (fabricated using procedures known from photonic crystal fibers [4]). could be used for multimodal nanoscale x-ray imaging with laboratory setups.

This work was supported by the Polish National Science Center (grant No. DEC-2013/11/B/ST2/04057).

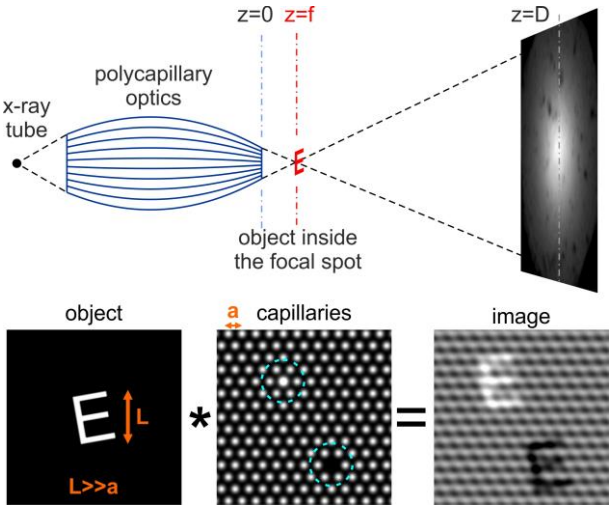

Figure 1: Idea of defect-assisted x-ray microscopy. Defects (missing or broken and larger capillaries - marked with dashed circles) break the periodicity and lead to the formation of distinct multiple x-ray images of the object.

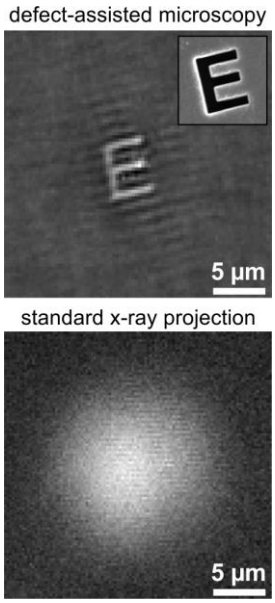

Figure 2: Comparison of defect-assisted imaging with standard x-ray projection imaging with the focal spot acting as a secondary source. Inset: SEM image of the object.

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STXM AT THE HERMES BEAMLINE: CAPABILITIES AND THE FIRST COMMISSIONING RESULTS

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Abstract
The first commissioning results and the experimental capabilities of the new Scanning Transmission X-ray Microscopy (STXM) installed at the HERMES beamline at SOLEIL are presented. The STXM instrument is designed to work in the soft x-ray regime with multiple detection possibilities and various possible sample environments.

A photomultiplier tube (PMT), a quadrant diode detector and a channeltron are currently installed for bulk and surface sensitive measurements. Various spectro-microscopic techniques and detection modes such as XANES, XMCD and XMLD are already implemented. The different sample mounts available include a temperature controlled sample mount (-150°C to 80°C), a magnetic system (vectorially variable ±200mT) and a rotatable sample mount. In addition, photon detection technique, such as using the PMT, allows the investigation of samples at atmospheric pressures. Special attention has been taken during the designing of the beamline to minimize the carbon contamination of the beamline optics [1]. Thus the STXM instrument is also capable of measuring organic samples. We present the very first commissioning and experimental results in this presentation.

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HIGH-ENERGY MICROTOMOGRAPHY USING SYNCHROTRON RADIATION AT P07 / PETRA III

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Abstract

The Helmholtz-Zentrum Geesthacht, Germany, is operating the user experiments for microtomography at the beamlines P05 and P07 using synchrotron radiation produced in the storage ring PETRA III at DESY, Hamburg, Germany. In recent years the software pipeline, sample changing hardware for performing high throughput experiments were developed. The current status of the setup for attenuation-contrast microtomgraphy and grating-based phase-contrast microtomography using high photon energies at the beamline P07 / PETRA III will be given. Furthermore, optimisation and automatisation of scanning techniques for attenuation contrast, will be presented. These are required to scan samples which are larger than the field of view defined by the X-ray beam. The integration into an optimized reconstruction pipeline will be shown

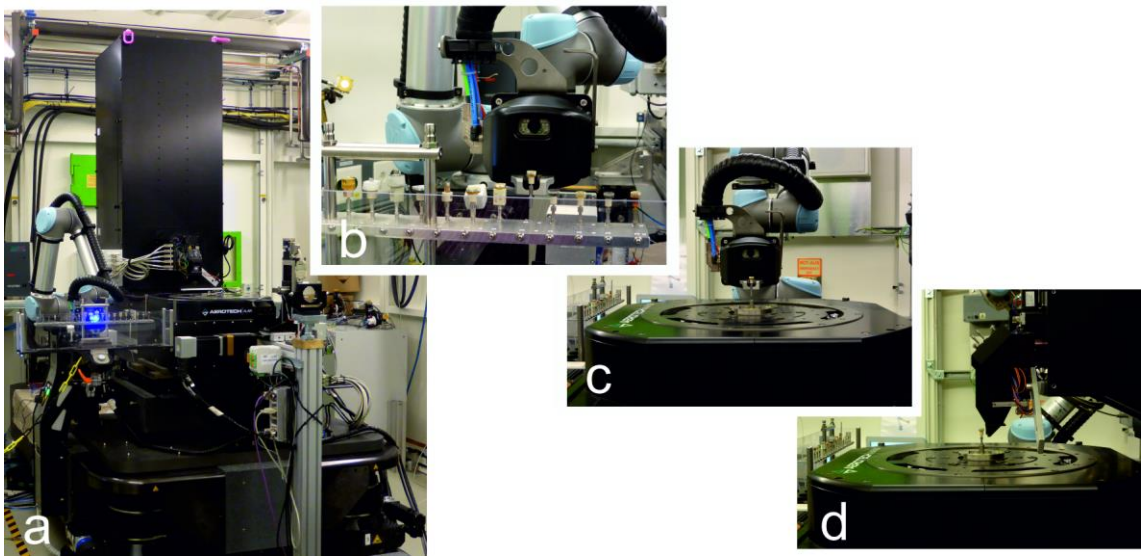

Figure 1: Sample changer for high-throughput tomography at beamline P07 / PETRA III. a) sample changer scanning the sample ids, b) selecting one sample, c) installation into rotation axis, d) sample changer in rest position for scanning the sample.

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SIMPLE AND ROBUST SYNCHROTRON AND LABORATORY SOLUTIONS FOR HIGH-RESOLUTION MULTIMODAL X-RAY PHASE-BASED IMAGING

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Abstract
Edge illumination X-ray Phase Contrast Imaging (XPCI) techniques [1,2] are capable of quantitative retrieval of differential phase, absorption and ultra-small-angle X-ray scattering [3,4]. We have recently developed a series of approaches enabling high-resolution implementations of these techniques, both using synchrotron radiation and laboratory-based setups [5-6]. Three-dimensional reconstruction of absorption, phase and dark-field can be achieved with a simple rotation of the sample, without requiring additional movements of the optical elements [7].

All these approaches share a common trait which consists in the use of an absorber, for structuring the radiation field, in order to make the phase modulations introduced by the sample detectable. We see this as a key point to enable sufficiently robust lab translation and scattering retrieval (Fig. 1). This enables a well-defined and high-contrast structuring of the radiation field as well as an accurate modeling of the effects that are related to the simultaneous use of a wide range of energies. Moreover, it can also be adapted for use with detectors featuring large pixel sizes, which could be desirable when a high detection efficiency is important. The details of the designs of the experimental set-ups, the models used for the retrieval and the experimental test for their accuracy will be presented and discussed.

Spatial resolution of the laboratory-based system was experimentally measured to 1.5 microns and the quantitative accuracy was tested against numerical simulations of simple objects. These implementations used high-energy and broadband X-ray beams without this affecting the quality of the images or the accuracy of the retrieval. The polychromatic nature of the beam can be built into the model and accounted for in the retrieval, effectively solving a potential source of problems in the quantitative retrieval of ultra-small-angle X-ray scattering images in the presence on non-negligible absorption.

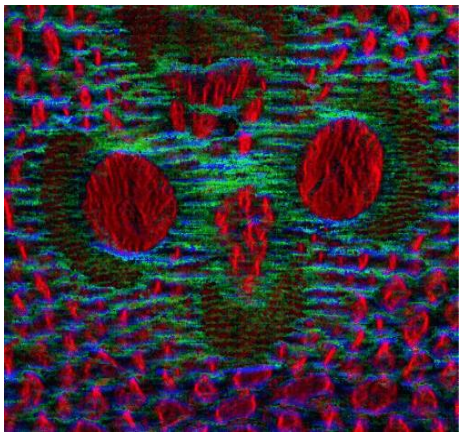

Fig 1: multi-modal image of bamboo wood. R absorption, G scattering, B |refraction|.

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HARD X-RAY MAGNETIC CONTRAST NANOTOMOGRAPHY

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Abstract
Scientific studies of magnetic systems have been limited to surface domain analysis of bulk samples, or thin magnetic films; however the properties of magnetic materials are dominated by their three-dimensional magnetic domain structures. The arrangement and interaction of microscopic magnetic domains determines macroscopic properties, and understanding the nature of magnetic domains will become essential as device features become increasingly smaller and denser.

Using polarized X-rays and tomographic methods we are developing a method for three-dimensional imaging of the magnetic domain structures of bulk magnetic systems, at spatial resolutions better than $(20nm)^3$.

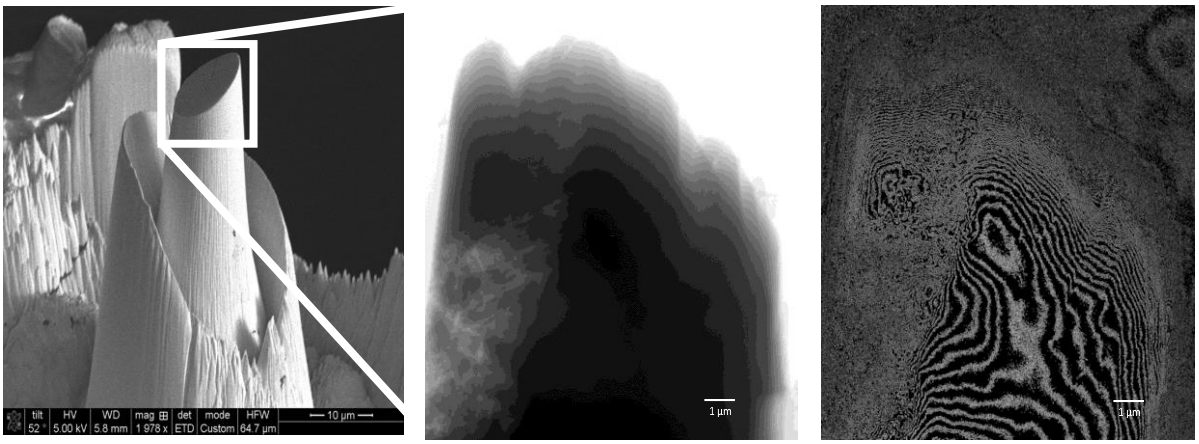

Figure 1: (Left) SEM image of a Focused ion beam milled Gadolinium pillar structure. (Center) Absorption projection image taken of the structure apex with 1 second exposure. (Right) XMCD flipping ratio projection image with edge-enhancement for visual clarity.

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INVESTIGATION OF METAL ASSOCIATIONS IN THE METALLIFEROUS BLACK SHALES OF THE NIUTITANG FORMATION BY X-RAY FLUORESCENCE MICROSCOPY

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Abstract

A transition from oxygen-deficient to oxygenated oceans triggered the evolution of complex multicellular life at the Precambrian-Cambrian boundary, 542 million years ago. At this time, both the evolution of metazoan radiation and the genesis of sub-economic Ni, Mo, As, Rare Earth Elements (REE) and Platinum Group Element (PGE) deposits were triggered in shallow marine environments, including on the Yangtze platform in China (e.g. [1]). This is one of the most enigmatic examples of a sediment-hosted base and precious metal deposit showing an association of ore-grade metals with organic matter (OM). The genesis of this and other strata-bound deposits is poorly understood.

X-ray fluorescence microscopy (XFM) combined with the Maia detector (384 detector array [2,3]) was performed on the Australian Synchrotron XFM beamline [4] to investigate the fine-scale distribution of Ni, As, Au, Ag, Se, V, Zn and U in this rare ore layer, providing further insights into the genesis of this metal accumulation. The new MPDA method [5], which provides rapid quantitative element images and improved spectral deconvolution of pixel spectra, was used in order to obtain accurate element concentrations per pixel. The high-definition images obtained with the Maia detector allow the detection of metal segregation on a wide range of spatial scales.

Phosphorite nodules are mostly surrounded by high concentrations of specific elements (i.e., As) accumulating on the edge of the nodules while V, Zn and Cu appear to be associated with the organic-rich matrix. The strong variations in metal distributions highlight rapid shifts in redox conditions. Hg was also imaged for the first time in the Cambrian metalliferous shales. This use of XFM combined with the Maia detector allows a greater understanding of metal associations in this unique, highly anoxic and sulfidic sedimentary system, in the context of the Cambrian bioradiation of metazoans.

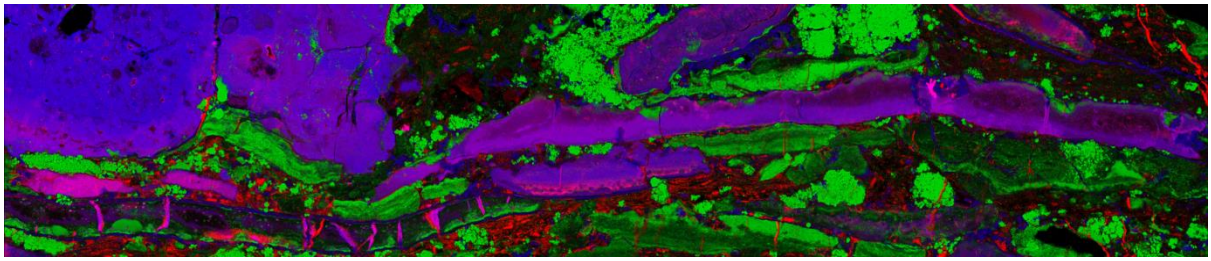

Figure 1: Maia RGB false colour image (Se in red channel, As green, Ni blue) of the metalliferous shale (18.5 keV, XFM beamline) illustrating multiscale compositional complexity.

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ADVANCED ZERNIKE PHASE CONTRAST: A NEW METHOD FOR PHASE CONTRAST IMAGING WITH X-RAY MICROSCOPY

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Abstract

Zernike phase contrast microscopy (ZPC) [1] is a consolidated approach to image weakly absorbing samples. This method, derived from visible-light microscopy, detects the phase shift induced by the sample and enhances that signal relative to the background by suppressing the unscattered wave relative to the scattered one. It has been successfully employed in synchrotron and laboratory X-ray microscopy [2-4].

The ZEISS Xradia Ultra 810 X-Ray microscope employs standard Zernike methods for phase contrast, whereby the sample is illuminated by a hollow-cone beam and then passes the illumination through a phase ring, which is inserted into the beam path after the objective. Using the Ultra 810 microscopy platform, a new Advanced Zernike Phase Contrast (AZPC) approach has been developed, utilizing a patterned phase mask in place of the conventional phase ring.

AZPC represents an imaging method to produce highly artifact reduced ZPC images at equivalent or increased imaging throughput compared to the conventional ZPC method. An example of absorption contrast and AZPC is shown in Fig. 1 and Fig. 2, respectively. AZPC offers an excellent level of detail of physical edges within a sample, which are not as clear in the absorption imaging mode. The reduction in artefacts (halo, shade-off, etc.) typically associated with ZPC will be shown to be reduced with the AZPC method.

A comparative study between absorption, ZPC, and AZPC has been performed and the results show that image resolution is similar amongst the modes with improved contrast to noise ratio from AZPC compared with standard ZPC.

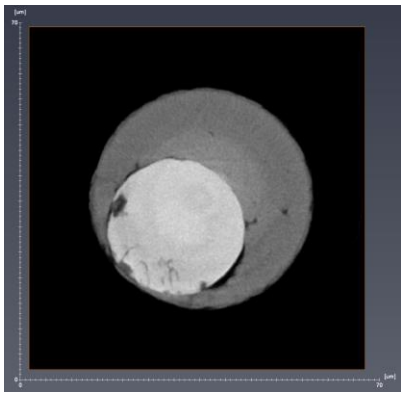

Figure 1: Absorption scan of an Iron-type micrometeorite sample.

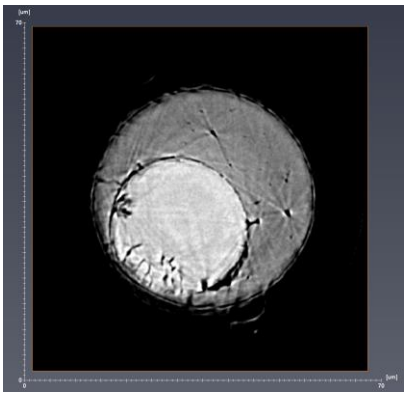

Figure 2: AZPC scan of an Iron-type micrometeorite.

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PTYCHOGRAPHIC IMAGING FOR THE CHARACTERIZATION OF X-RAY FREE-ELECTRON LASER BEAMS

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Abstract

Ptychographic imaging is an established X-ray scanning microscopy technique which exploiting iterative algorithms returns both the complex-valued transmission function of the imaged sample - or object - and the complex-valued function describing the wavefront impinging on it and generating the recorded diffraction patterns - or probe [1]. In the recent years this technique has found applications in the field of beam diagnostics as a suitable means to fully characterize individual pulses generated at X-ray free-electron lasers (XFELs) [2]. In particular we explore the characterization of randomly-stimulated self-amplified spontaneous emission (SASE) pulses on which little knowledge is available to date.

Several approaches have been developed for wavefront characterization at XFELs as this is instrumental in the achievement of the best possible outcome of most of the experiments performed there [3-7]. On the one hand, extremely high power densities and small focal spots are often employed for which it is critical to have an in-depth knowledge of the wavefront in order to choose a plane on which optics-induced aberrations are minimized thus reducing detrimental effects. On the other hand, coherent diffraction imaging (CDI) experiments greatly benefit from a more accurate estimate of the probe as this has an influence on the reconstruction algorithms and the achievable resolution.

Further complexity is added to the problem by the fact that XFEL pulses are all inherently different and often only an averaged characterization of the beam is available. This is especially relevant in the configuration we consider where the pulse-to-pulse variation is expected to be more pronounced. We tackle this problem using a multimodal decomposition approach in which the probe is decomposed into several coherent modes each contributing to a different extent to form the collected diffraction patterns [8]. Further analysis can determine how much each of these modes contributes to each individual pulse thus effectively achieving pulse-to-pulse characterization, giving an unprecedented insight into wavefront fluctuations and advancing the development of online diagnostic techniques for XFEL beams and high-brilliance coherent beamlines in general.

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EXTRACTION OF SCATTERING, REFRACTION AND ABSORPTION PROPERTIES IN TRANSMISSION X-RAY MICROSCOPY

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Abstract

X-ray microscopy with Zernike phase contrast is a three-dimensional high-resolution imaging technology that is very attractive for observation of low-absorption-contrast biological specimens [1, 2]. However, images obtained by using x-ray microscopy with Zernike phase contrast include the object's absorption information and phase information, and these two informations can't be separated and quantified. Furthermore, the scatter information of the sample can't be extracted, which limit x-ray microscopy's application, and the quantitative information of the sample is required. In this paper, a new method was proposed to determine independently the quantitative refraction, absorption and scatter width information of the object. With this method, only several images of the object were needed when the absorption ring was placed in the different positions of the back-focus plane of the zone plate, then the distribution of these three physical informations can be separated and reconstructed. Compared with the existing method, the new method not only simplifies data acquisition procedures, but also realizes to obtain the quantitative refraction, absorption and scatter width information of the object at the same time[3, 4]. Fig.1 shows the reconstructed absorption image, refractive angle image and scatter width image of Tungsten needle particle, which confirm the reliability of this new method. We underline that the new method is highly compatible with future hard x-ray microscopy's applications in biological imaging.

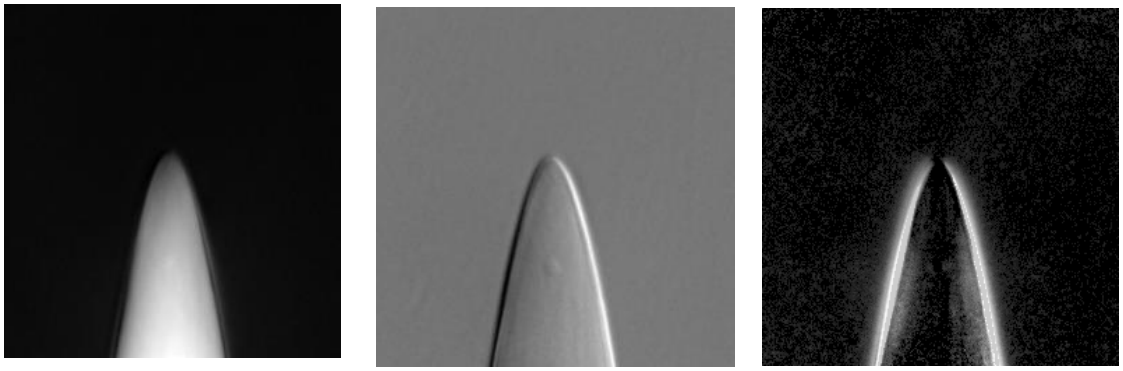

Fig.1 the reconstructed results of Tungsten needle.(a)shows absorption image μt , (b)refractive angle image θ and (c)scatter width image ω

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NANOARPES FACILITY AT DIAMOND LIGHT SOURCE

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Abstract

Angle resolved photoemission spectroscopy (ARPES) is a standard tool for the study of the electronic structure of solids and their surfaces. It is applied to semiconductors and metals for the study of strongly correlated electron systems, the physics of superconductors, charge density waves, and many other topics in material science. With a light spot below 1 micron, this technique acquires spatial resolution [1,2]. Using the scanning probe (microscopy) approach, we could study many systems that are difficult for traditional ARPES, like extremely small samples, samples with inherent phase separation, etc. Furthermore new applications could be made possible, like *in-operando* studies of micro-devices.

The nano-ARPES branch of I05-ARPES beamline of Diamond Light Source has been commissioned recently and is currently seeing its first users. The poster describes the main performance parameters achieved during commissioning, as well as early user experience.

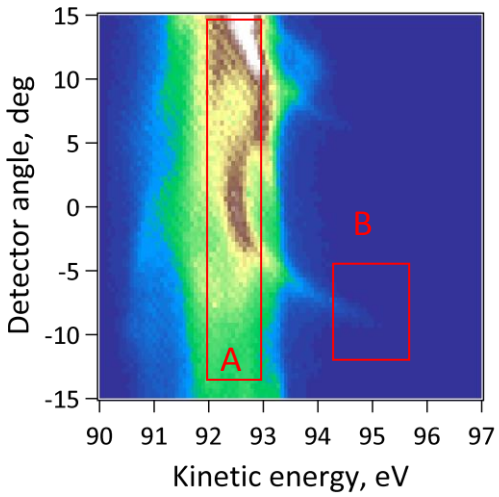

Figure 1: The micro-spot angular dispersion of electrons in graphene on Cu foil

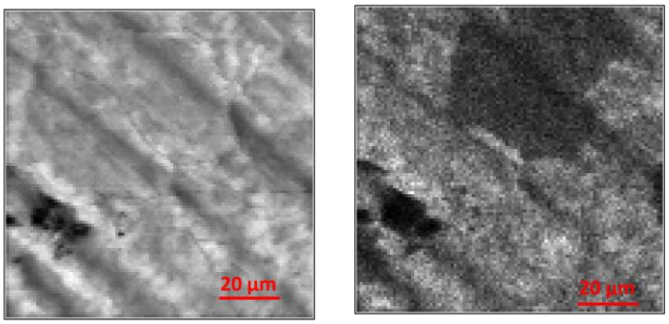

Figure 2: Image of graphene on Cu foil in photoemitted electrons, showing spatial map of electrons emitted in low binding energy core level (A) and near Fermi edge (B)

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USING LAB-BASED MICRO AND NANO COMPUTERISED TOMOGRAPHY TO ACHIEVE THREE PHASE SEGMENTATION OF NI-YSZ ANODE MATERIALS WITH OPERATIONALLY RELEVANT ENVIRONMENTS VIA NOVEL PREPARATION TECHNIQUES

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Abstract

Although offering high efficiencies, low emissions and fuel versatility, solid oxide fuel cells (SOFCs) do not come without limitations; redox cycling, poisoning, carbon deposition, delamination and cracking reduce performance and can lead to cell and stack failure.

Investigations [1, 2] suggest that thermal cycling is the dominant source of SOFC degradation and although microtubular SOFCs have shown good tolerance to thermal shock, rapid thermal cycling remains a challenge for the more widely adopted planar designs due to the thermal expansion mismatch between the nickel (Ni-YSZ) anode and yttria stabilised zirconia (YSZ) electrolyte. Current literature presents a host of electrochemical [3] and microstructural [4, 5] characterisation methods with the intention of increasing our understanding of such complex mechanisms.

This work is focused on achieving three-phase segmentation (Ni-YSZ-Pore) of anode materials with use of lab-based micro and nano-CT in order to achieve repeatable triple phase boundary mapping. Alongside this the authors are working on novel sample preparation techniques in the creation of geometrically ideal samples, which are also capable of withstanding operational temperatures.

Future work will look to combine three-phase segmentation with operationally relevant thermal cycling via homing in on the microstructural changes at the anode-electrolyte interface. Such studies will lead to improved understanding of the effects of geometry and support type of degradation during thermal shock.

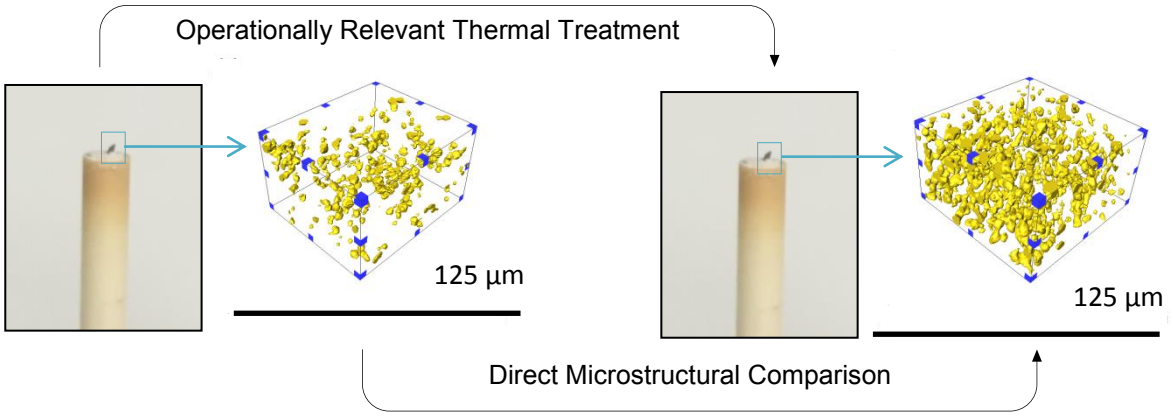

Figure 1: Novel sample preparation technique with pre and post porosity map collected via lab-based X-ray micro computerised tomography for the reduction of a NiO to Ni SOFC anode material

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X-RAY FLUORESCENCE CT BASED ON THREE-DIMENSIONAL RADON TRANSFORM

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Abstract

As one of the most sensitive methods, the synchrotron radiation X-ray fluorescence analysis (SR-XRF) is used widely to observe elements of specimen, chemical valence and electron configuration. The fluorescence X-ray computed tomography (FXCT) which combines compute tomography and SR-XRF is a nondestructive imaging method thanks to powerful penetrability of X-ray [1]. The FXCT can get distribution of elements with high spatial resolution in specimen and is used in many fields such as biology, environmental sciences, archaeology and so on.

Traditional FXCT that based on two-dimensional Radon transformation uses pencil-beam as the exciting light. The specimen rotates around a fixed axis and moves perpendicular to pencil-beam. Detector acquires the line integral of fluorescence data along the pencil-beam. The spatial resolution depends on the diameter of pencil-beam. So a higher spatial resolution can only be achieved by reducing the diameter of pencil-beam. But reducing the diameter of pencil-beam will result in the decrease of luminous flux and low signal to noise ratio (SNR). In other words, spatial resolution and SNR cannot be satisfied at the same time.

To solve the contradiction between spatial resolution and SNR, we proposed to replace pencil-beam with sheet-beam based on the principle of three-dimensional Radon transform [2], see (fig. 1). Detector acquires surface integral data of fluorescence excited by sheet-beam. The specimen rotates around a fixed point in 2π solid angle and moves along the normal of sheet-beam. Every time the specimen rotates, the sheet-beam should scan the whole specimen along the normal. Then we can get a complete three-dimensional Radon transformation data that can be used to rebuild the three-dimensional distribution of elements' concentration in specimen through the three-dimensional Radon inverse transform. At present, the specimen stage has been set up according to the strategy of acquiring data (fig. 2). The verified experiments are about to perform in the near future.

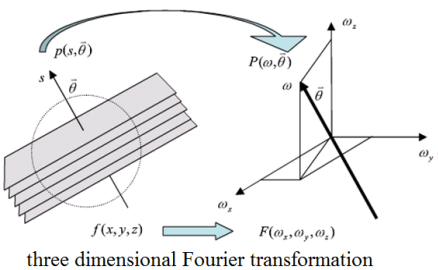

Fig. 1. 3D Radon transform

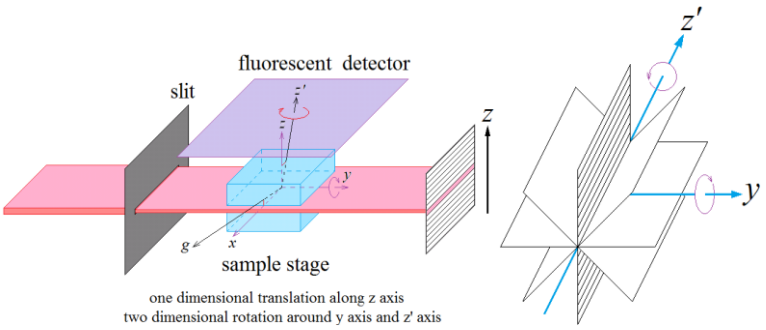

Fig. 2. The optical path layout

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HIGH-RESOLUTION ELECTRONIC AND CHEMICAL IMAGING USING SCANNING ANGLE RESOLVED PHOTOEMISSION

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Abstract

As one of the latest beamline built at the SOLEIL synchrotron source, ANTARES beamline offers a spectroscopic non-destructive nano-probe to study advanced materials. This innovative scanning photoemission microscopy combines linear and angle sweeps to perform precise electronic band structure determination by Nano Angle Resolved Photoelectron Spectroscopy (***Nano–ARPES*** or ***k-microscope***) and chemical imaging by core level detection [1]. The beamline integrates effectively insertion devices and high transmission beamline optics. The beamline is combined with an advanced microscope, which has precise sample handling abilities. Moreover, it is fully compatible with a high-resolution R4000 Scienta hemispherical analyser and a set of Fresnel Zone Plates (FZP) able to focalize the beam spot up to less than 100 nm, depending on the spatial resolution of the selected FZP. Figure 1 depicts some of our findings on few layers of graphene grown on SiC single crystals. The main conceptual design of the beamline and endstation, together with some of the more remarkable results will be presented [2-3].

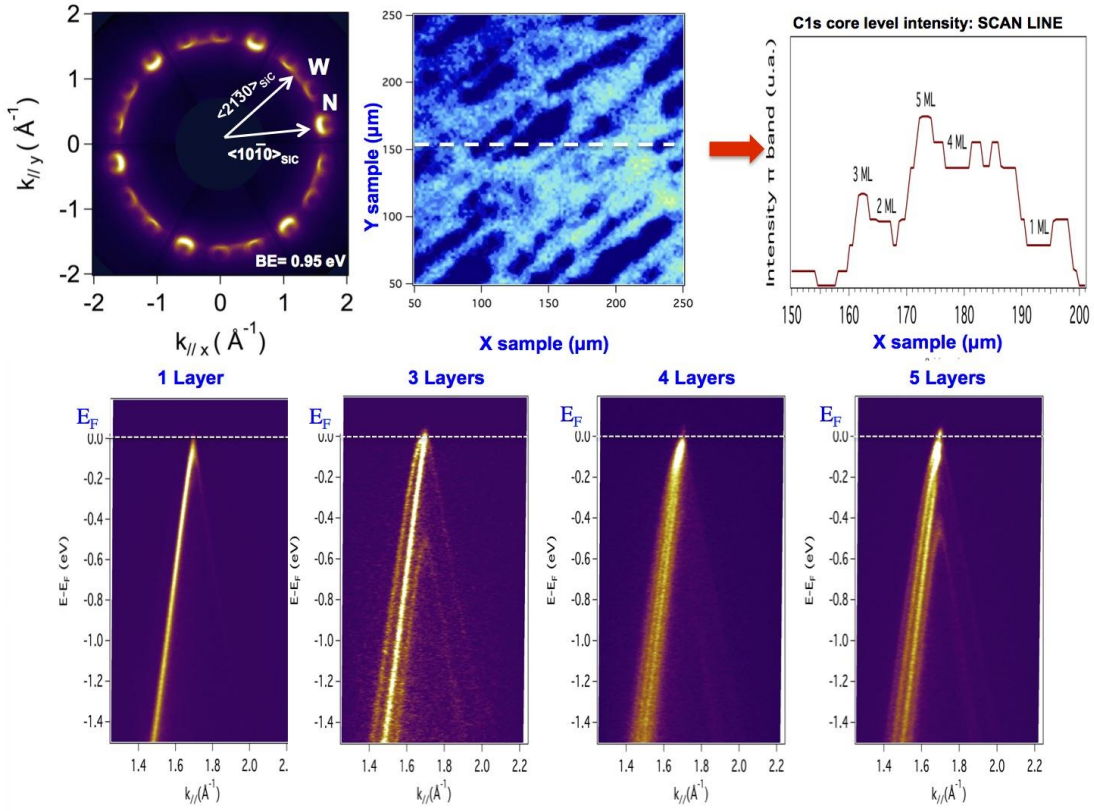

Figure 1: Top panel (From left to right) shows (1) the Fermi surface of a multi-grain few layers of graphene grown on SiC C-face single crystal, (2) an electronic imaging of the states at the Fermi level throughout the surface of the sample and finally (3) a scan line along the white line. The bottom four panels show the high-resolution ARPES bands close to the Fermi level at the areas identified as 1-5 layers of graphene at the electronic image of the top panel.

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FULL-FIELD IN-SITU NANO-TOMOGRAPHY ACTIVITY AT THE ADVANCED PHOTON SOURCE

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Abstract

A new Transmission X-ray Microscope (TXM) [1], optimized for in-situ nano-tomography experiments, has been designed and built at the Advanced Photon Source (APS). The instrument has been in operation for the last two years and is supporting users over large fields of Science, from energy storage and material science to natural sciences.

The flexibility of our X-ray microscope design permits evolutionary geometries and can accommodate relatively heavy (up to 5 kg) and bulky in-situ cells while ensuring high spatial resolution, which is expected to improve steadily thanks to the support of the R&D program led by the APS-Upgrade project on Fresnel zone plates (FZP). The robust sample stack, designed with minimum degrees of freedom shows stability better than 4 nm rms at the sample location. The TXM operates with optics fabricated in-house. Spatial resolution ranging from 60 to 20 nm has been demonstrated. In parallel, efficiency is being improved with opto-mechanical engineering (active FZP stacking system) and software developments (more efficient reconstruction algorithms like Tomopy [2] or Timbir [3] combined with different data acquisition schemes), enabling 3D dynamic studies when sample evolution occurs within a couple of tens of seconds.

Several applications will be presented, covering in-situ experiments with battery, fuel cells or diamond anvil cell, or ex-situ experiment on low-Z material like graphite electrode or soft tissues with Zernike-type phase contrast, but also correlative microscopy experiments between TXM and Scanning Electron or Transmission Electron Microscopy.

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IMPLEMENTATION OF PTYCHOGRAPHIC IMAGING AT MAXYMUS X-RAY MICROSCOPE

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Abstract

Ptychography is a recently developed X-ray diffraction imaging technique that allows to solve phase problem gaining spatial resolution down to few nanometers [1,2] with chemical [2] and magnetic [3] contrast sensitivity. During the last year MAXYMUS microscope, operated by the MPI for Intelligent Systems at the Bessy II synchrotron (Berlin, Germany), has been extensively upgraded for the purposes of implementing ptychographic imaging. The drastic increase in resolution synergizes with the unique capabilities of MAXYMUS microscope for high brightness, selectable polarization of X-ray light, variable magnetic fields (up to 250 mT) and cooling temperatures (down to 75 K).

The most important part of MAXYMUS upgrade included the acquisition of a fast in-vacuum CCD camera developed by PNSensor. This instrument allows spatial and energetically sensitive single photon detection in the soft X-ray energy range at rates up to 1000 fps with high quantum efficiency >70% (for E>300eV), 48 μm pixel size and a RMS noise per pixel less than $3e^-$ [4]. High dynamic range and fast read out rate allow efficient representation of diffraction data during ptychographic scanning. Also high efficiency ion beam lithography FZPs have been produced in our department specially for the purposes of ptychographic imaging.

We will present the results of commissioning of the new in-vacuum CCD camera and the implementation of ptychographic approaches at MAXYMUS microscope. The first ptychographic reconstructions with advanced resolution in comparison with conventional STXM imaging will be demonstrated (Fig.1).

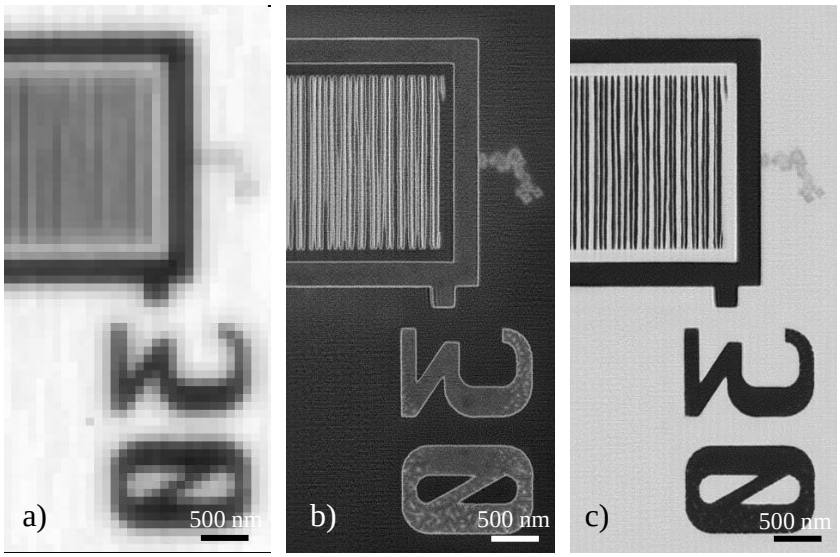

Figure 1: Zeiss resolution target with the smallest line width of 30 nm imaged at energy 800 eV using FZP with spatial resolution of 152 nm: a) STXM image; b) imaginary part of ptychographic reconstruction; c) real part of ptychographic reconstruction.

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A ITERATIVE METHOD FOR PROPAGATION-BASED PHASE CONTRAST IMAGING

A. J. Carroll[1], G. A. van Riessen[1], E. Balaur[1,2], I. P. Dolbnya[3], G.N. Tran[1], A. G. Peele[1,2,4]

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Abstract

We present simulation and experimental data demonstrating a propagation-based iterative phase contrast imaging method that allows quantitative determination of the thickness of an object given the refractive index of the sample. It can be used with both monochromatic [1] and broadband polychromatic [2] source spectrums.

The iterative method can be used with a range of different Fresnel propagators [3], allowing for phase contrast imaging experiments in regimes where the Transport of Intensity Equation (TIE) [4] and Contrast Transfer Function (CTF) [5,6] begin to fail. Further, our work has shown the iterative scheme allows higher-resolution imaging when compared to the TIE and less artefacts in the reconstruction when compared to the CTF.

The iterative method presented shows promise towards laboratory-based polychromatic iterative phase contrast imaging.

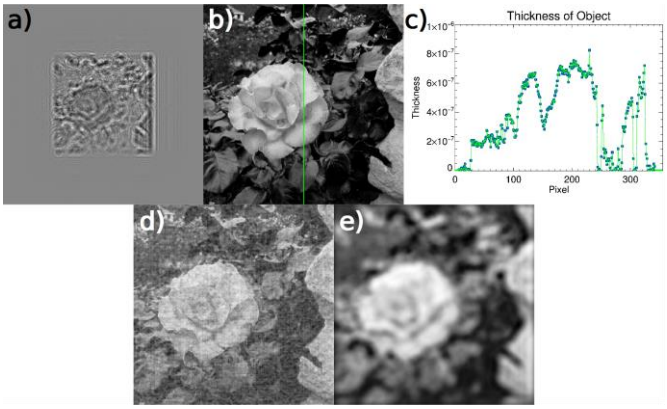

Figure 1: Demonstration of a monochromatic iterative phase contrast reconstruction. a) shows the simulated phase contrast image used for this example. b) shows the reconstructed object using the iterative method. c) is a line out across the green line seen in b). The blue line in c) represents the objects simulated thickness, while the green line represents the iterative reconstruction of the thickness. d) shows the CTF reconstruction. The CTF phase condition is violated under the simulation conditions employed. e) shows the TIE reconstruction. The TIE is valid under the simulation conditions employed.

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SOFT X-RAY COHERENT SCATTERING AND PTYCHOGRAPHY USING KB FOCUSING OPTICS

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Abstract

Soft X-ray spectromicroscopy is a powerful tool which can provide 2D or 3D imaging with chemical, electronic and structural information at the nanoscale. In this presentation, we will present the development of a new soft X-ray spectromicroscope system based on coherent scattering. Using Kirkpatrick-Baez (KB) focusing optics, a soft X-ray elliptically polarizing undulator beamline at the Taiwan Photon Source (TPS) delivers coherent photon flux in the order of 10^{11} $s^{-1}(0.02\%BW)^{-1}$ with a beam size of 3 μm by 3 μm in the energy range from 400 eV to 1200 eV. The spectromicroscope is attached to an in-vacuum diffractometer with two principal rotation axes, 2θ (detector) and θ (sample). The system operates in the transmission or the Bragg refection geometry for ptychography and Bragg coherent diffraction imaging (CDI), respectively. Through the resonance of soft X-ray scattering, a 2D area detector in the reflection geometry is used to image a superstructure diffraction to obtain the spatial information of spin, charge and orbital ordering. For ptychography, the probe is defined by a 0.5-μm pinhole placed 1.5 mm upstream the object and a special platform equipped with sample and pinhole scanning stages is developed.

The presentation will discuss the design and expectation of the instrumentation, test results and the data analysis for imaging strongly correlated electron materials with spectroscopic information at the nanoscale. Future improvements to realize tomographic imaging will also be addressed.

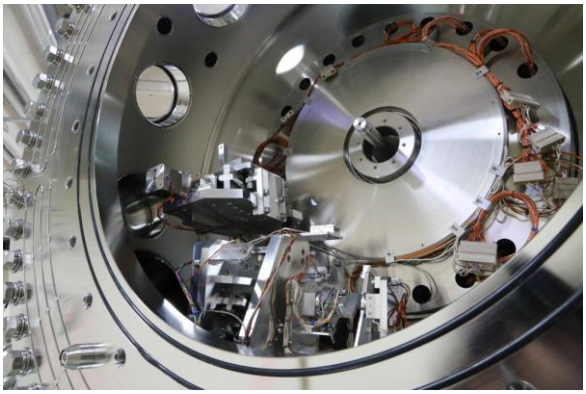

Figure 1: The TACoDE system with two principal rotation axes at the TPS.

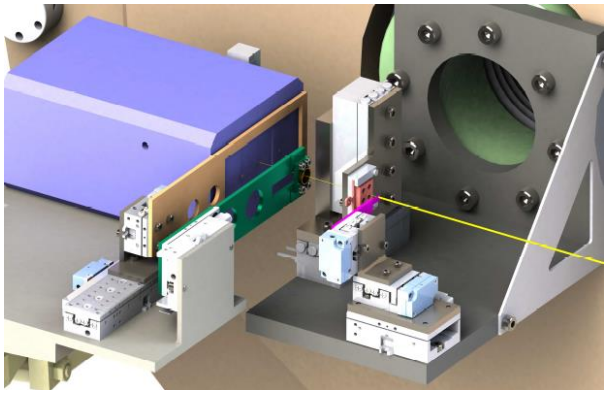

Figure 2: Design of the ptychography microscope.

SIGNAL-TO-NOISE CRITERION FOR FREE-PROPAGATION IMAGING TECHNIQUES AT FREE-ELECTRON LASERS AND SYNCHROTRONS

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Abstract

The resolution of conventional X-ray microscopy techniques at synchrotrons is limited by the radiation damage and is considered to be around 10 nm for biological materials [1]. Free-electron lasers (FEL) experiments overcome this limit by means of ultraintense and ultrashort pulses [2], making the resolution limited only by the the number of photons available in a FEL pulse.

With this work we will shed light on the following crucial questions regarding both FEL and synchrotron experiments: a) Is the resolution achievable on a single feature hampered by the feature embedded in a larger object? b) Do different imaging techniques exhibit different efficiency? We will address these issues with the signal-to-noise criterion, introduced in [3], and based on a Gaussian scatterer model, which predicts whether a feature of given size and scattering strength placed inside a larger object can be better retrieved either by projection microscopy (PM) in real space or coherent diffraction imaging (CDI) in Fourier space.

Our criterion, quantitatively validated through simulations, predicts that PM requires less photons per unit of area (fluence) than CDI, as shown in Fig. 1. Furthermore, PM has higher sensitivity compared to CDI since it is capable to retrieve smaller phase contrast, as depicted in Fig. 2. For this reason, PM results more suitable to identify weaker and smaller features than CDI. Our criterion explicitly accounts for the size of the full object, and predicts deterioration of the imaging performance with increasing the object size in contrast with previous studies. We will describe how to use this criterion in order to design optimized imaging experiments and feasibility studies at FEL and synchrotron facilities.

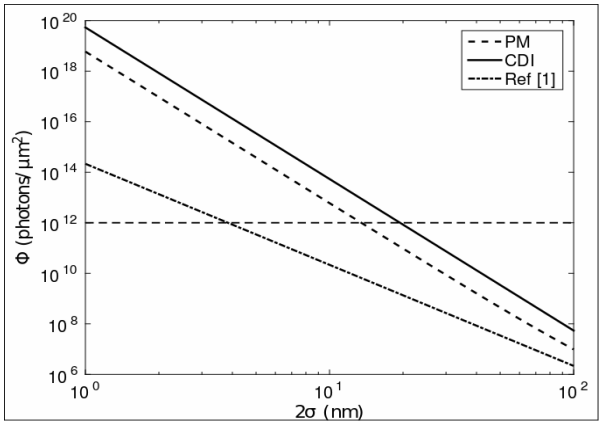

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Figure 1: Required imaging fluence as a function of the feature size for PM, CDI and the criterion of Ref [1].

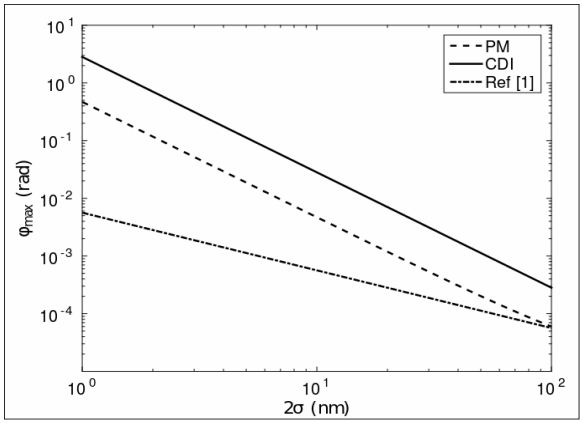

Figure 2: Phase-contrast sensitivity as a function of the feature size for PM, CDI and Ref [1],

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EVALUATION OF TALBOT-BASED X-RAY MICROSCOPE SYSTEM WITH WIDE FIELD OF VIEW

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Abstract

An X-ray imaging microscope using Fresnel zone plate (FZP) in combination with the Talbot interferometer can generate phase tomography with high spatial resolution and wide-field [1]. An application of Talbot-based X-ray microscopy with large-field of view (~300 μm) was reported earlier [2, 3]. The experiments were performed using 9 keV at undulator beamline 20XU, SPing-8, Japan and Talbot interferometer consisted of a π/2 phase grating and an amplitude grating both having 6 μm period. With x20.9 times magnification, a spatial resolution was obtained as 1.5 μm in the tomogram of bone specimens.

To elucidate bone formation mechanism more precisely, we need to improve the spatial resolution by keeping large-field of view, which would easily be improved by increasing the magnification. In this research work, same experimental conditions were used at long-length beamline 37XU, SPring-8 to compare the spatial resolution by keeping ~300 μm field of view. The beamline 37XU have 28 m length propagation space between two experimental hutches, hence the microscope optics with x100 times magnification can be constructed. That is about 5 times greater than optics constructed at 20XU. X-ray imaging detector using coupling system which had 14.8 μm effective pixel size, on the objective plane effective pixel size was 0.148 μm obtained. The quantitative measurements of the differential phase shift and phase-CT were performed using the fringe-scanning method. As a result, the spatial resolution was evaluated as 200 nm L&S in the 2D image of test chart (Figure 1) and as 600 nm from the profile across the glass capillary surface in the tomogram (Figure 2).

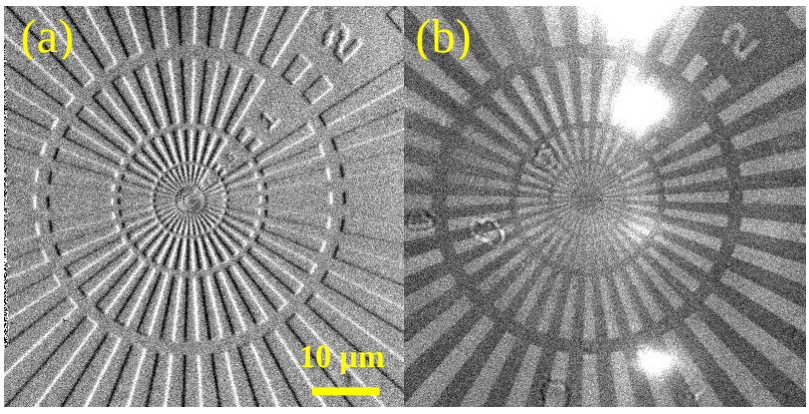

Figure 1: Image of the Ta test chart: (a) differential phase image and (b) absorption image.

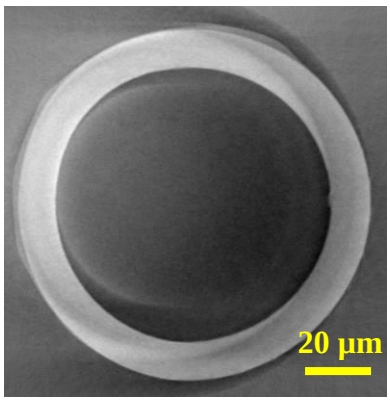

Figure 2: Differential phase tomogram of a glass capillary.

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NEAR FIELD DIFFRACTION IMAGING FROM MULTIPLE DETECTION PLANES

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Abstract

We present diffraction imaging results obtained from multiple near-field diffraction constraints [1]. An iterative phase retrieval algorithm was implemented that uses data redundancy achieved by measuring near-field diffraction intensities at various sample-detector distances. The procedure allows for reconstructing the exit surface wave of a sample within a multiple constraint satisfaction framework [2] neither making use of a priori knowledge as enforced in coherent diffraction imaging nor exact scanning grid knowledge as required in ptychography. We also investigate the potential of the presented technique to deal with broadband radiation as important for potential application in diffraction imaging by means of tabletop EUV and X-ray sources [3].

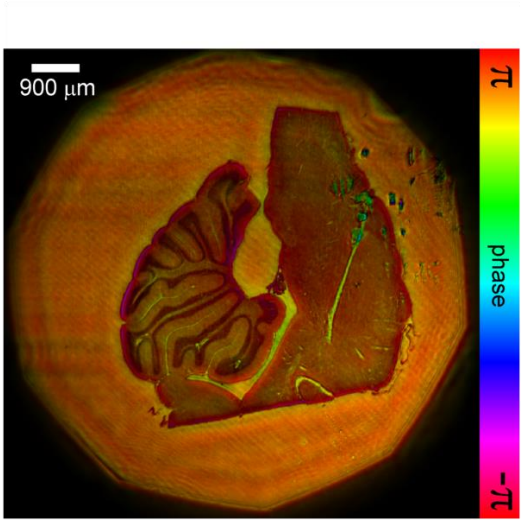

Figure 1: Hue-brightness plot of a histological sample reconstruction (mouse brain). Hue depicts phase, brightness represents absorption of the sample. The white and colored bars indicate length and phase, respectively.

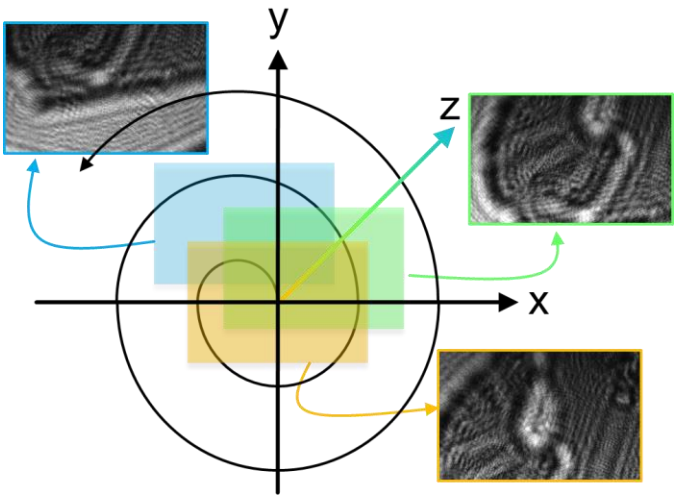

Figure 2: 105 near-field diffraction patterns at distances 200 mm to 450 mm downstream the sample were measured (λ=488 nm). A coregistration procedure automatically stitches overlapping regions in the diffraction data.

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X-RAY NANO-MICROSCOPY AT DIAMOND I13-2 BEAMLINE FOR THE INVESTIGATION OF BRAIN TISSUES

A. Khimchenko[1], G. Schulz[1], S. E. Hieber[1], H. Deyhle[1], I. Zanette[2], C. Bikis[1], M.-C. Zdora[2, 4], A. Hipp[3], J. Vila-Comamala[2], B. Müller[1]

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Abstract

Optic microscopy methods for biological cells visualization, such as fluorescence and electron microscopy, are usually limited to 2D investigations and frequently require extensive and time-consuming sample preparation. Hard X-ray phase-contrast micro-tomography (PC-µCT), on the other hand, provides sufficient spatial and density resolution for the visualization of individual cells [1] using rather simple specimen preparation procedures. Several PC-µCT imaging methods are available, such as X-ray grating interferometry (XGI) using a single- or double-grating setups or propagation-based phase tomography using single-distance phase reconstruction algorithms (SDPR), enabling to enables to compliment the higher contrast data by higher spatial resolution [2]. The present approach allows the three-dimensional characterization of sub-cellular structures, with details comparable to the histology and could become complimentary or alternative to it. In this study, we demonstrate three-dimensional characterization of a human cerebellum autopsy specimen by PC-µCT using synchrotron radiation at the beamline I13-2 (Diamond Light Source). This enables us to visualize not only the *Stratum moleculare* and *Stratum granulosum*, but also the individual cells (Purkinje, stellate and granule cells) without staining. By comparing images from SDPR with complimentary single- or double-grating setups [2], we conclude that SDPR can obtain useful information for pathological applications providing the ideal compromise between superb resolution and contrast. Although SDPR has a reduced sensitivity compared to XGI, it is sufficient to visualize the features of interest and phase-wrapping artefacts are avoided. Thus, we demonstrate an approach for visualization of cells within soft tissues to extend the histology in pathological, biological and biophysical applications with a substantial informational gain (3D data) and the potential of automation of quantification tasks.

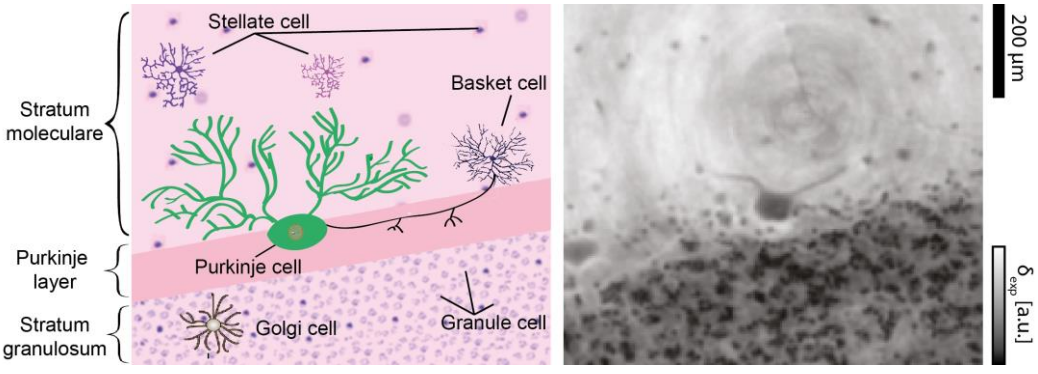

Figure 1: Graphical representation of the cerebellar cortex, left, and selected part of SDPR (right) slices (experimental results obtained at beamline I13-2 with an effective pixel size of 1.1 µm) of formalin-fixed paraffin-embedded human cerebellum highlighting the details of the brain micro-anatomy down to the sub-cellular level.

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SMALL ANGLE X-RAY SCATTERING WITH EDGE-ILLUMINATION

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Abstract
In phase contrast x-ray imaging sensitivity to sub-pixel sample features provides access to information on microscopic scales with macroscopic pixel sizes, which offers the opportunity for dose reduction and/or faster scans by exploiting larger pixel sizes. Edge-illumination (EI) is a non-interferometric and incoherent phase contrast x-ray imaging technique that utilises two aperture masks as optical elements to provide sensitivity to sub-pixel features [1].

We will present a method to obtain the angular-resolved small angle x-ray scattering distribution with EI, which extends the number of complementarity contrasts related to sub-pixel features from previously three to ten and more [2]. We demonstrate the complementarity of the newly available contrasts with a moment analysis of scattering distributions provided by different materials in powder form (Figure 1). Thus, the proposed approach provides the opportunity to exploit extended sub-pixel contrasts in biomedical research, materials science and security screening.

As an example application in a prominent area, we investigated the higher order moments of retrieved scattering distributions of excised murine lung samples in the context of pulmonary emphysema diagnosis (Figure 2). We used the ratio of the 4th and 2nd moment as an illustration model for the potential diagnostic power and found that the ratios of a control and an emphysematous lung differ by more than 40 confidence intervals averaged over the entire samples. Further, we show that the properties of retrieved scattering distributions are consistent with the larger average feature sizes of emphysematous lungs compared to healthy ones. Thus, we conclude that the proposed method has a high potential for the diagnosis of early stage pulmonary emphysema with low dose.

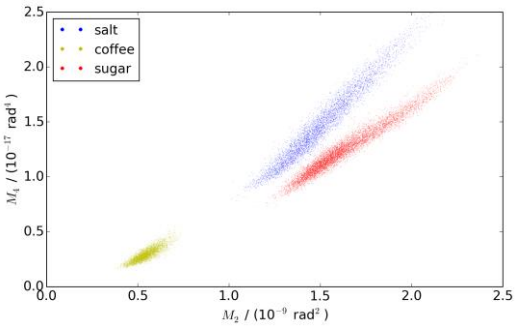

Figure 1: Complementarity of the 2nd and 4th moment of retrieved scattering distributions for different powder materials.

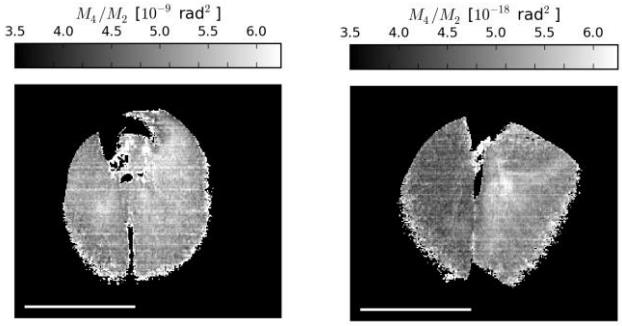

Figure 2: Differentiation of control and emphysematous murine lungs with extended sub-pixel contrasts provided by EI.

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RESONANT SOFT X-RAY SCATTERING AND PTYCHOGRAPHY WITH THE HORST CHAMBER

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Abstract

The holographic X-Ray scattering apparatus - HORST [1] is a vacuum chamber developed for scattering experiments at synchrotrons and free-electron lasers. The flexibility of HORST enabled the usage for different experiments like holography, ptychography or small angle X-Ray scattering on biological samples. Several motorized long range stages for detector sample and pinhole in combination with piezo-electric actuators provide the framework to successfully operate the different imaging methods. Recent upgrades enable soft X-ray ptychography to image nonperiodic objects at high spatial resolution [3]. Resonant experiments provide chemical information which can be exploited for polymer systems, catalysts, and frozen-hydrated biological specimen using a cryogenic sample environment [2]. As a model system for chemical contrast in mixed - compound samples, we exploited spectral differences of PMMA and SiO2 near the oxygen K edge [4]. The concept is currently extended towards imaging catalysts and biological samples.

a b

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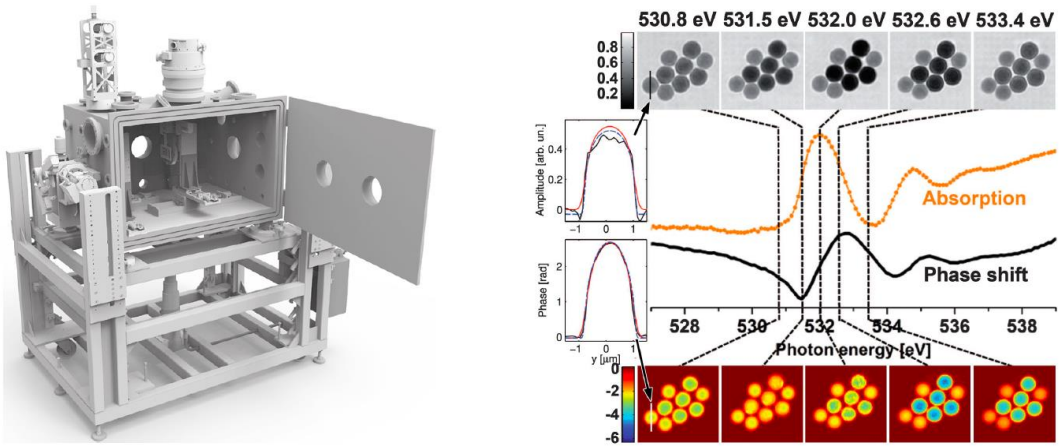

Figure 1: (a) X-ray scattering chamber HORST. (b) Resonant ptychographic reconstruction of the amplitude and the phase of polystyrene beads with different chemistry.

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EXPERIMENTAL 3D COHERENT DIFFRACTIVE IMAGING FROM PHOTON-SPARSE RANDOM PROJECTIONS

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Abstract

Single Particle Imaging at X-ray Free Electron Laser Sources requires collection of hundreds of thousands of diffraction patterns from a continuous stream of reproducible particles [1]. As the particles arrive at the interaction region in random 3D orientations, the 3D intensity has to be assembled in Fourier space from the typically very noisy patterns, before the phasing step can be performed to obtain the real-space structure.

In order to advance XFEL-based SPI to the resolution level required to solve the structure of biological macromolecules, a great variety of experimental and algorithmic challenges have to be overcome, such as sample injection, experimental background minimization, identification of hits, orientation determination, and density reconstruction [2]. Despite many recent advances, an experimental demonstration for orientation determination of SPI data in the very relevant weak scattering limit (<~100 scattered photons per pattern) is still outstanding.

We report here on an experiment at a synchrotron source in which a small (<1 μm) lithographically produced particle has been illuminated with a coherent synchrotron beam. This way, hundreds of thousands of very weak diffraction patterns in hundreds of particle orientations have been collected.

We will show how these data, without explicit knowledge of the individual frame orientations and with less than 100 photons per pattern, can be used to reconstruct a 3D diffraction volume in Fourier space using the Expansion-Maximization-Compression algorithm [3]. This diffraction volume is then used to reconstruct the real-space density of the particle by conventional iterative phase retrieval.

The influence of various levels of experimental background noise will be discussed.

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X-RAY MICROSCOPY IN FOUR DIMENSIONS

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Abstract

X-ray microscopy with high temporal and spatial resolution can unravel dynamic processes at the nanoscale under complex *in operando* conditions. Possible examples range from chemical reactions, flow phenomena, phases transitions in complex fluids and colloids, nanoscale devices and machines to living organisms.

In order to record tomographic projections at a sufficiently high rate, 4D microscopy is best performed with full field techniques, avoiding the overhead in scanning-based x-ray microscopy such as STXM or ptychography.

In this presentation we show time-resolved phase contrast x-ray imaging and microscopy based on free propagation, fast tomographic reconstruction, optimized illumination and object reconstruction. Starting from our previous work on combining phase retrieval and tomographic reconstruction [1,2], we discuss how (a) the solution of the phase problem, (b) the numerical implementation of propagation and (c) the tomographical reconstruction changes in a higher dimensional setting.

To this end, we present first time-resolved phase contrast movies of simple objects changing their 3D structure during a chemical reaction. Finally, we discuss tomography at 20 - 100 nm voxel size, see the example in Fig.1, and present the current status for high resolution time-resolved tomography at the dedicated waveguide imaging endstation GINIX at the P10 beamline of PetraIII/Desy.

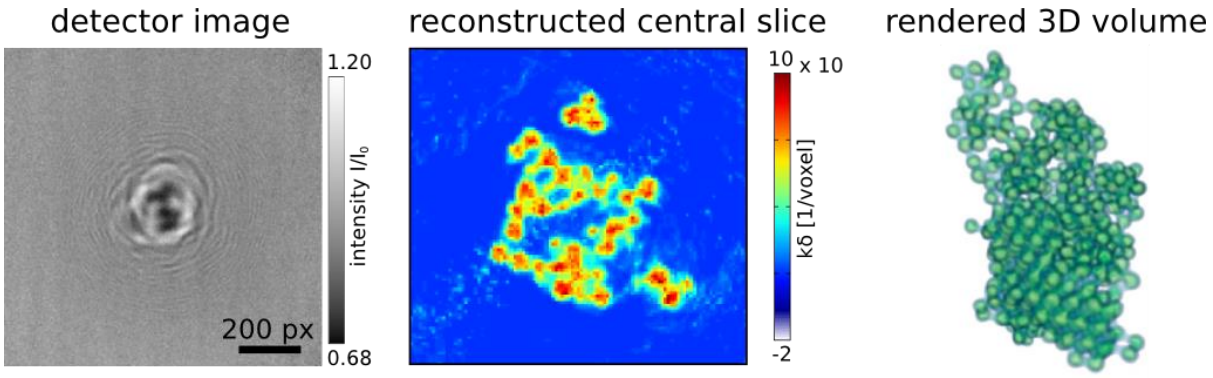

Figure 1: Holographic 3D tomography of a colloidal crystal consisting of polystyrene spheres with a diameter of 415 nm. The 3D volume was reconstructed from 249 detector images with an effective pixel size of 29 nm (photon energy: 7.9 keV, 7° missing wedge). The middle image shows a central slice of the reconstructed volume, where the position of each colloid can be determined, leading to the rendered 3D volume in the right image.

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COMPARISON OF A CMOS- AND A CCD-BASED CAMERA SYSTEM FOR GRATING-BASED PHASE-CONTRAST TOMOGRAPHY

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Abstract

Phase-contrast imaging has proven to be a valuable tool for the investigation of weak absorbing materials like soft tissue, due to its increased contrast compared to conventional absorption-contrast imaging

In addition to the propagation-based phase contrast, grating-based phase contrast is one of the two most widely used phase-contrast techniques at synchrotron radiation sources.

The advantage of the grating interferometer lies in the possibility to retrieve quantitative values of the refractive index. However, this comes with the disadvantage of a longer measurement time as for each projection at least four frames have to be taken.

We will present our implementation of a single-grating interferometer for high-resolution phase-contrast measurements at the PETRA III beamlines P05 and P07 operated by the Helmholtz-Zentrum Geesthacht. The microtomography systems are designed to easily switch between two permanently installed camera systems. Therefore, we were able to perform the same measurement of a test phantom with both a CCD-based camera and a CMOS-camera. The CCD-sensor shows a higher dynamic range and photon sensitivity, whereas the CMOS-sensor gives the benefit of a much shorter readout time and a smaller pixel size.

Based on the evaluation of these measurements we will give a comparison of the performance of the two systems, focusing on acquisition time, signal to noise ratio and accuracy of the quantitative values.

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TABLE-TOP COHERENT DIFFRACTIVE IMAGING – TOWARDS SUB-10 NM RESOLUTION

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Abstract

Coherent diffractive imaging (CDI) and associated techniques nowadays enable imaging with few-nanometers resolution by employing X-ray radiation provided either by synchrotron or free-electron laser facilities [1]. During the recent years, laser driven table-top sources of coherent XUV and X-ray radiation based on high harmonic generation (HHG) have seen enormous progress. Today, they can provide photon fluxes similar to beamlines at third generation synchrotrons (up to 10^{13} photons/s) in the XUV [2] combined with excellent coherence and femtosecond pulse durations, which makes them highly attractive for high-resolution imaging. In this contribution we present CDI experiments performed with a high-photon flux 18 nm wavelength XUV source based on high power femtosecond fiber laser technology. We demonstrate the recording of CDI images of simple structures with a high numerical aperture (NA=0.7). The good contrast and signal-to noise ratio promises sub-wavelength resolutions close to the Abbe-limit, similar to previous demonstrations at longer wavelengths [3]. The Abbe-limit for our particular setup is 13 nm and first successful reconstructions indeed indicate sub-20 nm resolution, which represents a record value for table-top CDI experiments. Due to the high-photon flux of our source such high-NA diffraction patterns are recorded within a few-minutes – and ~60 nm resolution can be achieved within 1 s. Due to the ever rising power of laser driven XUV sources, we expect a further reduction of integration times and the possibility of 3D tomography in future. In addition, the extension of available photon energies spanning across several absorption edges with significant flux will allow for material and oxidation state sensitive imaging. We will present the latest progress in power scaling of shorter wavelength HHG-based sources, which will enable table-top imaging within the silicon–window at ~90 eV and the water-window at ~300 eV. In this regard, we will present first CDI results at 8nm wavelength aiming at sub-10 nm resolution.

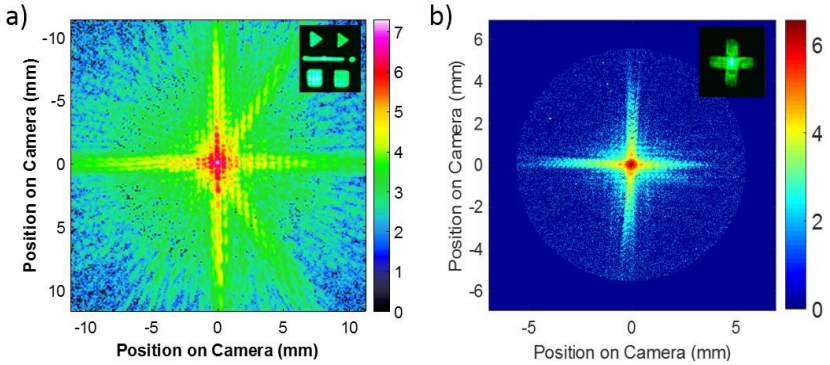

Figure 1: a) Recorded diffraction patterns with a) 18 nm and b) 8 nm wavelength. Insets: corresponding reconstructed real space images (brightness: amplitude).

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SIMULTANEOUS XRF AND PTYCHOGRAPHIC IMAGING USING A MAIA AND EIGER DETECTOR AT P06, PETRA III

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Abstract

While spatially resolved X-ray fluorescence (XRF) measurements provide information on element distribution, it can not compete with the spatial resolution obtained by ptychographic imaging, which in turn can not easily provide chemical information on multiple elements [1-2]. Besides this complementary nature of the two methods, their measurement schemes allow for simultaneous data acquisition [3]. By utilising the newest detector technology, the Maia detector for XRF and the Eiger X 4M for ptychography, simultaneous measurements with dwell times in the millisecond range come within reach.

Besides the technological challenges, the advantages and drawbacks of such a simultaneous detection scheme will be discussed. Results will be shown from the technical ability point of view using a siemens star reference sample, as well as from an application point of view based upon the imaging of fluid catalytic cracking (FCC) catalyst particles. These particles have been studied using several techniques, such as full-field transmission X-ray microscopy (FF-TXM) [4] and XRF tomography [5]. Correlating datasets that originate from different experiments has always been difficult since the data were not acquired simultaneously. Furthermore, the difference in spatial resolution between FF-TXM and XRF tomography complicates the alignment procedure between datasets. With the setup presented here no additional post-experimental alignment of morphological and chemical information will be required due to the simultaneous acquisition of both XRF and ptychographic data,.

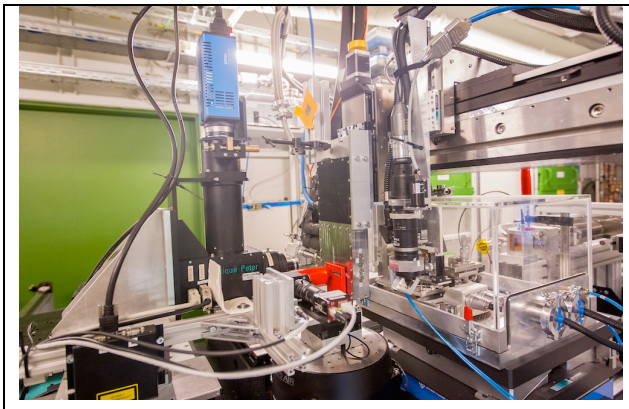

Figure 1: An overview image of the P06 microprobe, showing the Maia detector.

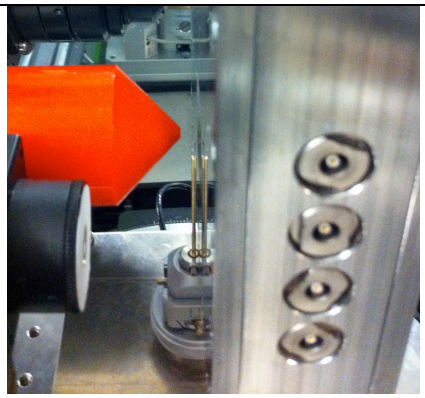

Figure 2: Showing an FCC particle (≈ 50 µm) mounted in a Kapton capillary in front of the Maia detector.

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DIFFRACTION-LIMITED MEASUREMENTS USING PTYCHOGRAPHY AT THE HARD X-RAY NANOPROBE BEAMLINE AT NSLS-II

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Abstract

The nano-focused beam produced by the Hard X-ray Nanoprobe (HXN) beamline provides a unique analytic tool that enables multi-modality measurements with a variety of contrast mechanisms simultaneously [1,2,3]. The successful implementation of ptychography technique further complements the versatile tool suite with the capability of achieving diffraction-limited spatial resolution. In this presentation, we will discuss precise beam characterizations using ptychography with 1D foci from flat/wedged multilayer Laue lenses (MLLs) [4,5], 2D foci from crossed MLL pairs [1] and zone plates. The combination of recovered high-resolution absorption- and phase-contrast images enhances detection sensitivity of light elements such as in battery materials. Ptychography measurements on two battery sample systems will be presented. We will discuss the opportunities and challenges on improving the performance [6,7] and applying ptychography method for in-situ investigations [8].

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CRITIR – DIRECT TOMOGRAPHIC 3D RECONSTRUCTION OF THE COMPLEX REFRACTIVE INDEX

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Abstract

Propagation based X-ray phase contrast tomography (XPCT) is widely used to reconstruct the 3D complex refractive index distribution of a sample from single distance measurements at multiple view angles of the object. In contrast to conventional X-ray computed tomography (CT), XPCT can be used at higher cone beam magnifications, and it also results in higher image contrast. However, conventional methods of reconstruction for XPCT typically assume the near-field condition for diffraction that limits the magnification and contrast [1]. The Fourier methods do not assume the near-field condition but they tend to amplify reconstruction noise [1]. This limits the variety of samples that can be imaged using XPCT.

We present an algorithm called complex refractive index tomographic iterative reconstruction (CRITIR) [2] that can reconstruct an object without making any approximations about the physics of X-ray propagation. CRITIR estimates the complex index of refraction such that it agrees with the measurements as modeled using the theory of Fresnel propagation while simultaneously satisfying the sparsity constraints enforced by a prior model. CRITIR is designed to work within and beyond the near-field diffraction region unlike the conventional reconstruction methods.

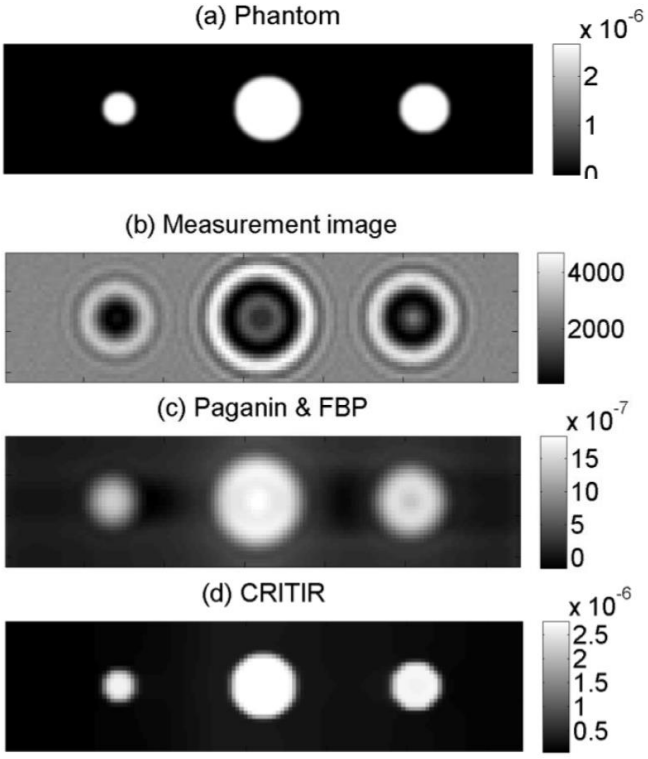

Figure 1: Comparison of reconstruction of latex spheres using the conventional method and CRITIR.

In Fig. 1, we compare simulated data reconstruction of latex spheres using CRITIR with the conventional method of Paganin's phase retrieval and filtered back projection (FBP). Paganin's method assumes the near-field condition for diffraction. Fig. 1(b) shows simulated data that was generated beyond the near-field. The simulated values of the X-ray energy and the object to detector distance are 3keV and 400mm respectively. By comparing Fig. 1 (c, d) with Fig. 1 (a), we can see that CRITIR accurately reconstructs the object while the conventional method results in severe blurring of edges and reconstruction artifacts. CRITIR will enable us to perform experiments that have not been possible until now due to the various approximations made by the conventional methods.

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LOW-DOSE, HIGH-RESOLUTION AND HIGH-EFFICIENCY PTYCHOGRAPHY AT STXM STATION OF SSRF

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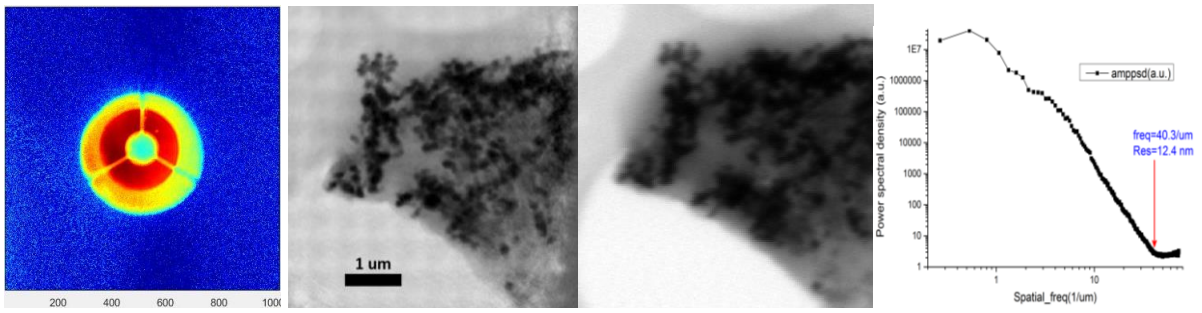
Abstract

The radiation damage is the key factor that limits the achievable resolution for biological samples in coherent diffraction imaging (CDI). A ptychographic CDI (PCDI) platform with relatively low dose, high resolution and high efficiency have been developed at STXM endstation of SSRF. Compared to other FZP-based PCDI setups, we used a much larger illumination spot with 3-5 μm diameter by moving samples downstream from ZP focus, and a larger step size of 500-1000 nm in our platform, which would result in fewer scanning positions and lower radiation dose when imaging the same sample area, although the larger light spot divergent from FZP focus will lead to a more difficult reconstruction due to the highly curved wavefront of the probe. By combining the multimode reconstruction method with the position-correction algorithm in our home-developed ptychography reconstruction software, we can obtain high-quality images from our PCDI platform with much lower radiation dose and less data acquisition time. In the demonstration experiment shown here, the Pt-Co alloy nanoparticles of 100-200 nm in size attached on the carbon membrane of a copper grid were used as the specimen, and the reconstructed amplitude image shows a much better resolution than that of the STXM image (Figure 1), while the radiation dose is only 1/12 of STXM, and the data collection time is only about 1/3 of STXM (Table1). These results indicate a great application potential of our PCDI platform especially in life science.



(a) (b) (c) (d)

Figure 1: A PCDI result from our CDI platform. The specimen is Pt-Co nanoparticles attached on a carbon film. A 3 um illumination spot with 710 eV energy was scanned across the sample with a step size of 500 nm in a raster grid of 6x10. The sample-CCD distance is about 70mm. (a) is the averaged diffraction pattern, (b) is the reconstructed amplitude image of the sample, (c) is the STXM image with 30 nm scan step, (d) is the PSD analysis of the reconstructed image (b) which indicate a resolution of 12.4nm, much better than the STXM ultimate resolution of 30 nm.

Performance	STXM	Ptychography	STXM : Ptycho
Exposure time (dose)	226 s	18.8 s	12:1
Data acquisition time	30 min	11 min	~ 3:1
Spatial resolution	> 30 nm	~ 12.4nm	~ 2.5 :1

Table 1: The performance comparison between STXM and ptychography in our station.

LARGE-SCALE NANOSTRUCTURE INVESTIGATIONS: SIX-DIMENSIONAL SAXS-CT

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Abstract
Nowadays, X-ray microscopy is able to image with impressive spatial resolution, down to a few nanometers [1]. This, however, comes at a price: the investigated samples typically have to be very small. Mapping nano-structural changes over a large distance remains a huge challenge.

We present six-dimensional small-angle X-ray scattering computed tomography (SAXS-CT) as a method able to investigate the nanostructure in objects several orders of magnitude larger. The combination of SAXS with tomography allows us to reconstruct the scattering distribution in each voxel of the investigated object [2].

X-ray experiments were carried out at the cSAXS beamline, Paul Scherrer Institut, Switzerland. A piece of human tooth served as a first sample. Dentin in teeth is bone-like, composed of mineralized collagen fibers with variable, albeit well-defined orientations, which give rise to strong SAXS signals. Until recently, it has not been possible to investigate structural changes in this nanocomposite in three dimensions.
We present results of this first full SAXS-CT reconstruction and the voxel-wise extracted collagen fiber orientations for a 4mm sized human tooth sample.

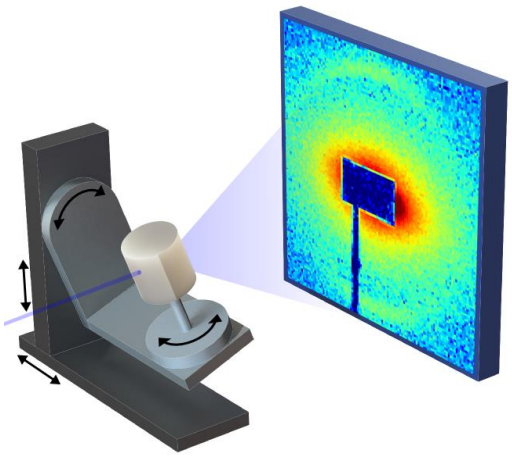

Figure 1: Schematic representation of the Six-dimensional SAXS-CT experiment.

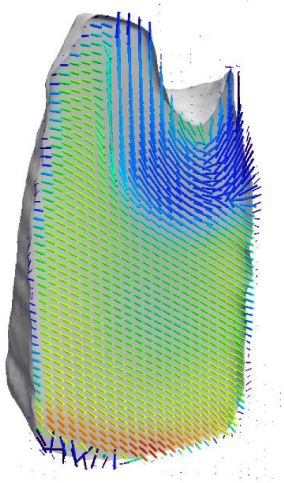

Figure 2: Visualization of the reconstructed collagen fiber orientation inside the tooth.

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SINGLE SHOT X-RAY COHERENT IMAGING FOR GENERAL SAMPLES

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Abstract
With the promise of diffraction limited resolution and the ability to provide high sensitivity phase images, coherent diffractive imaging (CDI) has received enormous interest in the past years in the x-ray imaging community. The challenges of CDI include the reconstruction convergence, especially when regions of the data are missing, and the limited ability for extended or complex-valued samples. These challenges can be overcome using ptychography [1] that exploits multiple measurements with probe overlaps. We have developed an alterative **single-shot** coherent modulation imaging approach [2] which overcome the limitations of conventional CDI.

Figure 1 illustrates the setup of the method. Besides eliminating the ambiguities in phase retrieval, the downstream modulator also allows for matching the diffraction intensity distribution to the characteristic of the detector. Experiments have been conducted at the cSAXS beamline, Paul Scherrer Institut, Switzerland at the energy of 6200eV. Extended samples with different diffracting power are imaged by the method. A 2 μm pinhole, 2 mm upstream of the sample was used to form the illumination. The 2mm Fresnel propagation resulted in a tapered probe amplitude which would present a difficulty for conventional CDI. As the support constraint, a round disk, about 70% larger than the vague extent of the exit wave, was used throughout our reconstruction. Images of high quality with a resolution of 35 nm were obtained within 100 iterations while recognizable structure appeared after only 30 iterations from random amplitude and phase initial guess of the object.

We will present details of the latest x-ray experiment of the modulation coherent imaging method [3]. The single-shot capability of the method makes it suitable for the study of ultrafast dynamics of samples. The potential of implementing the technique at x-ray free electron laser (XFEL) will also be covered.

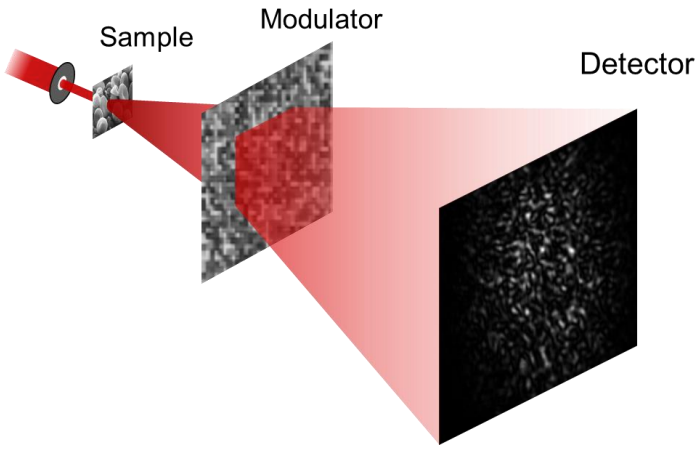

Figure 1: Schematic of the setup of coherent modulation imaging technique

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PTYCHOTOMOGRAPHY AT DLS COHERENCE BEAMLINE I13

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Abstract

Ptychography is a scanning transmission diffraction microscopy (STDM) technique in which a sample is scanned perpendicular to the path of X-ray beam, collecting diffraction patterns at each point. The points are chosen such that there is enough overlap of probe between adjacent points. The patterns along with the information of the points at which they were collected are then processed through iterative phase retrieval algorithms giving high resolution, high contrast transmission maps of the sample [1,2]. A natural extension of two dimensional ptychographic imaging is the three dimensional analog, ptychotomography. We exploit the high resolution 2D ptychographic projections obtained at various angles as a starting point which are then taken through the conventional tomographic process of alignment and 3D reconstruction thus obtaining a three-dimensional transmission map of the sample under study [3].

We will report about the implementation and execution of ptychotomography at I13-1, the coherence branchline at Diamond Light Source [4]. We were successful in collecting the three dimensional ptychotomographic data from the nanoporous gold sample, which is presently in analysis stage. We are currently in the process of achieving faster scan rates and making further improvements to the existing 3D data acquisition and reconstruction routines. We are headed towards execution and optimization of multi-wavelength imaging and on-the-fly scanning ptychotomography.

We acknowledge financial support through the European Research Council (ERC, starting grant “OptImaX”).

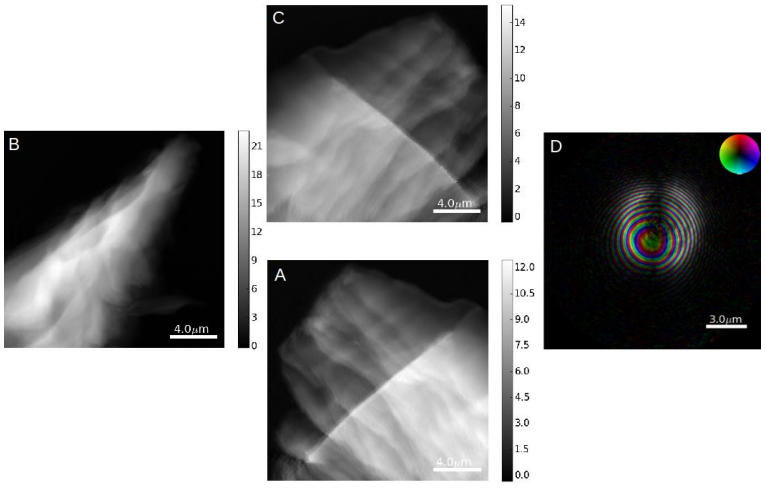

Figure 1: A,B,C. shows the phase unwrapped projections of nanoporous gold sample at -90,0 and 90 degrees respectively. The gray scale shows the phase shift in radians. D is the first mode of orthogonalized probe obtained from reconstructions.

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APPLICATIONS OF NEAR-FIELD PTYCHOGRAPHY WITH HARD AND SOFT X-RAYS

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Abstract
Near-field ptychography is a novel coherent diffraction imaging (CDI) modality where Fresnel diffraction patterns exhibiting an overlap of the incident illumination between adjacent scan points are recorded. Data redundancy provided through such a collection scheme is exploited post-acquisition to iteratively reconstruct both the object's transmission function and the scanning illumination profile [1, 2, 3]. Merging the high throughput and simplicity of inline holographic methods with the robustness of ptychographic approaches, the technique allows for images and tomograms of high resolution and quantitative nature. In addition, near-field ptychography is less prone to artifacts when applied to strongly absorbing and phase shifting samples [4, 5] compared to other full-field methods. These features make the technique applicable to a wide range of specimens. We demonstrate the power of near-field ptychography by showing tomographic reconstructions of paleontological samples which will help answer questions of vertebrate evolutionary history. Furthermore, we show results of the technique being used for the first time with soft X-rays, thus opening an additional path for imaging in this regime and as a consequence to a new field of specimens. Our experiments were carried out at the beamlines I13-1 and I08 at the Diamond Light Source.

We acknowledge financial support through the European Research Council (ERC, starting grant "OptImaX").

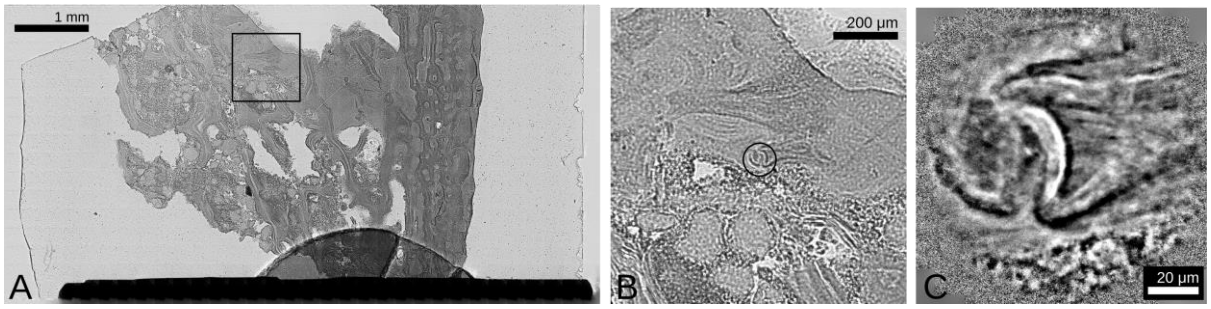

Figure 1: (A) Hologram of fossil bone slab. (B) Zoomed in hologram of region marked in image A. (C) Near-field ptychographic phase contrast image of region marked in image B.

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PROPAGATION-BASED PHASE RETRIEVAL: APPLICATIONS AT A LAB-BASED MICROCT SYSTEM

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Abstract

Single-distance phase-contrast imaging (PCI) is one of the rapidly evolving phase-contrast imaging techniques available nowadays [1, 2]. Because these imaging methods require (partially) coherent X-ray beams, they commonly have been used at synchrotrons. However, technical developments have enabled the translation of some of the techniques to laboratories, also using polychromatic X-ray tubes [3, 4]. The improvement of phase-retrieval algorithms, extended to cone-beam geometry [5] allows for the retrieval of amplitude and phase images, which usually hold complementary information. Propagation-based imaging is known to improve the visualization of weakly absorbing features in a sample. The application of this technique in radiography and tomography at a commercial device might be an asset compared to synchrotrons, as it will enable investigations of objects in phase-contrast modality at more easily accessible commercial setups.

The most common phase retrieval algorithms are based on pre-processing the acquired mixed projection images and then applying standard CT reconstruction. An alternative is to apply the phase correction in a post-processing step on already reconstructed data [6]. The latter approach seems preferred in cases, where access to the raw projections recorded by a proprietary system software are not directly accessible, as is the case in some commercial devices.

The general feasibility of phase-contrast imaging combined with a phase-retrieval step at the commercial device ZEISS Xradia VersaXRM-500 by Carl Zeiss (former Xradia, Inc.) has already been demonstrated in [7]. Here we now present additional application examples of this procedure to biological and material science samples, which demonstrate the broad applicability and value of this method in combination with a commercial system.

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HOLOGRAPHY-GUIDED PTYCHOGRAPHY WITH SOFT X-RAYS

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Abstract

In our work, we present a novel combination of two coherent imaging methods, namely X-ray holography [1] and ptychography [2] for imaging nanoscale objects using soft X-rays. Ptychography as a scanning imaging method relies on the exact knowledge (or post-experimental retrieval) of the position of the X-ray illumination on the sample during scanning [3]. Our method combination allows to directly encode the scan positions in the diffraction pattern without the need of accurate position encoders (Fig. 1) [4]. We demonstrate that holographically encoded positions significantly reduce the experimental and numerical requirements. Image artifacts originating from ambiguity in the dataset (e. g. missing low frequencies in the diffraction pattern) can be identified in comparison to the FTH reconstructions. We show, that these image artifacts can be reduced, using the FTH reconstruction as starting guess for the ptychographic solution (Fig. 2).

Our ptychographic reconstructions cover a large field of view with diffraction-limited resolution and high sensitivity in the reconstructed phase shift and absorption of the objects. Both phase shift and absorption are used to determine—the otherwise unknown—elemental composition and thickness of the Siemens star test structure.

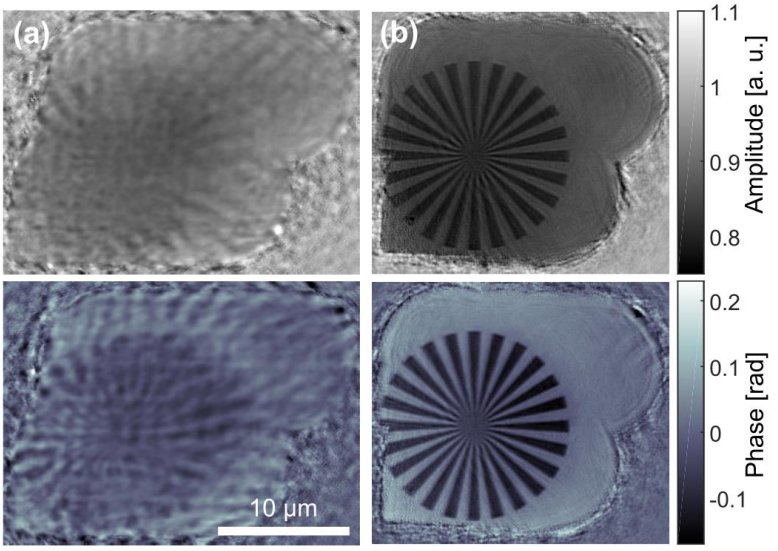

Figure 1: Comparison between ePIE reconstructions using (a) positioner encoded positions and numerical position correction (pcPIE) (b) FTH positions and additional numerical position correction (pcPIE). The first row of images shows amplitude, the second row shows phase contrast.

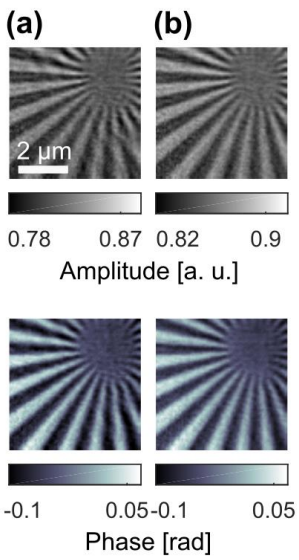

Figure 2: Magnification of the central part of the reconstruction of the Siemens star. Image artifacts in the ptychographic reconstruction as present in (a) are reduced in (b) when using a starting guess based on the direct holographic image.

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THE ENHANCEMENT OF PTYCHOGRAPHIC IMAGES THROUGH THE CORRECTION OF DETECTOR IMPERFECTIONS

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Abstract

Ptychography, a form of coherent diffractive imaging (CDI), produces high resolution (aberration-free), phase sensitive (complex-valued) images and is routinely implemented at synchrotrons around the world. The progress in electron ptychography has, despite some recent advances, generally lagged behind that in the x-ray and visible light regimes. There are several contributing factors to this, from instrument stability and beam coherence to the quality of the electron detectors.

In ptychography the function of an imaging lens is replaced directly with a detector and a computer algorithm; the recorded intensities are computationally phased before being propagated to an image plane. Although the transfer function introduced by the imaging lens is now removed, the quality of the detector becomes more critical. Any imperfections or non-linearity within the detection process acts to degrade the recorded coherence between data points and ultimately reduces the resolution and contrast in the final reconstruction.

The implementation of an improved reconstruction algorithm, which exploits the rich diversity within the ptychographic dataset to directly correct for the non-ideal response of detector pixels, has recently been used to improve the quality of previously published [1] electron data. The enhanced reconstructions have successfully demonstrated 0.14nm resolution with 30keV electrons (see figure 1). The issue of detector imperfections also extends to x-ray ptychography (and ptycho-tomography), where the effects of cosmic rays, dropped frames, and dead zones in multi-chip arrays must be accounted for pre-reconstruction.

The recent results produced from a 30keV scanning electron microscope (SEM) will be presented along with latest results from the I13 coherence branch at the Diamond Light Source.

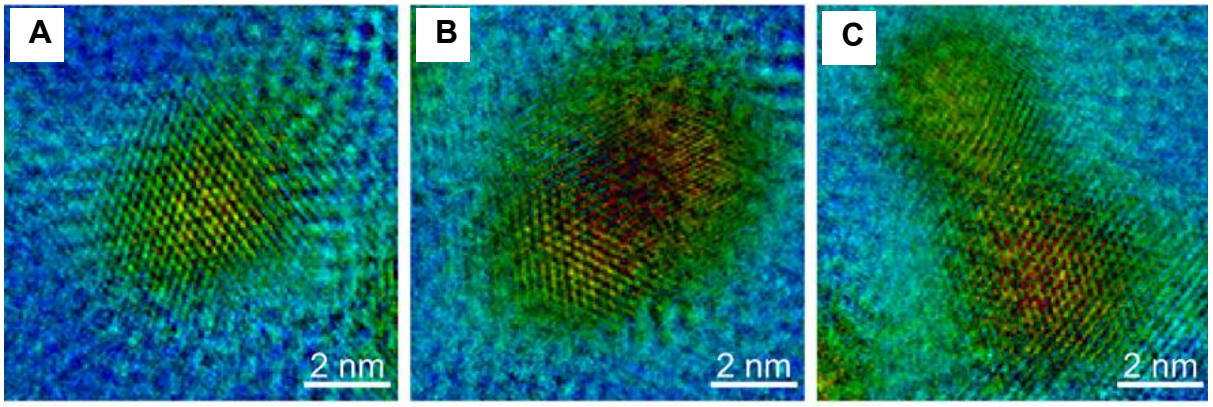

Figure 1: Ptychographic images of Au nanoparticles on a carbon support film as taken from a larger 100nm field of view.

Reference

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