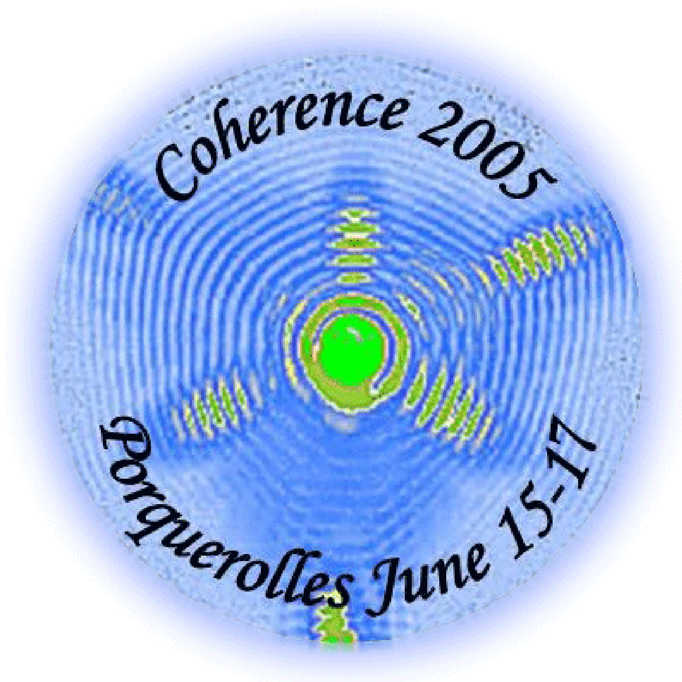
International Workshop on
Phase Retrieval and Coherent Scattering



Programme
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List of Participants

Joint DESY-ESRF-SLS Workshop

International Workshop on

Phase Retrieval and Coherent Scattering

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International Workshop on

Phase Retrieval and Coherent Scattering

Invited Speakers:	Institute, University:
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Gerhard Grübel	DESY Hamburg, Germany
Christian Gutt	University of Dortmund, Germany
Janos Kirz	LBNL Berkeley California, USA
Karl Ludwig	University of Boston, USA
Rick Millane	University of Canterbury, New Zealand
Keith Nugent	University of Melbourne, Australia
Ian Robinson	University of Illinois, USA
Frank Scheffold	University of Fribourg, Switzerland
Sunil Sinha	University of California San Diego, USA
Lorenz Stadler	University of Vienna, Austria
Dirk Van Dyck	University of Antwerp, Belgium
Timm Weitkamp	PSI Villigen, Switzerland

International Workshop on

Phase Retrieval and Coherent Scattering

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International Workshop on

Phase Retrieval and Coherent Scattering

Speakers' Abstracts

Phase Retrieval Methods

Phase Retrieval Methods: An Overview

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Phase-sensitive imaging has developed as a very important method in x-ray science. The methods that have been developed may be classified in a number of ways, depending on the form of the imaging required; the methods by which phase contrast is generated; or the experimental parameters.

Nugent et al [1] defined "phase-contrast" imaging as methods that render phase variations visible (an example of this is differential interference contrast imaging [2]); "phase imaging" as methods that produce images that are linear in phase (Zernike phase imaging [3]); and "quantitative phase imaging" as methods that yield a spatially resolved *measurement* of the phase (interferometry [4]; holotomography [5]).

Alternatively can also ask whether the phase sensitivity is acquired through propagation (holotomography; transport of intensity) or through interference (interferometry; Zernike phase imaging).

A third classification is according to the diffraction regime in which the measurement is made. The most common classifications are the "edge-detection regime" (transport of intensity methods [1]); the holographic regime (holotomography) and the far-field (which is the domain of coherent diffractive imaging [6]).

An additional form of phase-sensitive imaging that may have some interesting applications is the measurement of the coherence properties of the fields using phase-space tomography [7].

In this talk I will present an overview of the state of the field of x-ray phase imaging. I discuss and present examples of each of the above methods and explore some of their limitations. I will then develop a unified description in terms of partial coherence within the Fresnel diffraction approximation and discuss how these methods relate to the emerging area of coherent diffractive imaging.

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Phase Retrieval and Support Estimation in X-Ray Diffraction

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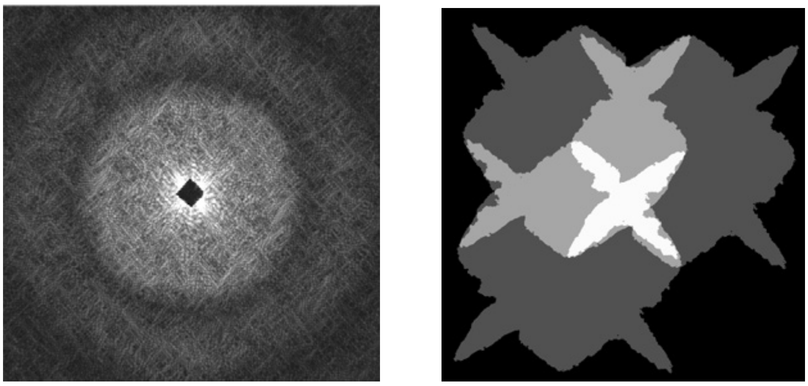
Phase retrieval algorithms originally developed for astronomy [1] and wavefront sensing [2,3] can also be used to reconstruct an image from an X-ray diffraction pattern of a nonperiodic object. Iterative transform algorithms, including the "hybrid input-output algorithm," transform back and forth between object space and Fourier space, enforcing the constraints and measured data in each domain. Gradient search algorithms typically optimize over an objective function that quantifies the difference between the collected Fourier data and the computed Fourier transform of a model estimate of the object. An analytic expression for the gradient enables efficient computations within an algorithm such as conjugate gradient search.

Important to the convergence of phase retrieval algorithms is determining a constraint for the support of the object, i.e., the set of points outside of which the object is known to be zero (or some constant). We have developed algorithms for placing upper bounds ("locator sets") on the support of the object from the support of its autocorrelation function [4-6], which can be computed by Fourier transforming the measured x-ray intensity data. Figure 1 shows an example on data provided by Henry Chapman at LLNL and Malcolm Howells at LBNL, from a collection of microscopic gold balls. The existence of the beam stop, preventing the measurement of the lower spatial frequencies, presents an additional challenge.



(a) (b)

Figure 1: (a) X-ray diffraction data, (b) upper bound on object support, shown in white: the intersection of three translated versions of the support of the autocorrelation function computed from (a).

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Physical Resolution Limits of Single Particle 3D Imaging with X-rays and Electrons

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Recent years have witnessed a boost of methods aiming at 3D imaging of single particles with nm resolution in life-science applications or sub Angstrom resolution in inorganic materials. New X-ray imaging methods such as lensless diffractive imaging combined with powerful phase reconstruction methods such as hybrid I-O or oversampling have proven their potentiality. Simultaneously we also witness a steady improvement in the brightness of the synchrotron sources or the development of Free Electron X-ray laser sources which allow to use extremely fast pulses and methods to combine the data of many identical particles or to orient them physically prior to the diffraction experiment. Combining these methods will continue to push the resolution even further.

Ultimately this evolution will be hampered by physical limits that cannot be surpassed such as radiation damage of the specimen and limits on the brightness and pulse duration etc.

For electron imaging the development of aberration correctors pushes high resolution electron microscopy into the domain where individual atoms can be resolved. Furthermore the brightness of the advanced electron sources exceeds that of the synchrotron. One reaches the situation where the information in real space i.e. in the image approaches the information in Fourier space i.e. the diffraction pattern. Then HREM or lensless diffractive methods will yield the same information.

Since the interaction between electrons and atoms is orders of magnitude larger as compared to X-rays but with less radiation damage, electron imaging methods have the potentiality for higher resolution. In HREM of inorganic materials the limit is posed by the total dose -Rose Criterium - which is limited by displacive radiation damage or by the combination of brightness and recording time. The figure of merit is the ratio between the elastic and inelastic cross section for the electron interaction.

The ultimate goal of all imaging methods is to visualise the ultimate building blocks. In case of electron imaging this are the constituting atoms. In case of X-ray imaging of life particles this may be larger but well known subunits. Once the building blocks can be resolved it is in principle possible to refine their positions quantitatively in the sense as done by classical electron diffraction techniques. Precise positions of the building blocks can then be used as input data for ab-initio calculations in order to understand the properties and eventually design new structures.

For HREM the ultimate challenge is to quantitatively determine atom positions in an amorphous structure with high precision. However, the information density, the number of data per unit area exceeds the physical capacity of the electron microscopic information channel. The only way out is to explore also the third dimension, i.e. electron tomography with atomic resolution. For amorphous materials which are subject to radiation damage it may require a lower voltage and the use of a Cs and Cc corrector. Since HREM is a parallel imaging technique it is suited for tomographic reconstruction. However we have doubts about the practical usability of optical sectioning by HAADF STEM.

X-Ray Phase Imaging with Grating Interferometers

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Hard X-ray grating interferometry is a relatively new method for wavefront sensing and phase radiography [1-4] in the energy range between between 8 and 30 keV. Different measurement modes such as phase stepping (Fig. 1) or moiré interferometry can be used to obtain quantitative phase maps of X-ray wavefronts and/or objects in the beam, and the combination with tomography allows three-dimensional reconstruction of the X-ray refractive index of samples.

While the spatial resolution of the technique can be as good as a few micrometers, the true promise of grating interferometry is to provide better images and new information where other phase-imaging methods cannot easily be used. This is at large fields of view, and with full-field beams of wider cross section than is usually available at synchrotron sources. X-ray tube generators such as those used in medical diagnostic imaging provide such wide beams, and many problems that impede the use of other phase-radiography methods with radiation from tube sources do not occur in a grating interferometer.

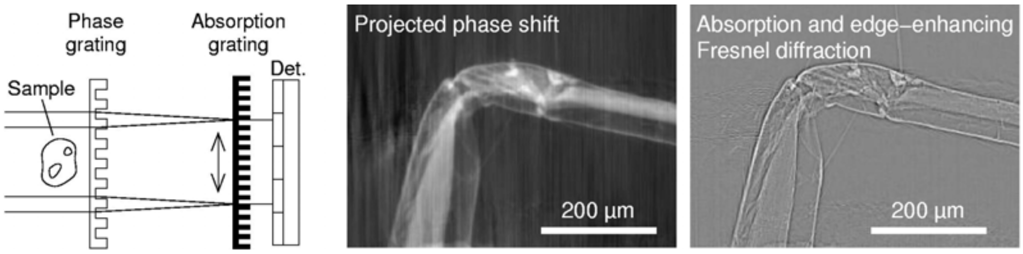

Figure1: Grating radiography. ***Left:*** Schematic setup. ***Center:*** detail of a reconstructed phase projection of a spider's leg, obtained from a phase-stepping scan (14.4 keV). ***Right:*** non-interferometric image containing absorption and edge-enhancing Fresnel diffraction contrast, extracted from the same data set.

However, in order to estimate whether the use of grating-interferometric radiography at laboratory sources is realistic, many questions remain to be answered. These include the following: How chromatic is a grating interferometer? What limits the photon-energy range accessible to grating interferometry? Can the device be used with illumination by a strongly curved wavefront? Can a tube source provide the coherent flux needed? What are the requirements on detector resolution?

The presentation will address these questions after an introduction into image formation and measurement modes.

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Phase Problem and the Radon Transform

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Under the phase problem we understand the problem of reconstructing the complex amplitude of the coherent field from intensity measurements. Two different solutions to this problem are considered. Both solutions are linear and make the use of the Radon transform. The mathematics of the Radon transform is applied to formulate the solutions in terms of convolution integrals.

In the first type of solution, a 3D phase problem for a completely polarized coherent monochromatic field is linearized by using the Wigner transform in the parabolic approximation. It is shown that the Wigner distribution function (WDF) can be obtained from intensity measurements by applying the inverse Radon transform. After the WDF is obtained, the amplitude of the coherent field can be determined up to unknown multiplicative constant. The method presents a nice theoretical approach that gives us a possibility of writing down linear equations relating intensity measurements to the scalar field amplitude. However, in spite of its theoretical elegance, a practical implementation of the method is quite cumbersome because the intensity measurements are required in many planes along the beam. The number of planes needed is governed by the Nyquist criteria for the discrete Radon transform and can be relatively large for a sophisticated WDF [1].

A more practical solution requiring less measurements can be found for applications of phase-contrast tomographic imaging. The linearization of the phase problem is achieved here by using sufficiently small distances in the near field of the Fresnel region. It is also assumed that absorption is weak and change slowly. Under these conditions a fundamental theorem is proved [2]. The theorem plays the same role in phase-contrast tomography as the Fourier slice theorem does in conventional absorption-based tomography, but it is based on the Radon transform rather than the Fourier transform. The theorem gives us the way of reconstructing a 3D object function by covering its Radon space and applying the 3D inverse Radon transform afterwards. Because computation of the complete Radon space is a demanding task, another way of using the results of the theorem is considered. In a similar way as the filtered backprojection (FBP) algorithm is derived from the Fourier slice theorem, we derive a practical FBP algorithm using the fundamental Radon theorem for the phase-contrast data. The algorithm requires intensity measurements in a single plane of the near field in the case of the purely phase object. Reconstruction of the mixed amplitude and phase object will require two planes. The theorem and the method not only provide a nice theoretical insight on the phase problem of phase-contrast imaging, but also give us the means for fast practical inversion of the huge volumes of high-resolution phase-contrast data. The application of the approach is now investigated at several synchrotron sources.

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Universal Iterative Phasing Method for Near-Field and Far-Field Coherent X-Ray Diffraction at the APS

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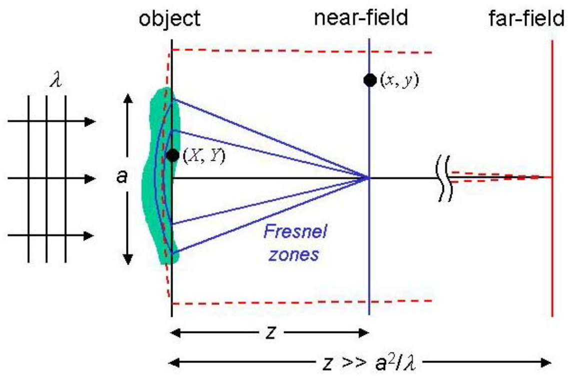
Coherent x-ray diffraction is usually categorized into two distinct regimes: the near-field Fresnel or in-line holography regime, and the far-field Fraunhofer regime. In the near-field regime, coherent imaging allows the detection weakly absorbing objects due to phase-contrast or phase-enhanced effects. Various methods of phase retrieval in the Fresnel regime include transport of intensity equation method and the holo-tomography algorithm using the self-imaging principle for different spatial frequencies at several detector distances. For far-field diffraction, because of the direct Fourier transform relationship between real space density and reciprocal space amplitudes, an iterative phasing method has been widely applied and proved to be very effective.

In this paper, we present recent activities to enhance the imaging capabilities at the APS, and a universal iterative method for evaluation of wave-field propagation and for phase retrieval of a continuous diffraction pattern in both far-field and near-field regimes. Our method (see figure at right) embeds Fresnel-zone construction on an original object to form a phase-chirped distorted object, which is then Fourier transformed to form a diffraction pattern. We show several examples of phase reconstruction to illustrate this approach, which extends the applicability of Fourier-based iterative phasing algorithms into the near-field regime. Based on our results, we discuss the potential advantages of near-field diffraction with few Fresnel zones in high-resolution structural investigations of noncrystalline materials.



This work is supported by US National Science Foundation and by US National Institute of General Medical Sciences through CHESS under Award DMR-0225180, by National Institute of Biological Imaging and Bio-engineering through Hauptman-Woodward Institute under Grant EB002057, and by U. S. Department of Energy, Office of Basic Energy Sciences, through APS under Contract No. W-31-109-Eng-38.

Effects of Partially Coherent Incident Illumination on In-Line Imaging

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We derive an analytical expression for the intensity distribution in in-line images collected in the "near-field" regime, which explicitly takes into account the effect of key parameters of the incident radiation, such as the spatial coherence properties of the source, its wavelength spectrum and the principal geometry of the imaging layout. The expression is valid in the cases of polychromatic quasi-plane and quasi-spherical incident waves, as well as for spatially incoherent, quasi-homogeneous and some other types of sources. We also discuss practical methods for measuring the relevant parameters of the incident radiation. More specifically, our formalism requires measurements of certain three-dimensional functions (such as the spatial distribution of incident intensity and phase at individual wavelenghts) as opposed to five-dimensional functions, such as e.g. mutual coherence, cross-spectral density or generalised radiance required in some previously reported alternative approaches [1-4]. Moreover, the experimental techniques that can be used to measure the relevant properties of the incident radiation ("source characterization") in our approach are exactly the same as the ones employed at the subsequent stages of the experiment for measuring the wave transmitted through objects of interest.

The results are expected to be useful in quantitative in-line imaging, phase retrieval and tomography with polychromatic and spatially partially coherent radiation. An application of the obtained formalism is presented in which analytical expressions are derived for the optimal defocus distance [5] and the critical source size [6] in in-line imaging of objects consisting predominantly of a single material using extended polychromatic X-ray sources. In particular, we show that in order for the in-line phase-contrast effects to be observable, the standard deviation of the intensity distribution of an incoherent source should be less than the following value:

$$\sigma_{\max} = \frac{M}{M-1}\left[R' \frac{\int S(\nu)\delta(\nu)\mathrm{d}\nu}{\int kS(\nu)\beta(\nu)\mathrm{d}\nu} \right]^{1/2},$$

where M is the geometric magnification (the ratio of the source-to-detector and the source -to-object distances), R' is the object-to-detector distance divided by the magnification, $n = 1 - \delta + i\beta$ is the complex refractive index of the object, k is the wavenumber and $S(\nu)$ is the frequency spectrum of the incident radiation.

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Phase Retrieval in Tomography with Kirkpatrick-Baez Mirrors

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Two x-ray mirrors reflecting in planes perpendicular to each other and bent to the desired elliptical shape produce a two-dimensional focus when placed in the parallel beam of a synchrotron source. A graded multilayer on the first mirror acts as broadband monochromator ($\Delta E/E = 10^{-2}$) and provides a very high flux (up to 10^{12} ph/s). We obtain a focal spot of 100 nm in vertical and horizontal direction, thus opening possibilities for various imaging techniques at a new level of spatial and temporal resolution.

Magnified radiographs can be obtained by putting the sample a small distance downstream (or upstream) of the focus and the detector at a large multiple of the sample to focus distance. The defocusing distance and magnification can be changed by translating the sample in the direction of the x-ray beam while keeping the detector fixed. The recorded images correspond to the Fresnel diffraction regime and a phase retrieval step has to be associated to the tomography in order to obtain interpretable information and optimum resolution.

One limitation of tomography with the KB system is due to mirror shape imperfections, leading to a sample illumination which is not a simple spherical wave. We propose an image correction method based on the knowledge of the distortion introduced by the two mirrors. The latter information is obtained by employing a grid as a wavefront sensor. Before phase retrieval itself can be performed another crucial step is to bring the radiographs at all planes to the magnification of the plane closest to the focus and to precisely align them relative to each other. Finally, different phase retrieval methods are compared on experimental data. Astonishingly, the best results are obtained when the samples are much larger than the field of view (local tomography (see Fig.1)).

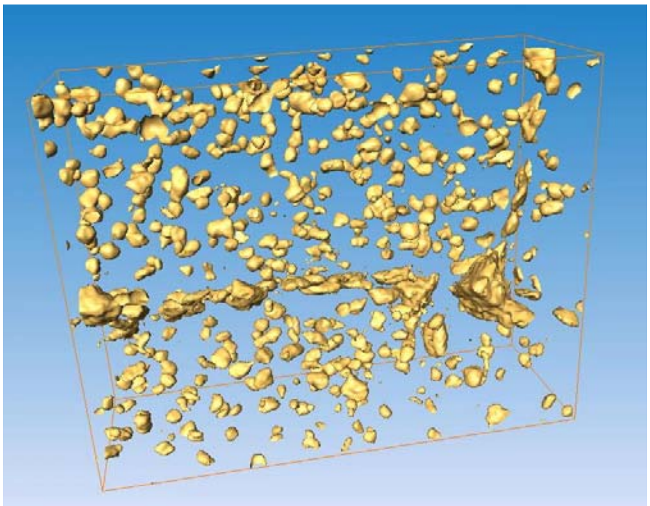

Figure 1: Resulting 3D image of an Al-Cu alloy near a grain boundary

Phase Retrieval with the Ptychographical Iterative Engine: Analysis of Success for STEM given Incorrect Initial Parameters

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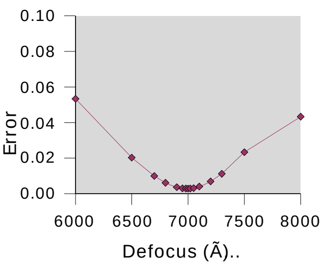
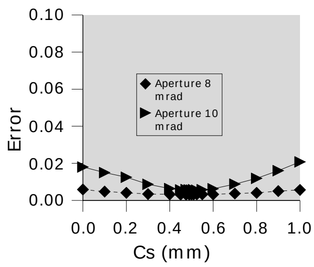
The Ptychographical Iterative Engine (PIE) algorithm [1] is a phase retrieval algorithm that uses measured wavefunctions, created with a probe which is assumed to interact multiplicatively with the specimen. It solves a problem previously thought to be intractable: that of iterative phase retrieval in STEM [3], and is applicable to many other situations. The algorithm combines useful properties of iterative methods, such as numerical stability and insensitivity to noise, with the ability to image at high resolution through the use of diffraction data.

The PIE algorithm may be used in many forms of microscopy, and is currently specifically being applied to STEM. To be effective in experimental practise, it must be tolerant of problems that arise in experiments, such as inaccurate characterisation of the imaging system, or incoherence effects. This paper studies the behaviour of the algorithm in such situations.

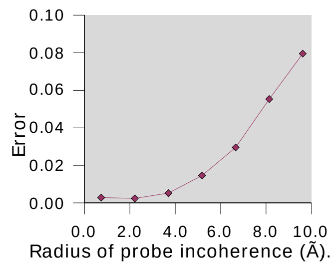
Figures 1(a) and (b) demonstrate the effect on PIE phase retrieval in STEM when a probe parameter is inaccurately known. In general, the PIE algorithm is tolerant of incorrect characterisations of the probe parameters, suggesting that this will not cause problems in experimental practise. Very poor characterisation of these parameters has a detrimental but well behaved effect on the algorithm. Therefore, iterating the algorithm over small variations in the assumed parameters may be used to test and improve the accuracy of the probe characterisation, increasing the success of that and future experiments using the same probe.



(a) Error with varying defocus Correct defocus was 7000Å



(b) Error with varying C_s Correct C_s was 0.5mm



(c) Error with increasing probe incoherence

Figure 1: Error results of iterative algorithm (after 400 iterations) when varying known probe parameters away from the correct value, and for varying probe incoherence.

Figure 1(c) shows the behaviour of the algorithm as the probe incoherence is increased. The phase retrieval accuracy decreases with increasing incoherence. However the algorithm is tolerant of much greater incoherence than would be present in a normal experiment. These results suggest that the PIE algorithm is a very good candidate for experimental success.

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X-Ray Phase-Attenuation Duality and Phase Retrieval For Soft Tissue Imaging

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Phase-retrieval is the key to the quantitative x-ray phase-contrast imaging of soft tissues. In order to retrieve a phase image of tissues, multiple phase-contrast images are needed in general. While the current TIE-based retrieval approach is largely successful, the approach requires low-level noise for retrieval-accuracy. In clinical imaging it would be desirable to find a way to retrieve the tissue phase-map from a single phase-contrast image for reducing the radiation dose and the artifacts associated with motion. Moreover a robust phase-retrieval approach is desirable to limit tissue-radiation doses to a reasonable level.

Quite recently we made a new observation of the phase-attenuation duality for soft tissues, and we showed how only a single phase-contrast image is needed for a successful phase-retrieval for inhomogeneous soft-tissues based on this duality [1]. In this talk the phase-retrieval formula in [1] will be discussed in more details. In addition we show how to extend the phase-retrieval formula into cases of polychromatic and partially coherent x-ray encountered in clinical imaging. The high robustness of these retrieval algorithms will be rigorously established. Taking the mammography as an example, we show that this new phase-retrieval approach based on the phase-attenuation duality may result in striking enhancement of mammography contrast-noise ratio per unit average glandular dose to beast.

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X-ray Photon Correlation Spectroscopy

X-Ray Photon Correlation Spectroscopy

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One of the outstanding properties of third generation synchrotron radiation sources is their capability of producing coherent x-ray beams several orders of magnitude more intense than previously available. The access to coherent x-rays opens up a variety of possibilities for new techniques such as x-ray photon correlation spectroscopy (XPCS), static x-ray speckle analysis and metrology and has major impact on imaging techniques such as topography, phase-contrast and holographic imaging. Much of the excitement about scattering with coherent x-rays arises from the possibility to perform atomic resolution correlation spectroscopy and we will focus on the progress towards studying the complex dynamics of disordered systems on length- and time scales inaccessible to other techniques.

X-ray photon correlation spectroscopy (XPCS) probes the dynamic properties of matter by analyzing the temporal correlations among photons scattered by the studied material. It can measure the low frequency dynamics (10^{7} Hz to 10^{-3} Hz) in a Q range from typically $1 \cdot 10^{-3}$ $Å^{-1}$ up to several $Å^{-1}$. X-ray photon correlation spectroscopy is in particular complementary to Dynamic Light Scattering (DLS) or Photon Correlation Spectroscopy (PCS) with visible coherent light which probes also slow dynamics ($\omega<10^{6}$ Hz) but can cover only the long wavelength $Q<4 \cdot 10^{-3}$ $Å^{-1}$ regime. XPCS is furthermore not subject to multiple scattering, a phenomenon frequently complicating the analysis of PCS data in optically opaque systems. Neutron based techniques (inelastic and quasi-elastic neutron scattering, neutron spin-echo) on the other hand can access the same Q range but probe the dynamic properties of matter at high frequencies from typically 10^{14} Hz down to about 10^{8} Hz.

X-Ray Intensity Fluctuation Spectroscopy Studies of Ordering Kinetics in a Cu-Pd Alloy

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While a number of studies have used x-ray intensity fluctuation spectroscopy (XIFS) to examine fluctuation dynamics, fewer have attempted to use it to probe the kinetics of phase transitions. This is also an area in which there have been few, if any, analogous dynamic light scattering studies. Here we report a XIFS study of the coarsening kinetics in the classic long-period superlattice (LPS) Cu-Pd alloy using the ID10A Troika beamline at the ESRF. In particular, our measurements probed the evolution of the two-time correlation function $C(q,t_1,t_2)$ in the alloy on length scales of 10^1-10^3 nm and time scales of 10^2-10^4 sec. The 23 at.% Cu single crystal was annealed at 510 C in the disordered state and rapidly (10 sec) quenched to 435 C, in the region of equilibrium 1-d LPS structure. The evolution of the speckle intensity was examined with a direct illumination Princeton Instruments CCD area detector near the centers of both a superlattice peak (associated with local $L1_2$ order) and a satellite peak (associated with 1-d antiphase correlations). A careful analysis of the superlattice and satellite peak intensities and widths was used to determine the onset of late-stage domain coarsening. During the coarsening regime, the decay of $C(t_1,t_2,q)$ was independent of direction examined and was similar for the superlattice and satellite peaks. In agreement with published Langevin theory and simulations[1], the decay time τ of the two-time correlation function increases linearly with average time $t_m = (t_1 + t_2)/2$ and is relatively independent of wavevector near the peak centers. However, τ increases much more slowly with increasing t_m than is expected. During the coarsening process, the superlattice and satellite peak centers shift, though the speckles themselves remain relatively stationary. Fluerasu *et al.*[2] have observed similar shifts in the superlattice peak of Cu_3Au coarsening during coarsening and suggested that it may be due to inhomogeneous lattice distortion relaxation at domain walls.

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Soft Matter Surfaces Investigated with XPCS

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X-ray photon correlation spectroscopy (XPCS) in grazing incidence geometry allows the investigation of surface dynamics on small length scales. I will give examples ranging from the first proof of principle experiments of bulk liquid surface dynamics to recent experiments studying the freezing in of surface dynamics at the glass transition. Dynamic and static properties of metal-polymer composite systems and thin wetting films will be discussed. Perspectives and limitations of surface XPCS in terms of time and length scales with regards to the current CCD-technique available at ESRF and APS will be addressed.

Probing Particle Dynamics in Dense Colloidal Suspensions with Coherent Radiation

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We discuss the application of light and neutron scattering to dense colloidal suspensions ranging from the nano-scale to the micron-scale and covering such different systems as for example nanoparticle and globular protein solutions as well as ceramic slurries. We will furthermore give an outlook to the application XPCS. We expect that this new technique will provide access to many systems where multiple scattering of light and the limited q-range has prohibited quantitative measurements in the past.

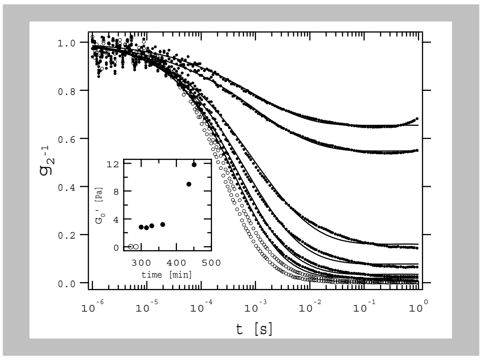

Figure 1: DWS intensity autocorrelation function before (open symbols) and after gelation (solid symbols). Inset: Gel elastic modulus (from [4]).

Particular emphasis will be given to the phenomenon of the so-called dynamical arrest, where dense colloidal or soft matter systems exhibit dramatic changes of their viscoelastic properties and 'solidify'. This means that on the time scale accessible to experiment these systems do not fully relax and in a photon correlation spectroscopy (PCS) experiment they appear nonergodic [1]. In dense complex fluids, such as colloidal suspensions and gels strong multiple scattering of light further complicates the situation. To characterize the sample properties over the full range of interest we employ a rather unique set of static and dynamic light and small angle neutron scattering (SANS) experiments [2]. In moderately turbid systems we use multiple scattering suppression schemes, such as 3D-dynamic light scattering [3] while for even more turbid samples diffusing wave spectroscopy (DWS) is suited to study the internal dynamics on nanometer length scales by measuring the intensity fluctuations of the diffusively transmitted light.

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Probing Slow Dynamics in Complex Systems with 2D-XPCS

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With a well defined coherent beam X-ray Photon Correlation Spectroscopy (XPCS) experiments can be performed to study the dynamics of complex systems. The visible light counterpart to XPCS is Dynamic Light scattering (DLS) which, however, is subject to two main limitations: **i)** the opacity to light of many samples (thus producing multiple scattering and making the experiment impossible) and **ii)** the accessible Q-range which for DLS implies that nanometre length scales cannot be reached ($Q < 4 \cdot 10^{-3} Å^{-1}$).

XPCS overcomes these limitations and we will present two cases where XPCS performed with a CCD detector (2D-XPCS) was successfully used to investigate the dynamics of complex systems inaccessible by DLS. In particular, 2D-XPCS considerably extends the available Q-range and enables the probe of non-ergodic dynamics of strongly opaque samples.

In the first example an opaque dispersion of hard-sphere colloidal particles in pure glycerol was studied [1]. In the experiment the Q-dependence of the diffusion coefficient D(Q) was measured and 2D-XPCS allowed to access the correlation functions up to QR=19 (where R is the particle radius). On these lengthscales the self-diffusion properties of the system are probed.

The second example is related to the study of aging phenomena in glassy soft-matter, which for the moment attracts considerable interest from the scientific community. These disordered systems are in a meta-stable state far from equilibrium and they typically relax very slowly. As a result, the correlation functions have an unusual behavior far from what is known from systems in equilibrium. If the temporal intensity autocorrelation function is measured over a relatively short period of time, it reflects the quasi-equilibrium properties over the measurement period and hence characterizes the system at a certain age. However, the correlation functions can vary substantially if they are measured at a different age, as the material will have relaxed, or aged, into a new state. In this case, time-resolved 2D-XPCS allows to probe the dispersion relations of magnetic nano-particles in a glassy state i.e. the age- and wavevector-dependence of the α-relaxation [2].

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Signal to Noise Ratio of XPCS using High Efficiency Area Detectors

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X-ray Photon Correlation Spectroscopy [1,2] is a novel measurement method which allows us to explore dynamics in new regions of time wave-number space. Although XPCS is a powerful technique, the selection of systems we can study, and the length- and timescales we can achieve are limited by the signal to noise ratio (SNR). In this talk a simple study of SNR is presented, which shows, that apart from the trivial compromises (limiting ourselves to large length scales, slow dynamics or strongly scattering samples) we have plenty of room for improvement in our detectors. Inspired by this calculation we developed a new area detector optimized for XPCS[3], which proved to be useful at many experiments at the 8-ID beam line of APS. The new camera improved our SNR by a factor of 100. The utility of the new detector is demonstrated by presenting the first XPCS measurements on the dynamics of block copolymer lamellae [1].

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X-Ray Intensity Fluctuation Spectroscopy of the Ordering in Cu_3Au

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Cu_3Au has a first-order order-disorder phase transition at T_c=383 C which has been well studied. Recent work[1] using x-ray intensity fluctuation spectroscopy (XIFS) measured the two-time correlation functions during ordering in this system after quenching from above T_c to below. The dynamics of the fluctuations are well characterized by the extension of dynamical scaling arguments for calculating these two-time correlations[2]. One puzzle in these measurements was the presence of an *incubation time* before the onset of this scaling behaviour. This incubation time, which can be as long as 40 minutes, occurs well after the time at which the system has reached the scaling region as determined by measurements of the average domain size (inverse of peak widths).

With upgrades to the IMMY/XOR side station at the Advanced Photon Source, both the intensity and the coherence factor required to perform XIFS measurements has been improved. This has allowed us to obtain better data in the early time region (many seconds and up). During this incubation the time, the central position of the (100) Bragg peak changes a small amount, the asymmetry in the ratio of in-plane and out of plane peak widths decreases slightly and the speckles shift in wavevector. The shift in speckle positions is more than the shift in the Bragg peak position and these shifts become less pronounced the further the sample is quenched below T_c. Using the intensity-intensity correlation function $\langle I(q_1,t_1)I(q_2,t_2)\rangle$ instead of $\langle I(q,t_1)I(q,t_2)\rangle$ allows us to compensate for the speckle shifts and obtain measureable correlation times. The implications on ordering kinetics in Cu_3Au will be discussed.

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Heterodyne Measurement of X-Ray Speckle Fluctuations

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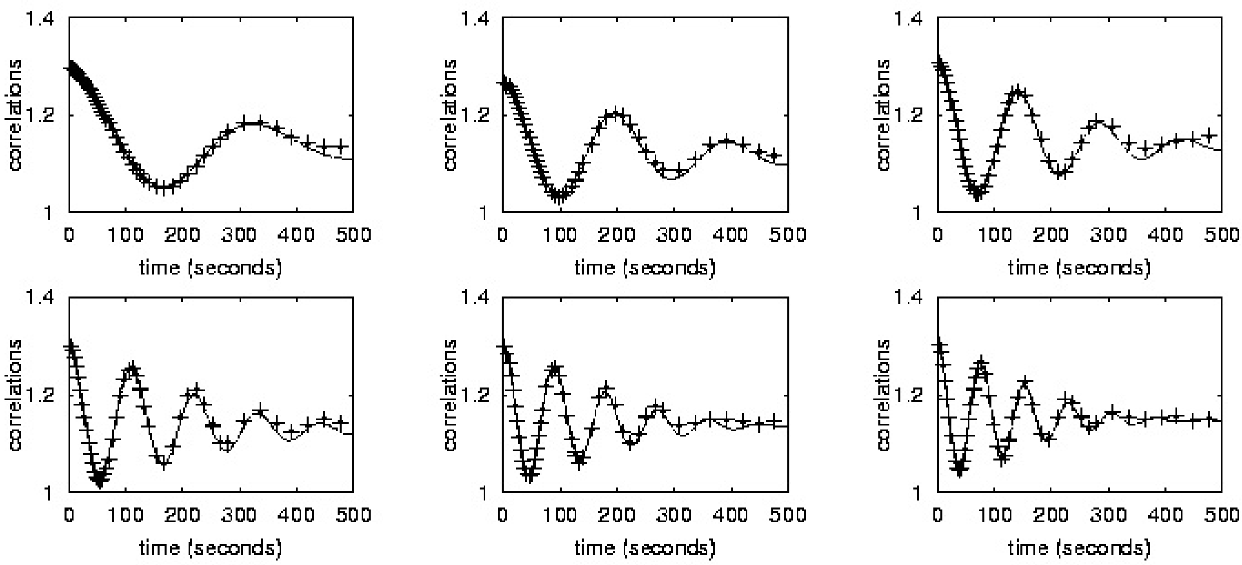
The dynamics of the fluctuations has been studied by coherent small angle x-ray scattering at the APS IMM-CAT beamline. We have used a simple experimental setup in order to obtain homodyne and heterodyne speckle patterns in the same conditions. Measurements were carried out by means of a direct illumination CCD used as area detector. We compare the intensity correlations observed in the case of latex spheres in glycerol. These correlations, averaged over a region of the detector where incoherent intensity is nearly constant, have an exponential behaviour, and we have checked that the heterodyne fluctuation time was twice that of the homodyne one (see [1]).
This method has been extended to the study of the mechanical relaxation of rubber samples made with an elastomer filled with carbon black or fused silica. The relaxation could be observed in the homodyne mode from the movement of the speckles in the detector. In the heterodyne mode, correlations have a strong oscillating behaviour (see Fig. 1) corresponding to the interferences between the moving sample (vector velocity **v**) and the static reference [2]. For a given |**q**|, the period of the oscillations is connected to the angle ϕ between **q** and **v**. This makes possible the observation of relative velocities as slow as 1.5 nm/s.

Figure 1: The oscillating correlations at |**q**|=6.4 10^{-3} Å^{-1}, for various angles ϕ between **q** and **v** observed during relaxation of a carbon black filled elastomer.



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Interpretation of Specular XPCS Measurements of Smectic Liquid Crystal Membranes

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We present a comprehensive account of the dynamics of layer-displacement fluctuations in smectic liquid-crystal membranes as studied by x-ray photon correlation spectroscopy (XPCS) [1-3]. The relaxation behaviour in smectic membranes can be divided into three regimes, characterized by oscillatory relaxation, surface dominated exponential and bulk-elasticity dominated exponential relaxation, respectively. A transition from oscillatory to exponential relaxation is determined by a crossover wave vector q_c with only fluctuations with wave vectors $q > q_c$ showing exponential relaxation.

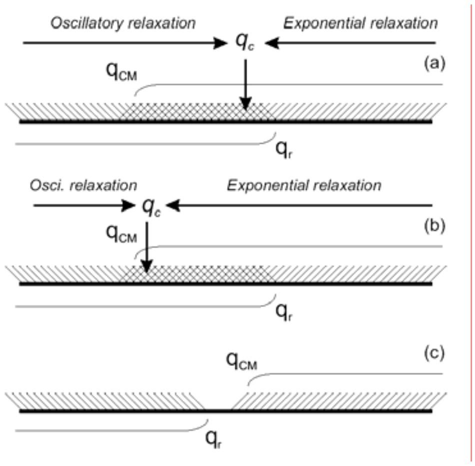

Figure 1: Schematic representation of the window defined for specular XPCS measurements.

XPCS measurement at specular positions are dominated by a window of wave vectors cutting larger and smaller values [3]. This window results from a combination of the finite mosaic of the smectic membranes (as given by the with qr of the rocking curve) selecting long wavelength fluctuations ($q < q_r$), and the size of the coherence volume inside which short wavelength fluctuations ($q > q_{CM}$) perturb the density profile, and is given by the overlap of these two regimes. For thin membranes this window is dominated by fluctuations with $q < q_c$, resulting in oscillatory behavior of the intensity correlation function (Fig. 1a). For thicker membranes the cross-over wave vector q_c shifts towards smaller values and the window of contributing fluctuations is dominated by exponential relaxation (Fig 1b). For extremely well-ordered membranes characterized by a narrow rocking curve < 1 mdeg, the wave vector window is empty, which results in the absence of any contrast in the specular correlation function (Fig. 1c).

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X-Ray Photon Correlation Spectroscopy Study of the Dynamics of a Polymer Bilayer

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We have used x-ray photon correlation spectroscopy (XPCS) to measure the dynamics within a polymer bilayer. The bilayer was comprised of 100 nm polystyrene (PS) film on top of an 80 nm polybromostyrene (PBrS) film, supported on a silicon substrate. XPCS was performed on the surface diffuse scattering from both the PS-vacuum and PBrS/PS interfaces. In order to distinguish the two interfaces measurement were made at grazing incidence in each of two standing wave conditions; below the critical angle for the PS/vacuum interface and at a standing wave condition with a node at the PS/vacuum interface. In the first case the diffuse scattering was dominated by the PS/vacuum interface scattering, and in the second condition the scattering was dominated by the buried interface. Dynamics from the bottom interface show a single slow exponential relaxation mode. Dynamics from the top interface display both slow and a fast relaxation modes. The measured time correlation functions will be compared with the predictions of hydrodynamic theory. This work was supported by the U.S. National Science Foundation under grant DMR-0209542 and by the U.S. Department of Energy under grant BES-Materials Science, under contract W-31-109-ENG-38.

Coherent Diffraction Imaging

Inversion of Diffraction from Objects with Complex Density

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The use of hard X-rays on crystalline materials gives rise to Bragg diffraction. In the simplest approximation of ideal crystals, this yields an identical copy of the forward diffraction centered around each Bragg peak, which contains the information about the shape of the crystal. The use of diffraction patterns surrounding Bragg peaks allows individual grains to be selected for analysis one-by-one, but greatly facilitates the recording in three dimensions (3D), as demonstrated for micron-sized gold crystals. The inversion is formally identical to that of the forward scattering, but reveals only the density of the crystalline part of the sample, so is highly sensitive to defects.

The use of diffraction also opens the new possibility of directly imaging the strain fields within the crystal, an opportunity that is exploited in the current work. It easy to demonstrate that the presence of strain breaks the local symmetry of a diffraction pattern about the Bragg point, which would otherwise show inversion symmetry (as it does about the origin according to Friedel's law). It has been shown that, without loss of generality, the density of a crystal can be considered to be complex function whose magnitude is the physical electron density and whose phase is the projection of the local strain onto the reciprocal lattice vector of the Bragg peak about which the diffraction is measured.

Since there are twice as many independent measurement points for an asymmetric diffraction pattern than a centrosymmetric one and twice as many variables needed to describe a complex density function as a real one, the information content of the problem is the same. The oversampling criterion is the same to allow such a pattern to be inverted. In our experience with test calculations, we have found no additional difficulty in the convergence of the HIO-like algorithms for the complex problem, and this has been confirmed by others. The greatest sensitivity arises from the choice of the support constraint, just as it does for the real problem.

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Coherent Diffraction Imaging at Third and Fourth Generation X-Ray Sources

Chapman H. N.[1], Barty A.[1], Beetz T.[5], Cui C.[2], Hajdu J.[6], Hau-Riege S.P.[1], He H.[3], Howells M.[2], London R.[1], Marchesini S.[1], Shapiro D.[4], Spence J.C.H.[3], Weierstall U.[3]

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We are assessing X-ray diffraction microscopy as a means to perform high-resolution three-dimensional characterisation of non-periodic isolated objects (particles). Several recent experimental and computational developments have enabled us to perform full 3D X-ray diffraction imaging, with high resolution in all three dimensions. These 3D reconstructions were performed from the diffraction data alone. These developments include the Stony Brook diffraction apparatus [1], which allows 3D diffraction datasets to be quickly acquired; the *Shrinkwrap* phase-retrieval algorithm [2], which allows images to be reconstructed *ab initio* from incomplete diffraction datasets; and a fast distributed FFT [3] and reconstruction software implemented on a computer cluster, which allows 1024^3 diffraction datasets to be phased in several hours. We have achieved high-resolution 3D reconstructions of both well-characterized test objects and of mesoporous foam particles that cannot be otherwise characterized. We find that high resolution imaging of thick objects can only be attained in the context of 3D measurement and reconstruction. Reconstruction from diffraction data acquired over many sample orientations allows one to avoid defocus (depth of field) artifacts as well perform a quantitative measurement of refractive index that is not possible from single-view diffraction data.

Resolution of X-ray diffraction imaging will ultimately be limited by radiation damage [4]. One eventual goal is to surpass damage resolution limits of individual particles by using streams of identical particles, such as protein macromolecules, using flash-imaging by X-ray free-electron lasers (XFELs) [5]. Models show that these methods should allow close to atomic resolution imaging. Upcoming experiments at the DESY VUV-FEL will be described where we will attempt to perform diffraction imaging of the damage of particles as they are exposed to short, intense X-ray pulses, to verify models of short-pulse damage.

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Antiphase Domains and Coherent X-Rays

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A decade ago Sutton et al. reported the first experiment with partially coherent X-rays where a *static* speckle pattern, related to antiphase domains contained in the coherently illuminated sample volume, was resolved in the superstructure peak of Cu_3Au [1]. Meanwhile synchrotron sources have become powerful enough that even the *dynamics* of antiphase domains can be measured applying X-ray photon correlation spectroscopy (XPCS). Recently, results for the B2-ordered $Co_{60}Ga_{40}$ intermetallic phase were reported [2]. There, XPCS data were analysed by the so-called detrended fluctuation analysis (DFA) technique, which turns out to be particularly suited for analysing very slow dynamics. A brief introduction to this method will be given.

Besides the results from the dynamics measurements one could wonder whether it is possible somehow to find out the exact arrangement of the antiphase domains in real space. It was shown that in principle it is possible to reconstruct the electron density of the scattering object from (oversampled) diffraction data by means of iterative algorithms that transform back and forth between real and Fourier space, applying appropriate constraints in each domain [3–5]. Thus it would be obvious to apply such algorithms also to antiphase domain data. Antiphase domains, however, cannot be distinguished by their electron density since there are the same kind of atoms present in each domain. Though, photons scattered by two adjacent antiphase domains get phase shifts that differ by π. This means that the antiphase domain structure is mirrored in the reconstructed *phases* [6]. Another problem results from the fact that the antiphase domain structure is not a compact object. Instead the beam spot on the sample determines the overall shape of the real space image. An attempt to reconstruct the antiphase domain structure of a B2-ordered $Fe_{65}Al_{35}$ single crystal will be presented.

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Imaging Magnetic Domains by X-Ray Spectro-Holography

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While holography has evolved to a powerful technique in the visible spectral range, it is difficult to apply at shorter wavelength as no intrinsically coherent (soft) x-ray laser is yet available as a light source. The progression from visible light towards shorter wavelength is motivated by the increase in spatial resolution that can be achieved. Of equal importance is the possibility to exploit special contrast mechanisms provided by scattering in resonance with transitions between electronic core and valence levels. We demonstrate magnetic imaging by x-ray spectro-holography, exploiting x-ray circular dichroism as a contrast mechanism. Images of magnetic domain patterns forming in thin film Co-Pt multilayers with perpendicular anisotropy are presented. The images are obtained by direct Fourier inversion of the scattering pattern, without the need of phase retrieval or an iterative computing process. Currently, we achieve a spatial resolution of 50 nm at an x-ray wavelength of 1.59 nm. [1] Holography at this wavelength is made possible by combining the sample with a nanostructured mask. An advantage of this approach is that there are no severe space constraints around the sample, making it easy to realize extreme sample conditions such as high magnetic fields or low/high temperature. Here, we present domain images in an applied magnetic field for several magnetic multilayer systems and discuss future opportunities for single shot imaging experiments at free electron x-ray lasers.

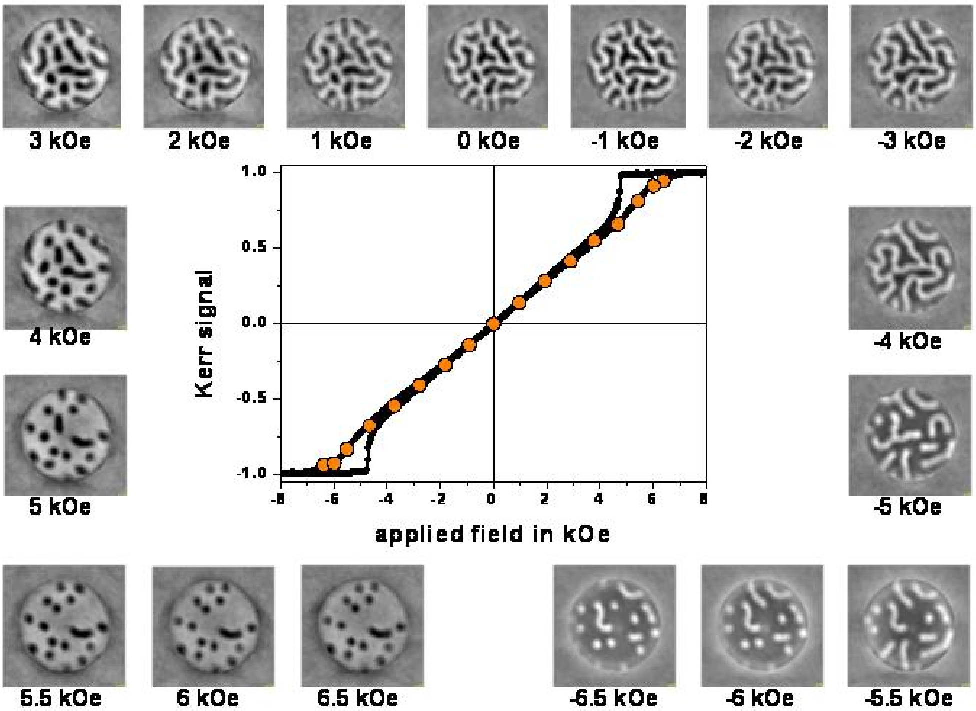

Figure 1: Spectro-Holography images of a reversal sequence during an external field sweep for a Co/Pt multilayer. The MOKE hysteresis loop of the sample is shown in the center.

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Diffraction Microscopy of Biological Specimens: Imaging of a Freeze-Dried Yeast Cell

Shapiro D.[1,4], Lima E.[1], Elser V.[2], Howells M.R.[3], Huang X.[1], Jacobsen C.[1], Kirz J.[1,3], Miao H.[1], Neiman A.[5], Sayre D.[1], Thibault P.[2]

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In our experiment, the X-ray diffraction pattern of a freeze-dried yeast cell was collected at 750 eV and phasing was performed using the difference map algorithm [1]. Data collection made use of an undulator beamline at the Advanced Light Source [2], and a specially designed apparatus [3]. The freeze-dried yeast cell was 3 microns in diameter and its exit wave was complex-valued at 750 eV. The reconstruction of complex valued objects has been found to be particularly challenging [4,5], and the success of the reconstruction illustrates a step forward in this technique. The reconstructed image shows the nucleus and cell membrane clearly in 30 nm resolution. The reconstruction of a freeze-dried yeast cell gives us confidence that the phasing algorithm would work when one has diffraction data from frozen hydrated biological samples, which most resemble the living biological state.

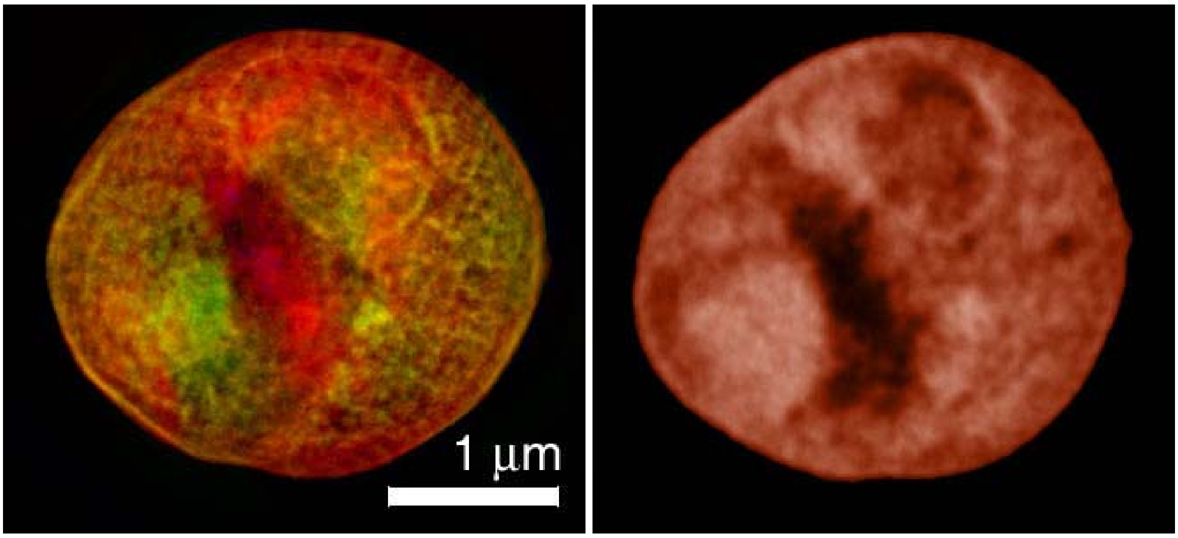

Figure 1: diffraction microscopy reconstruction (left) and scanning transmission x-ray micrograph (right, made with a 45 nm outermost zone width zone plate) of a freeze-dried yeast cell.

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Coherent X-Ray Diffraction on Quantum Dots

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Coherent x-ray diffraction (CXD) is a new experimental method for studying perfect and imperfect crystals. The method has become available by the recent development of high-brilliance third generation sources of synchrotron radiation (ESRF, APS, SPRING-8). The beams coherence volume being of the order of few microns can entirely enclose a nano-size object. Instead of incoherent averaging, a coherent sum of amplitudes produces a coherent diffraction pattern originating from the real space arrangement of the sample. If high-quality x-ray lenses were available as they are for electrons, such diffraction patterns could be transformed to magnified images directly.
In this talk we will show how the objective lens of the microscope can be replaced by a special iterative phase reconstruction procedure that inverts intensity measurements of the CXD pattern to real space image. The method is based on the fact that the diffraction pattern can be oversampled relative to its spatial Nyquist frequency so that the Fourier transform can be overdetermined in spite of missing phase information. In principle this method does not have any limitations on the available resolution.
In our previous publication [1] it was shown how 3D images of the interiors of Au crystals of micron size can be obtained applying this technique. The size of the objects can be further reduced to the size of the quantum dots samples if repetitive motive in the form of 2D crystal is used. It will be demonstrated that in the case of coherent illumination of these samples the correct shape and orientation of individual island can be obtained. In the case of partially coherent illumination the correct shape of the particle can be obtained only when the coherence of the incoming beam is reduced to match the size of the island [2]. In the last example experimental results of CXD scattering on the sample of specially fabricated GeSi islands of nanometer size and in a regular array embedded to Si substrate will be shown [3]. Two geometries of scattering that is grazing incidence diffraction (GID) and grazing incidence small angle x-ray scattering (GISAXS) were used. Applying a microfocuse coherent beam on our sample give rise to coherent diffraction pattern with Bragg spots and broad diffuse maxima in GID geometry. The GISAXS pattern has a typical shape resulting from the periodic array of identical islands. This diffraction pattern was used to reconstruct the average shape of the islands using a model independent phase retrieval algorithms.

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Toward Sequential Image Reconstruction with Large Area Detector in Hard X-Ray Diffraction Microscope

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In x-ray diffraction microscopy, it recently became possible to reconstruct the sample image only from the oversampled Fraunhofer diffraction pattern due to a couple of proposed techniques in data analysis and in data acquisition. An example is the iterative normalization algorithm proposed by our group [1]. These techniques have opened a way for sequential image reconstruction in parallel with measurement, which can provide quick feedback for data acquisition. To perform faster image reconstruction during experiment, we are developing a dynamic reconfigurable processor to quickly perform fast Fourier transform, which makes it possible to take a quick look at the reconstructed image in a few minutes.

To achieve higher spatial resolution, we have been using short wavelength hard x-rays at SPring-8. In addition, we are developing a large-area in-vacuum imaging plate detector and a vacuum chamber. The imaging plate is a radiation image sensor using photo-stimulated phosphor. It is easy to make the detector area large and in our design it is 125 mm square, while the single pixel size is as small as 25 micron square to satisfy the oversampling condition.

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Aligned Protein-Beam Diffraction

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Apparatus is under construction at ASU physics (electrons) and at the Advanced Light Source in Berkeley (X-rays) to obtain diffraction patterns from a single-file submicron liquid droplet stream. The aim is to solve proteins which cannot be crystallized, and help understand protein folding. Each water droplet contains, on average, one protein. The droplets freeze by evaporative cooling to from a vitreous ice jacket. The molecules are aligned by a 100 watt CW fiber laser - induced polarization generates a torque as for the laser-wrench, which depends only on the RMS value of the CW laser intensity. Each molecule receives much less than the critical damage dose during its transit across the X-ray beam, but many doped droplets fall within the beam at any instant. All three beams, laser, X-rays and droplets, run continuously, and diffraction data is aquired continuously until adequate signal-to-noise is achieved. The laser polarization is then rotated into a new orientation using a quarter-wave plate, allowing tomographic diffraction data collection for three-dimensional reconstruction. The phase problem is solved by iterative Gerchberg-Saxton-Fienup methods to about 0.7nm resolution. The requirements of laser power and droplet temperature needed to achieve sub-nanometer resolution and so observe the secondary structure of proteins will be described in detail, together with damping and thermal fluctuation limits. Experimental images of our monodispersed Rayleigh droplet beam will be shown, together with the layout of the X-ray water jet diffraction camera. The project is described in detail elsewhere [1]. Supported by NSF funding.

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Coherent Nanoarea Electron Diffraction and the Solution of Phase Problem

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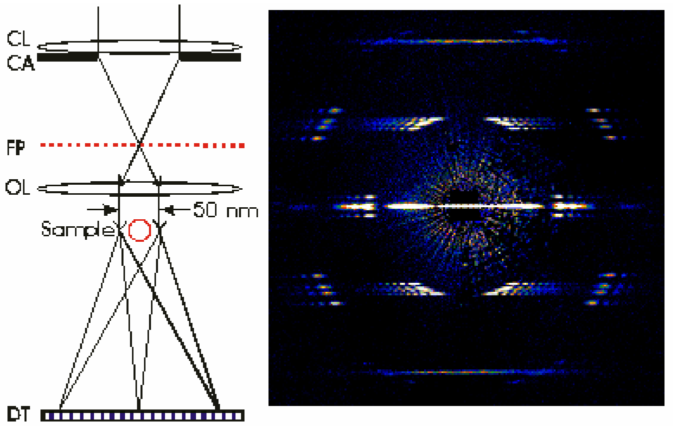

(left) A single double wall nanotube is illuminated with a narrow beam of electrons. (right) The diffraction pattern of the tube

This talk reports the new electron diffraction technique using a coherent nanometer-sized parallel electron beam and how the missing phase in the diffraction pattern can be retrieved to reconstruct the image. The talk will be organized in two parts. The first section covers the electron probe formation, its coherence and recording of electron diffractions from individual nanostructures, such nanoparticles, carbon and boron nitride nanotubes. The second part describes our approach to phase retrieval and looks into the issue of the missing central beam, convergence and uniqueness of phase solutions, which are general with significance to X-ray diffraction. Examples of phase retrieval and image reconstruction include carbon nanotubes, bundles and multi-wall boron nitride tubes.

*In collaboration with M. Gao, J. Tao, I. Vantanyants, R. Zhang, L. Nagahara, R. Twesten and I. Petrov

Coherent Magnetic Scattering at the ALS

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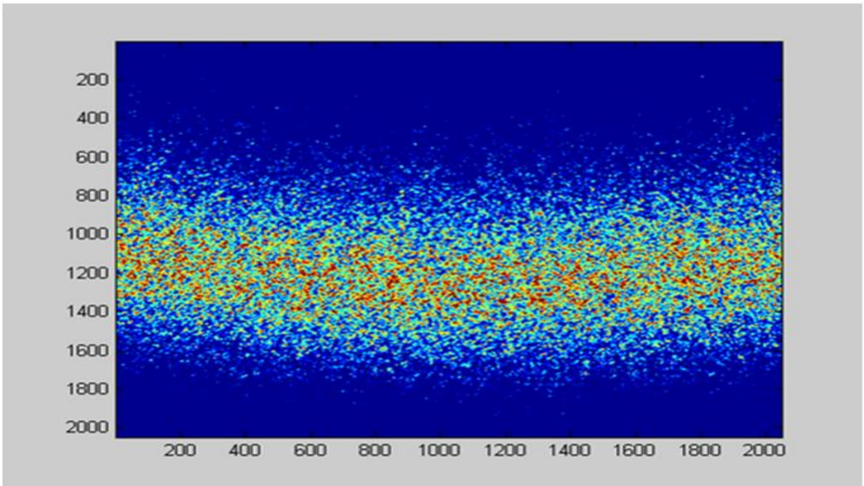
The development of Resonant Magnetic Scattering (XRMS) in the soft X-ray range provides increasing opportunities to image magnetic domains [1], as well as study magnetic order and reversal processes in nanostructures [2,3]. A new endstation dedicated to XRMS studies with the specific use of coherent light has been installed at the Advanced Light Source (ALS), Berkeley and commissioned in early 2004. This new setup includes a cryogenic sample holder, a scattering chamber with 2-dimentionnal detector and an octopolar electromagnet that allows to apply an in-situ magnetic field in any direction. XRMS measurment can be performed either in transmission or reflection geometry. Besides the chemical selectivity, this scattering technique gives the possibility to penetrate thin layer in depth and study the magnetic ordering at the nanoscopic scale. Furthermore, the polarization sensibility and the ability to rotate the magnetic field allow to discriminate different components of the magnetization. The use of coherent light and 2D detection provides remarkable speckle patterns (see Figure below) that are related to the local magnetic topology. Moreover, the evolution of the speckle pattern can be monitored in time, in order to study slow dynamic effects in correlation with different parameters like temperature and applied magnetic field. First measurments have already been performed on different kind of systems, as manganites, thin magetic film with perpendicular exchange bias and superparamagnetic nanoparticles [4]. First results obtained on Co and Fe3O4 nanoparticle assemblies [5] will be presented, showing in complementary way scattering results with incoherent and coherent light, as well as the evolution of the speckle pattern under different conditions.



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Poster Abstracts

International Workshop on

Phase Retrieval and Coherent Scattering

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P62 **Phase Sensitive Imaging with X-Rays and Neutrons - Parallels and Differences -**
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P64 **Hard-Sphere Colloids in the Fluid Phase Probed by X-Ray Photon Correlation Spectroscopy**
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High-Resolution *Ab Initio* Three-Dimensional Coherent X-Ray Diffraction Microscopy: Status and Challenges

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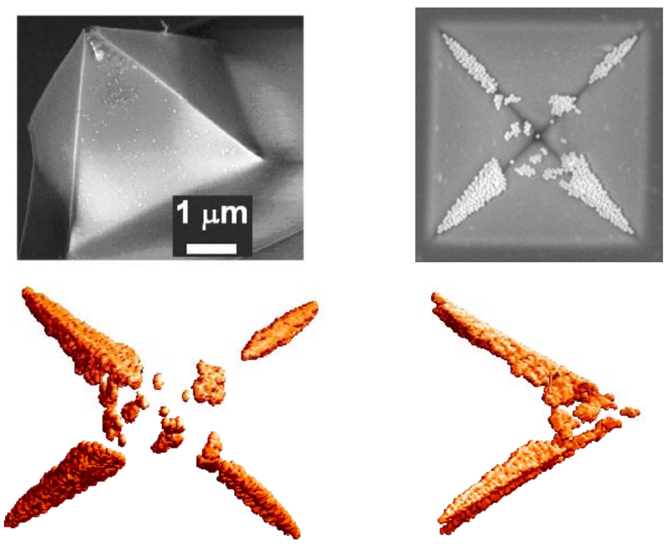
Three-dimensional diffraction microscopy is an imaging mode that relies on the numerical inversion of a diffraction volume to recover the object function, and offers the potential for high-resolution aberration-free diffraction-limited 3D images without the resolution and depth-of-field limitations of lens-based tomographic systems.
Critical steps in obtaining a high-quality image are assembly of the diffraction data into a diffraction volume and the application of phase retrieval techniques to the diffraction volume to obtain an object function. Key issues include data assembly and alignment, how to treat missing data within the beamstop region and elsewhere, techniques for determining the object support, and stability of the three-dimensional phase retrieval process.
We have recently had success in obtaining high-quality 3D reconstructions of several test objects using only X-ray diffraction data as input to the reconstruction process. This is an important step, as it does not require a low-resolution image to fill in the beamstop region. In this paper we will present recent 3D reconstruction results, discuss key aspects of the reconstruction process, and describe how we can produce a high-quality reconstruction in less than 2 hours of computer time. The structures obtained will be compared to other 3D analysis techniques to demonstrate that the diffraction imaging reconstruction matches results obtained using other techniques.



Example of high-resolution 3D reconstruction from diffraction data. Top row are scanning electron microscope images of the test object and below are 3D surface renderings of the reconstructed object made using X-ray diffraction data alone.

Evolution of Co/Pt-Covered Nanolines under Magnetic Field using Coherent Soft X-Ray Resonant Magnetic Scattering

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We report on the soft x-ray resonant magnetic scattering (SXRMS) from etched lines in silicon covered by a Co/Pt multilayer with perpendicular magnetization. This system was previously studied by incoherent radiation [1]. Using a 10-µm diameter pinhole at 7 mm in front of the sample, we increased the transverse coherence of the incident x-rays while illuminating only a small sample area. These conditions allow us to obtain a speckle pattern in magnetic scattering by using a CCD camera [2]. The experiment was performed in reflection geometry on beamline ID8 at the ESRF. The linearly polarized incident beam was tuned to the energy of the Co L3 edge (778 eV).

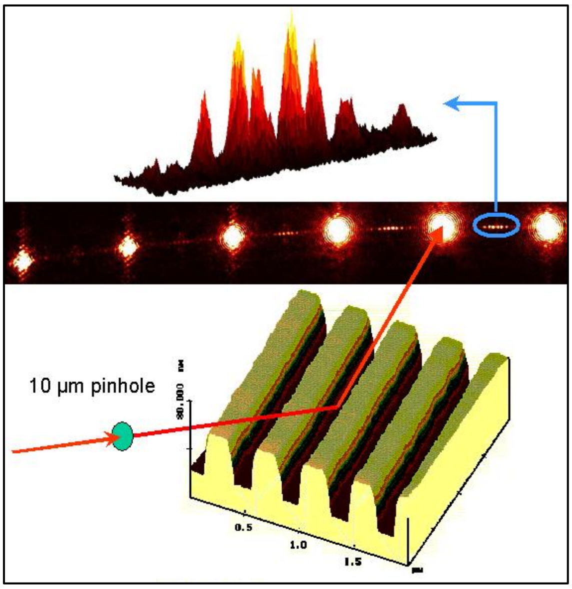

Figure 1: The speckle pattern in coherent SXRMS from Co/Pt-covered etched lines.

The speckle pattern is related to the local magnetic configuration of the lines that are illuminated by the beam through the pinhole. Using a pulsed perpendicular magnetic field, we studied the reversal of the lines through the changes in the speckle pattern [3]. The comparison between major and minor magnetization loops shows that saturating the sample erases its magnetic memory, whereas a smaller field does not prevent to retrieve the original magnetic state. These results are of major importance for application of this type of material to spinelectronics, such as non-volatile data storage materials.

In order to retrieve the real-space magnetic configuration, we developed an original reconstruction algorithm based on Monte Carlo with simulated annealing, benefiting from the two important advantages of our system: the lines are almost monodomain, so that we can associate a single unknown to each line, and their perpendicular magnetization restricts these unknowns to only two possible values: +1 and –1.

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XPCS and Self-Organization of Metal Surfaces

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X-ray photon correlation spectroscopy (XPCS) probes the dynamic properties of matter by analyzing the temporal correlations among scattered photons.
XPCS has already shown the potential to impact several areas of statistical physics and provide access to a variety of important dynamic phenomena, like the time-dependence of equilibrium critical fluctuations and the low frequency dynamics in disordered hard and soft condensed matter materials.
In this contribution, we present results obtained at ESRF (beamline ID10A) in two class of experiments devoted to study self-organizing phenomena in metal surfaces.
The first series of experiment is dedicated to the study of the growth and time-evolution of rippled surfaces; the rippling effect is induced by bombarding a metal surface with energetic ions ($E \leq 1$ KeV).
The second series of experiment is dedicated to the de-wetting process of metal films grown on an inert substrate and then heated at high temperature.
In both systems it is possible to follow the evolution of the diffracted intensity but the data analysis is complicated by the fact that the system is not ergodic and its average scattering evolves continuously in time, without reaching the equilibrium
In this contribution we will present experimental results with a preliminary interpretation.

The Phase of the Coherent Forward Scattering Amplitude as Revealed by Stroboscopic Detection of Nuclear Resonant Scattering of Synchrotron Radiation

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The loss of phase information in an experiment prohibits direct conversion of the measured x-ray intensities into structural information like atomic coordinates. X-ray interferometers have been applied for various problems, like precise measurement of atomic scattering factors, phase contrast imaging, etc. [1].

Interferometry has recently been combined with nuclear resonant scattering (NRS) of synchrotron radiation (SR) [2,3] in order to extract hyperfine-specific structural information from nuclear forward scattered time spectra. The phase problem prohibiting direct conversion of energy from time domain spectra can be solved by four flux measurements using a triple Laue (LLL-) interferometer [3].

A novel NRS method uses stroboscopic detection of forward scattered [4] and grazing incidence [5,6] SR. The signal is recorded in a 2D array as a function of both the time elapsed after the SR pulse and the velocity v of a reference scatterer on a Mössbauer velocity drive that scans through the hyperfine splitting range. The stroboscopic spectrum is essentially a sum of periodically shifted energy domain spectra (the 'stroboscopic orders') the period being determined by T, the SR bunch period [4]. The phase information is encoded in the measured 2D array by the interference of varying the phase between the resonance lines of sample and reference at various velocities. Inversely, grouping the data into time windows, an energy spectrum, similar to a conventional Mössbauer spectrum can be constructed. Selecting different time windows relative to $T/2$, a known phase is mixed to the unknown phase of the scattering amplitudes and the hyperfine-field-specific structural (phase) information becomes accessible. The intensity of the n-th stroboscopic resonance corresponds to that of an LLL interferometer [4].

In the accessible grazing incidence angular range around the critical angle and the multilayer Bragg reflections, due to the nuclear speed-up, the stroboscopic orders heavily overlap. This allows for an extraction of the phase difference between the electronic and the energy dependent combined (electronic + nuclear) reflection [5].

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Fast APD Arrays and Highly Resolved X–Ray Beam-Position Monitoring for XPCS

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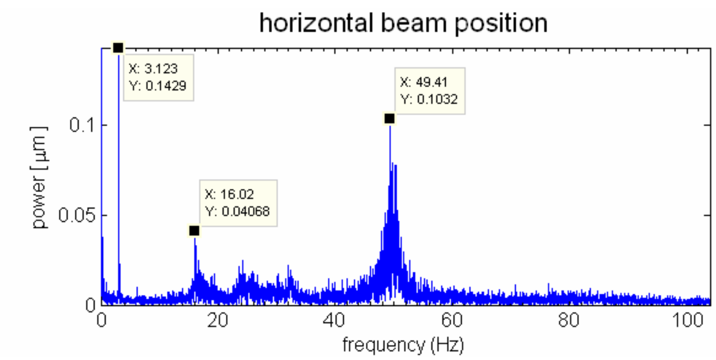
X-ray Photon Correlation Spectroscopy (XPCS) is one of the modern techniques that fully exploit the coherence of x-rays generated by third generation sources. It has a high potential due to the possibility to study systems that are opaque for visible laser light and due to the accessible q-range. Prerequisite for XPCS measurements are (1) highly coherent and (2) stable sources and (3) fast detection systems. We addressed (2) by x-ray beam position measurements with sub-micron resolution at 200 Hz and (3) with a high speed APD array for that the readout electronic is currently developed at PSI. Spatially resolved measurements of the complex coherence function (1) are discussed in a separate contribution.

X-ray beam-position measurements were performed at the undulator beamline X06SA of the Swiss Light Source (SLS). A fast x-ray camera built in-house with 5 μm effective pixel size and sub-region frame rates beyond 200 Hz was used as the detection system. A Gaussian was fitted to each of the recorded beam profiles. This procedure results in 2D beam position data with sub-pixel resolution in the 0.1 μm regime. We show that even periodic perturbations of the electron beam like the SLS booster frequency are easily detected. The strength of the system is the characterization of vibrations of optical components caused by pumps, cooling systems etc.

The real breakthrough of XPCS is currently hampered by the detection systems available. To this end, high speed readout electronics are developed at PSI for an Avalanche Photo Diode (APD) array of eight times eight pixels. The ultimate goals are event rates beyond 10 MHz with a dynamical range of 10^7. The current status of this project is presented.



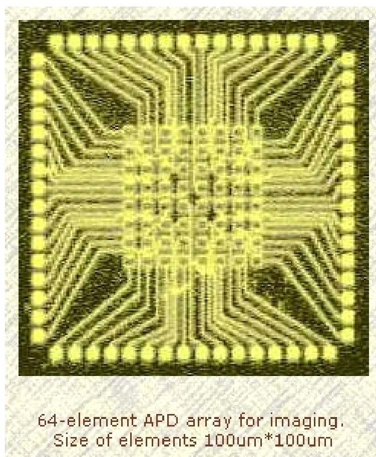


Figure 1: Perturbations of the horizontal beam position as a function of frequency (left) and a prototype of the high speed 8x8 APD array (right).

Reference

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Heterodyne X-Ray Speckles. A Novel Low-Angle Scattering Technique

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X-Ray speckles can be generated by spatially filtering the beam with pinhole arrangements, so to make the radiation falling onto the sample spatially coherent. At variance, we will present evidence that high quality, low contrast speckles can be observed by simply placing a high resolution CCD sensor immediately behind a static sample.

Measurements have been performed at the BM05 beamline of the European Synchrotron Radiation Facility (ESRF) in Grenoble with an energy of 12 KeV. Microporous filters with pore size in the range [0.45-8] μm were placed at 50m from the source, the only optical component in between being a double crystal monochromator. The diffracted intensity was collected by using a Frelon camera and the sample-camera distance z was varied from some millimeters up to about 5 meters.

The diffracted intensity has the typical speckle distribution as routinely observed with optical coherent sources. The speckle pattern has been analyzed by studying the two point intensity correlation function. The speckles are remarkably circular and their width does not change for distances typically up to about 50 cm. As z is further increased, the speckles begin to loose contrast, and eventually become asymmetric.

It is argued that the speckles are due to a self-referencing scheme where both the scattered radiation and the heterodyne local oscillator originate from the same rapidly changing, local coherent beam patch. It is suggested that these X-Ray speckles are of the same type of those obtained with the newly reported optical “Near Field Scattering” method [1,2] that has been shown to be equivalent to static light scattering.

The simple lensless arrangement described above could be used as a new ultra low-angle scattering method operative where conventional X-Ray scattering methods fail.

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Coherent X-Ray Diffraction for Phase Retrieval: Shape and Strain Reconstruction of a Single Microcrystal

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It has been shown that the oversampling of the diffraction pattern can solve the phase problem [1]. The 3D electron density distribution can be obtained through inversion algorithms without the need of a priori hypothesis. While it is well established for optics, this technique remains challenging for x-rays, where a highly brilliant source, such as the one delivered by third generation synchrotrons, is needed [2]. We will present first x-ray coherent diffraction experiments performed at the ID1 beamline at ESRF (France) [3]. The 3D coherent diffraction pattern from a single micrometric Au crystal is investigated. After the description of the experimental setup and the presentation of the first results (diffraction and inversion, fig. 1), we will discuss the issues emphasized by this experiment: (i) use of a beamstop, (ii) source size related contrast, (iii) possible use of KB mirrors for the investigation of nanometric particles.

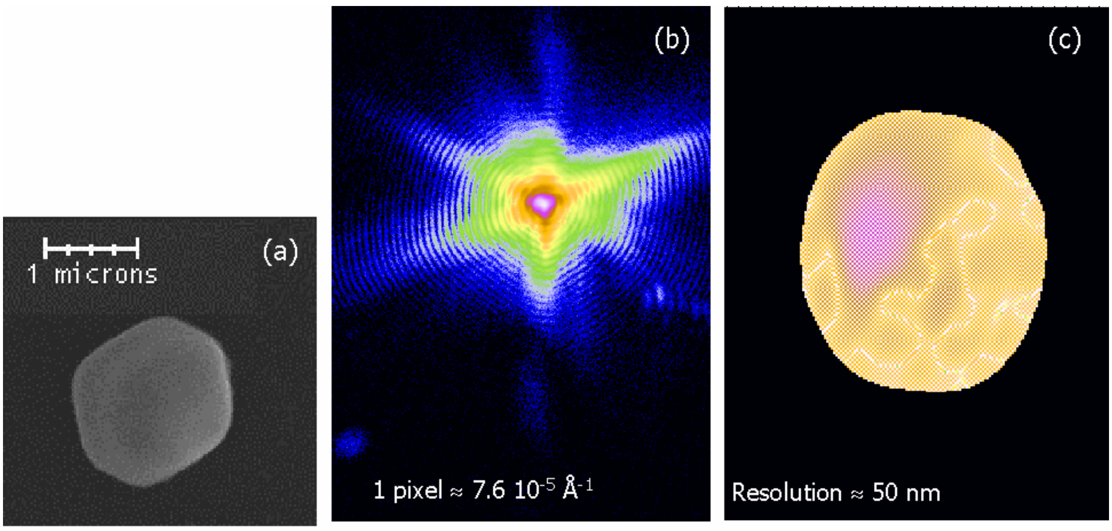

Figure 1: Coherent X-ray diffraction experiment. (a) Scanning Electron Microscopy image of a typical Au crystal, (b) oversampled diffraction pattern measured in the center of the (111) Bragg reflection and (c) the corresponding reconstruction of the electron density projected perpendicularly to the (111) direction.

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Hard X-Ray Wave-Front Sensing with Moiré Interferometry

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We present a method to measure wave front distortions in the hard x-ray range based on a grating interferometer. It consists of two transmission gratings, of which the first with phase shifting lines essentially splits the incoming wave into the +1st and -1st diffraction order. The interference of these waves create a periodic intensity modulation downstream of the beam splitter grating, which is analyzed by looking at the transmission through a second grating with absorbing lines. A slight rotational misalignment of the two gratings around optical axis generates a pattern of moiré fringes. For a plane incident wave, (Fig. 1a), a pattern of parallel fringes perpendicular to the grating lines is observed (Fig. 1b). A plane but tilted wave (Fig. 1c) creates a shift in the moiré pattern (Fig. 1d), while a non spherical (Fig. 1e) wave causes a slope of the moiré fringes (Fig. 1f).
We have applied the method to measure the wave front distortions caused by the imperfections of a W/Si multilayer mirror. We were able to detect moiré distortions of 1/50 fringe period, corresponding to wave front distortions as small as 10^{-12}m (i.e. $\lambda/100$). This sensitivity translates mirror slope error of about 100 nrad. This means, that the method is capable of distinguishing between a perfectly planar mirror and a mirror curved with a radius of many kilometers.

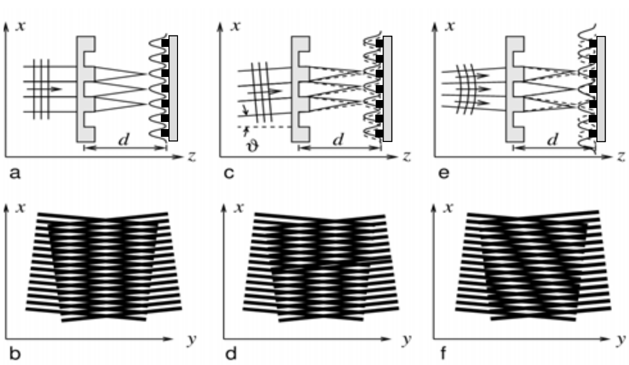

Figure 1: Different wave front distortions and their effect on the moiré fringes observed in a grating interferometer.

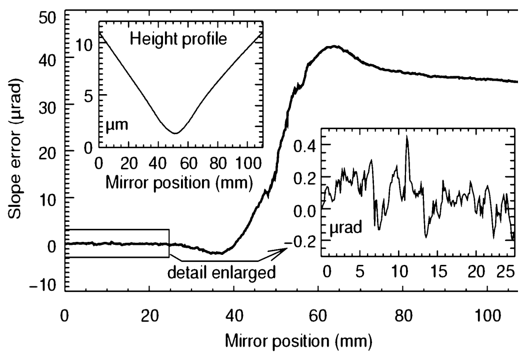

Figure 2: Slope error measurements of a W/Si multilayer mirror measured at 12.4 keV energy with a sensitivity in the range of 100 nrad.

Reference
T. Weitkamp, B. Nöhammer, A. Diaz, C. David, E. Ziegler, *X-ray wavefront analysis and optics characterization with a grating interferometer,* Appl. Phys. Lett. 86, 054101 (2005)

Fabrication of Si and Au Microgratings for Hard X-Ray Interferometry

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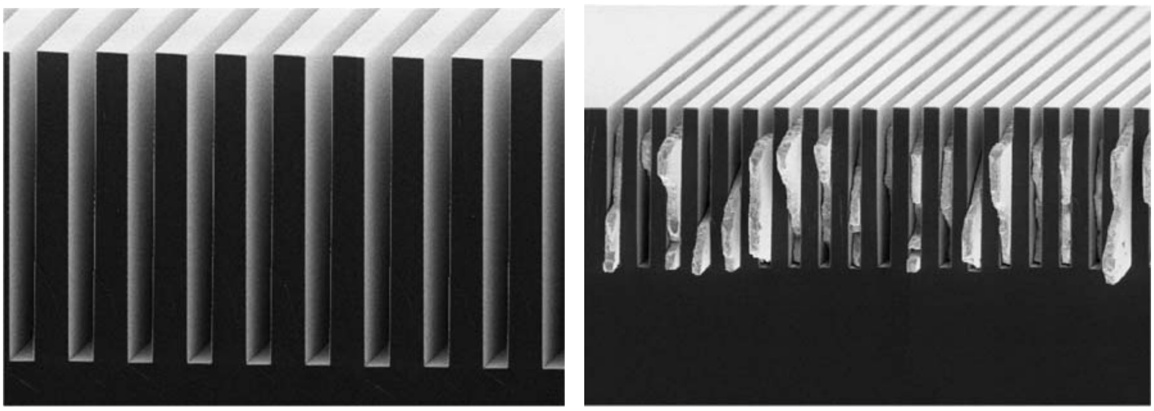
X-ray grating interferometers are used in the 10-20 keV energy range for different experiments related to coherent x-rays. These include phase contrast imaging [1], coherence measurements [2], and wave front analysis for x-ray mirror characterization [3], all of which are topics for this workshop. The interferometers developed by us for these purposes essentially consist of two transmission gratings: a Si phase grating acting as a beam splitter and a Au amplitude grating used as analyzer. In this contribution, we would like to present the fabrication methods to obtain these structures, which typically have periods of 4 and 2 microns. E-beam lithography is used to define the grating pattern. By anisotropic wet etching of the Si <110> substrates, the pattern can be transferred into high aspect ratio structures with very precise dimensions. Electroplating is applied to fill the grooves of Si gratings with Au to obtain high-contrast analyzer gratings.



Cross sections of the gratings used in the x-ray grating interferometer: 4 µm period Si phase grating with a height of 22 µm (left) and 2 µm period Au amplitude grating with a height of 12 µm (right).

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Diffraction Imaging with Complex-Valued Electron Density Reconstruction

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The recent development in coherent x-ray diffraction imaging shows necessity of retrieving the electron density distribution as an complex-valued object caused by the anomalous scattering of atoms. For using synchrotron x-rays, the imaginary part of dispersion correction noticeably contributes to the function of atomic scattering for heavy atoms. Using the Elser's approach developed for the complex-valued object reconstruction [1, 2], we have shown that the neglect of the x-ray absorption leads to significant errors during electron density retrieval. Within the framework of the complex-value reconstruction a new approach for auto-retrieving of the missing intensity data [3] has been realized.
The same algorithm is also applicable to the electron diffraction imaging. Since the electrons interact strongly with the crystal potential of the sample, instead of the electron density distribution, the projected crystal potential of thin sample is retrieved in a kinematical diffraction regime. Electron diffraction patterns of [001] and [111] from a MgO particle of 25nm in size were recorded. The retrieved crystal potential maps from these two crystallographic orientations will be presented. The methodology and retrieved potential maps will be discussed in detail in the workshop.

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Diffraction from Periodic Arrays of Oxide-Filled Trenches in Silicon: Investigation of Local Strains

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Mechanical stresses in thin films and nanostructures have very important consequences on the reliability of devices made from such structures. With the general trend of reduced dimensions in microelectronic devices (less than 0.2 μm), mechanical stresses are more and more considered as a very important issue. A number of experimental methods are available for average strain determination but local strain measurements at the scales required here are much less common. Moreover these fine-scale strain measurement methods generally do not preserve the stress state of the sample being investigated (Convergent Beam Electron Diffraction) or do not have sufficient spatial resolution (micro-Raman spectroscopy). We propose here to investigate the periodic strain field induced in a single crystal substrate by High Resolution X-ray Diffraction (HRXRD). HRXRD is shown to be very sensitive to local strains and has the distinct advantage of being non destructive.

We investigate here periodic arrays of SiO_2-filled trenches in single crystal (001) Si. These trenches induce a periodic strain field in silicon, which gives rise to distinct satellites in reciprocal space. The local strain field is worth investigating because Shallow Trench Isolation (STI) is a key step in the production of non-volatile-memories. The trenches are etched either along [110] or [100] and we have investigated different periods Λ (0.58 μm and 2 μm) as well as different w/Λ ratios, where w is the trench width. The HRXRD experiments have been performed with a laboratory source, a 4 reflections Ge (220) Bartels monochromator and a 3 reflections Ge (220) analyzer. For each sample we have recorded Si 004 and Si 224 or Si 404 reciprocal space maps. The results will be discussed with respect to mechanical modeling of strain fields with Finite Elements Method.

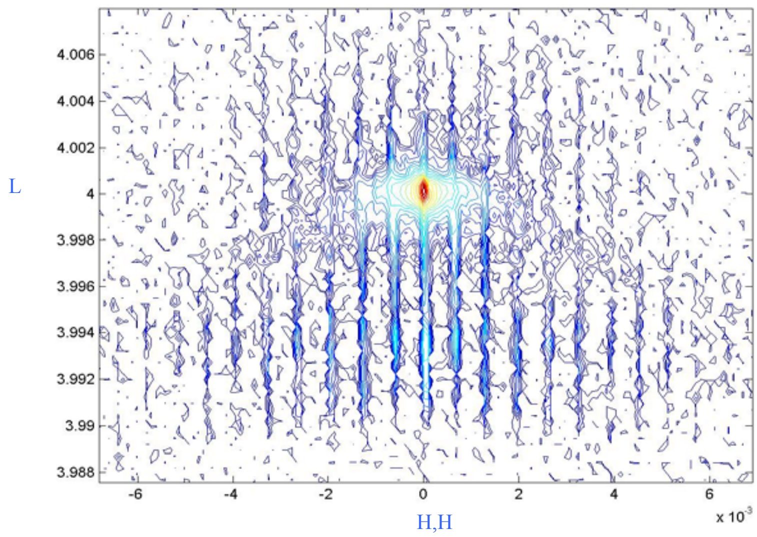

Figure 1: Map of reciprocal space around Si 004 from trench array with a 0.58 μm period.

Dynamics of Fluctuations of Colloidal Particles under Shear Flow with XPCS

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X-ray photon correlation spectroscopy (XPCS) offers an unique way to probe slow nanoscale dynamics in various soft- and hard- condensed matter systems. In this work we will show how XPCS can be combined with microfluidics in order to probe the intrinsic dynamics of liquid samples under shear flow.

Microfabricated fluid circulators are being used nowadays to study micro-scale flow regimes both with the purpose to achieve new functionality or as an experimental tool. A particular benefit of such an experimental arrangement is that it allows to study samples that otherwise may suffer from beam damage.

In our experiments we chose a "standard" sample consisting of monodisperse sterically-stabilized polymethylmethacrylate (PMMA) colloidal solution which was studied in detail with XPCS. The sample was flown through an in-house designed fluid circulator compatible with SAXS. The correlation functions of the fluctuating SAXS intensity were recorded by a hardware correlator for several values of the scattering vector q, and for a wide range of flow rates measured by Reynolds numbers covering more than three decades. An example of our results is shown in Fig. 1. The autocorrelation functions of the scattered intensity, $g_2(t)$, at a single wave vector are show for different flow rates measured by the Reynolds numbers indicated in the legend. Our analysis show how the correlation times measured by XPCS scale with q and with the flow rate.

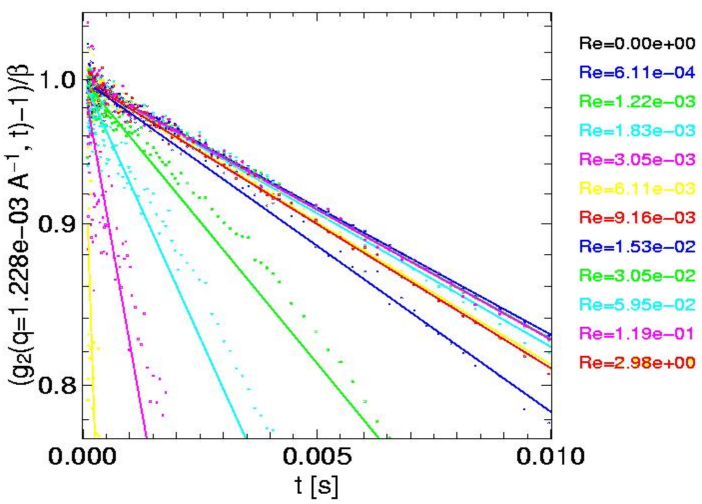

Figure 1: Correlation times in the short-time diffusion limit for different flow rates.

Circular Surfactants Hemi-Micelles at the Air-Water Interface, a System for Coherent Diffraction Imaging ?

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Semi-fluorinated alkanes (SFA, $C_nF_{2n+1}C_mH_{2m+1}$, FnHm) exhibit a lipophobic–hydrophobic character inducing interesting properties[1]. Despite the lack of polar head group, these molecules are able to form Langmuir monolayers, *ie.* monomolecular thick layer at the air-water interface. However the structure of pure SFA monolayers remains controversial. Models based on homogeneous films fail to describe the diffraction data.

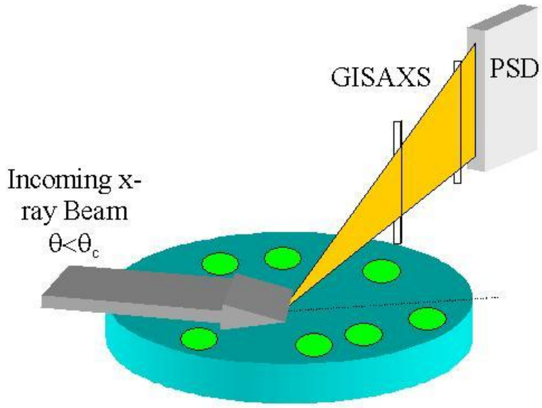

Figure 1: Experimental geometry for GISAXS measurement on Langmuir monolayer in situ at the air-water interface.

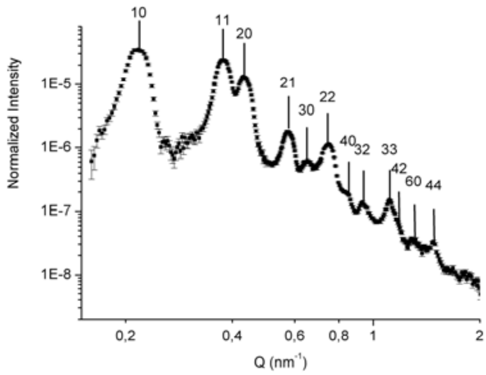

Figure 2: GISAXS spectrum of a Langmuir monolayer of F8H16 measured *in situ* at the air-water interface at surface pressure 3mN.m^{-1}.

After transfer on solid substrate, Atomic Force Microscope (AFM) evidences round domains of 30nm diameter identified by x-ray reflectivity as surface hemimicelles with an hydrogenated core and half corona of fluorinated chains. To prove unambiguously the presence of hemimicelles on the surface of water, Grazing Incidence Small Angle x-ray Scattering (GISAXS) were performed in situ at the surface of water (ESRF ID10B, fig. 1). The GISAXS (fig. 2) evidences 11 diffraction peaks indexed on a hexagonal network of parameter 29.1nm in agreement with the size of the hemimicelles detected by AFM. This experiment demonstrates that SFA form spontaneously hemimicelles of water surface[2]. Although these experiments closed the controversy, the question arises if these hemi-micelles exist at low surface density where there is no organisation of the micelles. We wonder if coherent diffraction imaging could evidence the presence of such micelles. Moreover, a beamline optimised for scattering on soft interfaces and complex systems with tender x-rays (2-10keV) will be built on the SOLEIL synchrotron, and could be an opportunity to test such techniques with tender energy x-ray.

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Waveguide-Based Hard X-Ray Holography

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Two-dimensionally confined x-ray waveguides [1,2] are promising point-like sources for high-resolution x-ray imaging. These sources provide a highly coherent divergent beam that is probably well-suited for in-line holography. Due to the divergence of the exiting beam the spatial resolution is not limited by the detector pixel size but rather by the cross-section dimensions of the waveguide, i.e.typically some tens of nanometers. For one-dimensionally confined waveguides, this was already shown by Lagomarsino *et al* who achieved a resolution of 140 nm in the the direction of the confinement using a planar waveguide with a 130 nm thick guiding layer [3].

We present numerical simulations of waveguide-based x-ray holograms and show the results of the corresponding reconstructions to evaluate contrast and resolution as well as the required flux and optical contrast of the test samples.

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Characterization of the Diversion of Liquid Alkali Metals Structure on the Transition to Nonmetal

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Based on the experimental data on the equilibrium thermodynamic, transport, electrical and structural properties and based on the theoretical investigation, it has been known that liquid alkali metals turn gradually into nonmetals as the temperature is increased and as the liquid is expanded. As the temperature is increased, the valance electrons of the metallic state become more correlated with their parent ions, and close to the critical temperature nonmetallic character dominates the thermodynamic state of the fluid system. According to this diversion of interatomic forces, clusters of difference sizes are formed in the liquid state, while the main component of the vapor state is dimers.

The metal-nonmetal transition in the liquid cesium and rubidium has been investigated widely due to their rather low critical point. Experimental structural investigation has brought meaningful explanation on the transition. The result is that upon transition interatomic distance does not change appreciably but the coordination number drops sharply.

We present results on the metal-nonmetal transition by using statistical mechanical methods and show that the transition occurs on energy as well as range scale. As the critical temperature is approached two distinct common bulk modulus is indication of tractions from one liquid system to another one. The results offer recommendation for further investigation on the nature of the transition with potential applications.

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Phase Contrast Computed Tomography: A Different Approach

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In contrast to conventional hard x-ray absorption radiography (or tomography) based on differences in absorption, phase imaging exploits the coherence of the source and differences in the real part of refractive index distribution of an object to form an image.
As far as phase tomography (quantitative reconstruction of the phase or the refractive index decrement in 3D from 2D phase images) is concerned, all non-interferometric phase retrieval methods [1] are based on a two step approach. First, the projections of the phase are retrieved and then the object function, i.e. the refractive index decrement is reconstructed by applying a conventional filtered backprojection algorithm.
On the other hand, the reconstruction algorithm suggested by Bronnikov [2] presents an alternative approach which eliminates the intermediate step of 2D phase retrieval and provides a direct 3D reconstruction of the object. The reconstruction algorithm is very interesting from the experimental point of view since for a pure phase object it requires only one single tomographic data set.
We implemented Bronnikov's reconstruction algorithm and examined its performance experimentally and numerically. One reconstructed slice of the polyethylene tube with 2 polymer fibers and a hair inside is given in Fig. 1, using filtered backprojection (a) and using the Bronnikov algorithm (b).

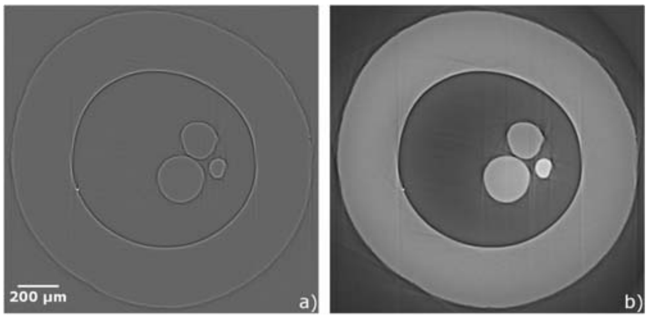

Figure 2: Reconstructed slice of a polyethylene tube with polymer fibers inside: a) using filtered backprojection and b) using the Bronnikov method. The sample to detector distance was 15 cm and energy 13.5 keV.

The advantage of the Bronnikov algorithm compared to conventional filtered backprojection is evident: actually, in a) only the edges are visible while in b) weakly absorbing materials can be clearly differentiated. This indicates that the approach can be used as a tool in imaging biologically relevant objects. Our plan is to offer this technique as one of the methods accessible to general user at the new tomography beamline (see poster by M. Stampanoni).

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Analyzer Based Imaging of Slowly Varying Phase Objects

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Two phase sensitive imaging methods are widely used in synchrotron X-ray optics: propagation based imaging (Fresnel diffraction or in-line holography) and analyzer based imaging (diffraction enhanced imaging). They are examples of space-invariant imaging systems. Transfer functions relating the Fourier transform $\tilde{I}(f)$ of the image intensity to the Fourier transform of a slowly varying phase or a weak amplitude modulation can be associated to such systems, as described in [1,2]. Usually, these transfer functions refer to the ideal case of a perfectly coherent incident beam.

In the analyzer based case, we have calculated these transfer functions which depend, of course, on the angular setting of the crystal-analyzer, taking into account the actual structure of the incident beam coming out of the monochromator (particularly the wavelength spread). We shall discuss the validity conditions of this transfer function approach. It turns out that the phase-transfer function (PTF) is given by a very simple expression, in terms of the rocking-curve function of the crystal-analyzer, if the normalized spatial frequency is small in comparison to the width of the rocking-curve. We shall compare the PTF approach to the geometrical-optics approach generally used in analyzer based imaging [3].

We shall discuss the advantages and drawbacks of analyzer based imaging with respect to the propagation mode [4]. DEI may have a better sensitivity to the low frequencies of the phase modulation, because its PTF is then proportional to the frequency f, whereas the PTF of the propagation mode is then proportional to f^2. In contrast to the propagation mode, there are no zeros (others than for $f = 0$) in the PTF of DEI, but the transfer of higher spatial frequencies is limited by the interaction with the crystal.

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Coherence Measurements using a Partial Talbot Effect

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In classical optics, the Talbot effect is the self-imaging, under perfectly coherent illumination of wavelength λ of a grating of period a, at distances multiples of $D_R=a^2/\lambda$. Previous work [1], with a hard synchrotron x-rays beam of very good coherence, from the ID19 beamline of the ESRF, showed that the finite size of the source can be measured from small deviations of the Talbot effect, due to the non perfect coherence. This measurement was based on the deviation from unity of the ratio of the Fourier component of order 1of the intensity recorded at two distances D_0 and D_0+D_R , with a 1-dimensional phase grating

We present here measurements performed in lower coherence conditions, on the BM5 beamline of the ESRF, with phase or mixed (amplitude and phase) 2-dimensional gratings, in order to allow simultaneous measurements in the horizontal and vertical directions. With a mixed grating of period a, it is possible to use a pair of images at distances D_0=0 and D_R and the degree of coherence $\gamma(a)$ between two points in the grating plane, a distance a apart, can be measured directly.

During the experiments, we realized that a Fourier intensity component $I(D;m)$ of order m is, in the case of perfect coherence, a periodic function of the distance D , with a period $D_{Rm}= a^2/\lambda m$, therefore smaller than D_R for m>1; this is an important advantage for the experiments. From the theoretical point of view, we consider this property as a "partial Talbot effect". Furthermore, measuring the derivative of $I(D;m)$ with respect to D allows to obviate the lack of contrast at $D=0$, in the case of a phase grating. We can obtain a sampling of the degree of coherence $\gamma(x)$ for $x=pa$, for some values (in our experiments p=1,2,3) of the integer p.
These new features are confirmed by our present experiments. They increase significantly the potentialities of the Talbot method for coherence measurements [2].

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Can the Transport of Intensity Equation and the Transfer Function Approaches be Reconciled?

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Propagation based phase contrast imaging extends tremendously the possibilities of X-ray transmission imaging. Phase retrieval is a crucial step in this context for quantitative and for three-dimensional imaging. In the Fresnel diffraction regime, two types of approaches are commonly used: the transport of intensity equation (TIE) which is valid in the limit of 'small distances' [1,2] and the transfer function approach [3,4] which is valid for objects with a 'slowly-varying phase'.

TIE leads to a unique solution from a theoretical point of view in most cases. However, due to the small distance assumption, the object is not imaged under optimal conditions in terms of contrast and/or the spatial resolution is spoiled in the phase retrieval step. It is therefore important to consider approaches that are valid at a finite distance from the object. Whereas iterative approaches were proposed in this context [5], faster and more direct methods similar to the transfer function approach are crucial especially for tomography.

In the rather theoretical case of a pure phase object, the slowly-varying phase approach (SVP; often called weak phase approach) can be considered as an extension of TIE, i.e. the SVP solution converges to the TIE solution in the limit of small distances. However, in the case of a mixed object, the familiar formula for an object with a weak amplitude and a slowly-varying phase modulations

$$\tilde{I}(f) = \delta(f) - 2\cos(\lambda D f^2)\tilde{B}(f) + 2\sin(\lambda D f^2)\tilde{\phi}(f) \tag{1}$$

does not coincide with TIE for a small defocusing distance *D*. Indeed, we then get from (1), in direct space, $I(x) = I_0(x)[1-(\lambda D/2\pi)\phi''(x)]$, whereas the TIE is $I(x) = I_0(x) - (\lambda D/2\pi)[I_0(x)\phi''(x) + I_0'(x)\phi'(x)]$.

Our purpose is to generalise (1) to arbitrary amplitude modulations in order to eliminate this disagreement with TIE. Different solutions are proposed some of which are close to the formula obtained by Wu and Liu [6]. We have performed numerical simulations in order to test the different approaches, for varying magnitudes of the phase and amplitude modulations. Finally, this new approach with broader validity is applied to experimental data on non-trivial objects.

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Phasing of X-Ray Diffraction from Objects with Complex Densities

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Nanometer scale objects illuminated with x-ray beams that are both temporally and spatially coherent give rise to continuous diffraction patterns containing a wealth of information. Not only the shape of the illuminated object can be garnered but also characteristics of it's internal structure[1].

When the object of interest is crystalline, a coherent diffraction pattern can be recorded from around each Bragg peak of the crystal[2]. If this diffraction pattern lacks local centrosymmetry, the object must be reconstructed as a complex density function; the imaginary part of the density can then be viewed as due to internal strains. An additional complication that often arises during the measurement is an imperfect alignment of the diffractometer with a given Bragg peak. Understanding the impact any misalignment will have on the subsequent phase retrieval, and the resulting image, is vitally important in the interpretation of the result. Examples of obviously misaligned diffraction patterns and the inversion of those patterns using HIO-type methods will be discussed.

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Modelling and Comparison of Phase-Retrieval Algorithms for Hard X-Ray Imaging

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Phase imaging for the hard x-ray regime was intensely studied and developed during the past decade. The simplest experimental implementation is based on propagation of an x-ray wavefield in free space but requires a partially coherent illumination. In this case, the phase modulation can be retrieved from different object-detector distances which, in combination with tomography, allows one to reconstruct the spatial distribution of the complex-valued refractive index.
Based on the Born approximation, different forward models for propagation based hard x-ray phase-contrast imaging are introduced and according reconstruction algorithms are proposed [1]. A reconstruction scheme which is based on a linearization in the scattered wavefield will be discussed and will be compared with earlier results [2]. An assessment of the algorithms with experimental data will be presented.

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Phase Determination by Means of Three- and Four-Photon Correlation Measurements

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We propose that the phase problem may have a solution from three- and four-photon correlation measurements. The motivations for this suggestion are the experiments by Hanbury-Brown and Twiss (HBT) using intensity interferometry (2 detectors, measuring the two-photon correlation function) to measure the diameters of stars and by other workers in astrophysics who have extended the HBT method to get the phases of the mutual intensity function from multiphoton correlations. We note that the classical results obtained from studying intensity fluctuations of the electromagnetic field are the same as obtained from quantum mechanics by studying correlations of bosons. While the original HBT type experiments studied emitted particles, advances on the theoretical side have shown that similar experiments can be done using scattered radiation. The requirement to meet the conditions to measure phase is to have an incoherent radiation source. Synchrotron-radiation sources generally satisfy this requirement (electrons emit independently of each other) and even in free electron lasers the emissions may be effectively incoherent. In the X-ray region we propose that an incoherent X-ray source with a subpicosecond pulse length and a multi-element detector capable of making a readout between pulses should be sufficient to extract phase information for X-ray crystallography and possibly single particle experiments. We discuss the underlying principles and potential usefulness of this type of experiment.

Coherent X-Ray Diffraction Microscopy: Fundamental and Technical Limits

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In this paper we restate the basics of a coherent x-ray diffraction microscopy experiment with two goals in mind. The first is to allow the ultimate limits of the technique to be evaluated. We consider the limitation of imaging speed due to both currently-available and projected-future x-ray beam lines and the limitation of resolution due to radiation damage in the case of biological samples. The second goal is to determine the technical requirements for spatial and temporal coherence, x-ray wavelength, motion control and stability requirements etc that must be met if our future goals for resolution, statistical accuracy and 3D imaging speed are to be reached. This study is motivated in part by plans at the Advanced Light Source in Berkeley USA to build a new undulator and beam line for x-ray diffraction microscopy. In light of the fundamentals considered above we discuss the technical choices that we propose for the new diffraction microscopy facility.

Exit Wave Reconstruction in High-Resolution Electron Microscopy using the Transport of Intensity Equation

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Since many samples in electron microscopy are phase objects, as is the case for optical microscopy, obtaining phase information is critical to understanding their structures. Moreover, using a wave function reconstructed from phase information and an observed amplitude, we can correct electron-optical aberrations, like spherical aberration that has remained a persistent aberration of electron microscopes. Consequently, phase measurement is very important in electron microscopy.

About twenty years ago, Teague [1] derived an equation, which we call the Transport of Intensity Equation (TIE), for wave propagation in terms of a phase and intensity distributions, and showed that the phase distribution may be determined by measuring only the intensity distributions. The TIE was recently applied successfully in electron microscopy at medium resolution to observe static potential distributions of biological and non-biological samples [2] and to measure magnetic fields [3]. It has been very recently verified that the TIE will be applicable to even atomic resolution images [4]. In this report, we study practical problems in applying the TIE to high-resolution electron microscope images and then show some real results obtained from material science specimens.

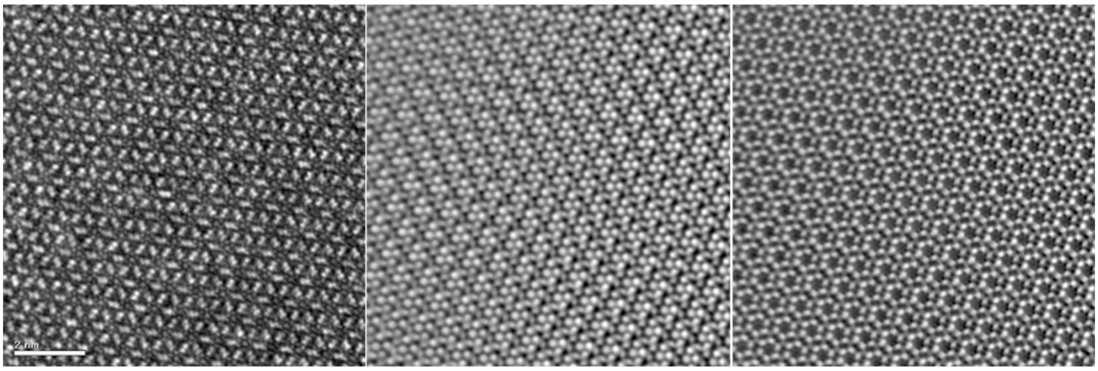

Figure 1: (left) the center image among a set of three images of Si_3N_4 used to calculate (center) the phase at this image plane, and (right) the phase of the wave function back-propagated to the plane close to the specimen exit surface, where an amplitude variation is minimum. Here, we have corrected spherical aberration and attenuation due to partial coherency. The last phase map bears remarkable resemblance to the results reported by Ziegler et al. [5], where the whole set of twenty images is used to retrieve the wave field. The scale bar is 2 nm.

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Towards Community Software for Diffraction Imaging

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Diffraction imaging is a coherence-based technique that could not exist without modern computers: iterative algorithms are used to phase the measured diffraction data and produce a real-space image, and computers are also integral to reliable collection of data throughout a tilt sequence.

Various groups in this area of research are in friendly competition with each other, in that we are all exploring different tricks in phase retrieval algorithms and processing procedures that are peculiar to our particular experiments and data. This is assuredly good, because competition stimulates creativity. At the same time, we are all using many of the same tools, including algorithms that involve hundreds or thousands of Fourier transforms, application of common constraints, and similar issues in 3D data assembly and visualization. As the size of our data sets increase beyond the capabilities of standard desktop computers, parallel computational approaches are beginning to be employed including message passing interface (MPI) enabled routines, yet there is relatively little standard code presently available for many of the common operations needed for diffraction imaging data processing.

Just as specialized experimental apparati can be constructed in part by bolting together common subunits such as motorized translation stages and CCD cameras, specialized software can be constructed from a framework that includes available subroutines. When common standards are employed, these subroutines can be written, tested, and documented by researchers worldwide, so that any individual can make greater progress by writing specialized code that exploits a common framework. This approach has been exploited to great advantage in the protein crystallography community, where programs required for tackling different steps of structure determination have been written to accept input and provide output to each other; numerous other examples exist, including the development of the Linux/GNU operating system and experimental control systems such as EPICS and SPEC.

Based on a workshop held in 2004 (http://xray1.physics.sunysb.edu/~jacobsen/workshop.html), we have begun to develop standards and available software modules for diffraction imaging (http://xray1.physics.sunysb.edu/~micros/diffmic/diffmic.pdf). This presentation outlines the general approaches chosen thus far, and seeks community input for future efforts.

Phase Retrieval Algorithm for Monotonically Changing System

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There have been enormous progresses in phase retrieval from coherent diffraction patterns using oversampling methods. Most of the work has been focused on stationary objects. In many cases, however, electron density of an real-valued object might increase or decrease monotonically as time or configuration changes. Depositions of thin films or electrochemical etchings are examples of such a process. Another case is related to partial reconstruction of a system using an aperture. One can adjust the size of post-aperture to a part of a whole image by using routine HIO algorithm. If one increases the size of post-aperture without moving its center, this system can be considered as a system with monotonically increasing electron density as well. We propose an iterative phase retrieval algorithm that is applicable to a wide range of system in which absolute value of electron density changes monotonically. The procedure builds on the classic HIO algorithm. The algorithm assumes that we have partial knowledge on an object. We'll describe the computational algorithm and experimental test results. We'll discuss possible applications as well as the limitations of this algorithm.

This work is supported by Korean Ministry of Science and Technology through the National Research Laboratory (NRL) program on sychrotron.

Dynamics of Block Copolymer Films by X-Ray Photon Correlation Spectroscopy

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Over the last years, tremendous research efforts have been dedicated to the study of potential applications of block copolymers in advanced technologies, such as information storage, drug delivery, photonic crystals, etc. The potential of block copolymers is based on their tendency to self-assemble into ordered nanostructures on a scale comparable to the chain dimensions. In this work, x-ray photon correlation spectroscopy (XPCS) was employed to study the dynamics of thin supported block copolymer films of poly(styrene)-b-poly(dimethylsiloxane) in grazing angle geometry [1]. We investigated the dynamics of the film material itself (for example, being characterized by micell diffusion) as well as the surface capillary wave dynamics, which are both accessible independently. The films were examined above the glass transition temperature at temperatures between 120ºC and 230ºC. Film thicknesses ranged from 40 to 300 nm. Our results will be discussed with the theory of overdamped thermal capillary waves on thin films. The lateral length scales examined lie between 600 and 6000 nm.

This work was supported by the International Cooperation Research Program of Ministry of Science and Technology of Korea (M6-0403-00-0079) and Interdisciplinary Program of Integrated Biotechnology of Sogang University.

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Effect of Fresnel Illumination on Oversampling Iteration Method

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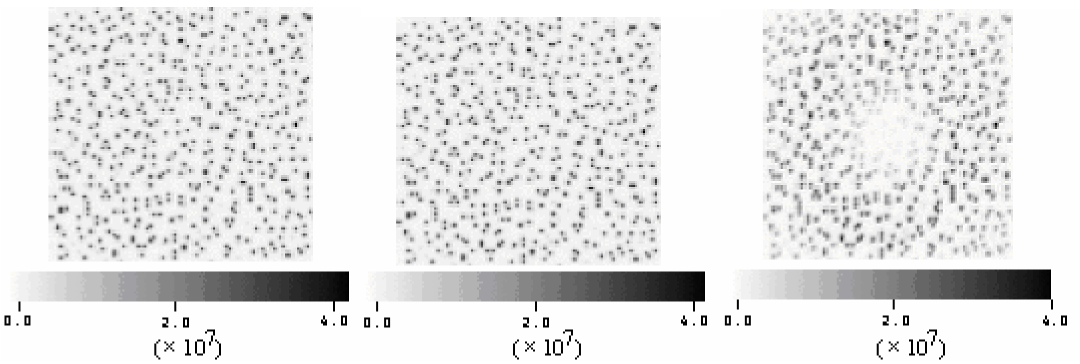
Coherent diffraction microscopy requires plane wave illumination on a specimen. In practice, a small pinhole or a focused beam is often used to reduce the illumination area, which unavoidably distorts the illumination wave. We quantitatively studied the effect of distorted illumination wave on phase retrieval by using computer simulations[1]. We have shown that various experimental conditions, such as the Fresnel number, pinhole size, alignment error and photon statistics, severely affect the quality of phase retrieval. As a specimen, silicon clusters in a random network structure was assumed, consisting of $2.821*10^{10}$ atoms. Figure.1 shows the assumed specimen and the results of the reconstruction for different Fresnel number (F_N) with the fixed pinhole radius of 25μm. The results will be of practical use for the design of coherent imaging experiments using the 3rd generation synchrotron radiation and future X-ray free electron lasers.



(a) (b) (c)

Figure 1: Electron density distribution of (a) a silicon specimen, of the reconstructed image for (b) F_N =1 and (c) F_N =10. The sample size and the pixel size are $4.4*4.4\mu m^2$ and 50 nm.

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X-Ray Tomographic Imaging of Crystal Structure at Atomic Level

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A two dimensional pattern of the directional absorption fine structure of white x-ray was recorded for GaP crystal using secondary electron signal. The measurement was performed by rotation of the sample relative to the incident white x-ray beam (mean x-ray energy E~40keV, energy spread ΔE~10keV). This directional structure of white x-ray absorption arises from coherent interaction between the incident beam and beams forwardly scattered on atoms inside the sample [1]. The main features of the recorded pattern, presented in Fig.1(a), are bands, which can be interpreted as a distorted projection of the real-space of the illuminated crystal. Through the application of a direct tomographic reconstruction algorithm, the electron density distribution within a unit cell of GaP could be determined (Fig 1(b)).

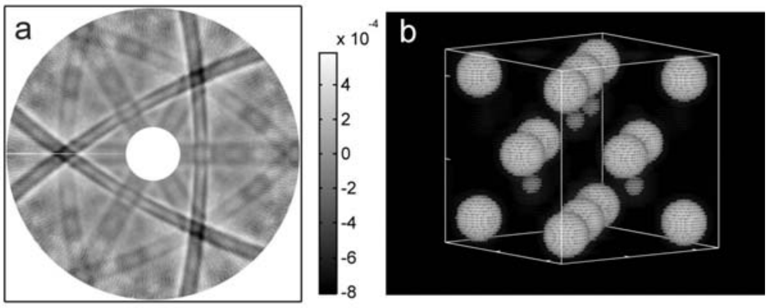

Figure 1: Directional fine structure of white x-ray absorption recorded for GaP crystal. (a) The pattern is shown as a stereographic projection. The colorbar refers to the background. (b) Electron density of GaP directly recovered from the measured data with a tomographic algorithm.

In spite of the small dynamical range of the directional signal, the presented proof-of-principle experiment was successfully performed using radiation from a bending magnet. The use of the wiggler radiation will increase the number of photons by several orders of magnitudes and will allow to perform white beam absorption experiments with detection of characteristic radiation. This will give a possibility to study diluted and low dimensional systems.

This work was supported by Volkswagen Foundation, Federal Republic of Germany. The access to synchrotron radiation was granted by the European Community (FP6, Integrating Activity on Synchroton and Free Electron Laser Science)

Reference

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Exact Determination of the Phase in Time-Resolved X-Ray Reflectometry

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We discuss a new and accurate method of solving the phase retrieval problem found in X-ray reflectometry. The approach is applicable to the characterization of thin films whose X-ray reflectivity can be measured *in-situ* during growth. Since the chemical composition of the incoming flux of particles generating the layer deposition may vary with time, the dielectric permeability of the film changes with depth. By measuring the reflectivity *in-situ* as a function of the time t, both the reflectivity R(t) and the derivative dR/dt are known. Hence, both the real Re[r(t)] and the imaginary Im[r(t)] parts of the amplitude reflectivity r(t) can be found exactly, in a very simple manner and following an explicit analytical form.

During the time interval necessary to measure the derivative dR/dt, we postulate that the composition of the film material located underneath remains unchanged. This is obviously valid at certain stages of the growth and not valid at others, e.g., in case of implantation or diffusion of atoms when several materials are present. Because the method assumes proportionality between the polarizability ε-1 and the density of the material, it can be applied to X-ray or neutron reflectometry but cannot be used with visible light. In addition, we demonstrate that having access to the temporal dependence of the reflectivity R(t) at a fixed grazing angle θ, it is possible to infer the depth-distribution of the dielectric constant $\varepsilon(z)$.

First experiments on the exact phase retrieval are discussed. The measurements were performed at the beamline BM5 of the ESRF. A special vacuum chamber intended for the sputter deposition of materials was installed at the beamline allowing to measure *in-situ* and in real-time both reflectance and scattering from a growing film [1]. A comparison of two methods of reconstruction of the dielectric constant profile is presented. The first one applies the exact phase retrieval method on the temporal dependence of the reflectivity R(t) measured at a fixed grazing angle θ of the probe beam. The second one solves the inverse problem of reflectometry, as described in [2], using angular-dependent reflectivity data $R(\theta)$ measured at fixed film thickness (after deposition). A good agreement between the two methods was obtained.

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Coherent X-Ray Diffraction and Charge Density Wave Dislocations

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Diffraction patterns obtained from a coherent X-ray beam are sensitive to any phase field deformations of the medium. In hard condensed matter, information on dislocations embedded in the bulk can thus be obtained from coherent hard X-rays. Our study of a Charge Density Wave (CDW) system provides an illustration of this phenomenon.
The blue bronze ($K_{0.3}MoO_3$) is a model CDW system which undergoes a electronic density modulation at twice the Fermi vector $2k_F$ and an incommensurate lattice distortion. As any crystal, this electronic crystal can exhibit intrinsic defects like dislocations. Detailed knowledge of these dislocations is essential to understand the CDW dynamics[1] or the narrow band noise generated by the CDW sliding[2]. From an experimental point of view, no direct observation of such defects has been measured so far, even though indirect evidence of CDW dislocations close to contacts has been reported by classical x-ray diffraction[3].

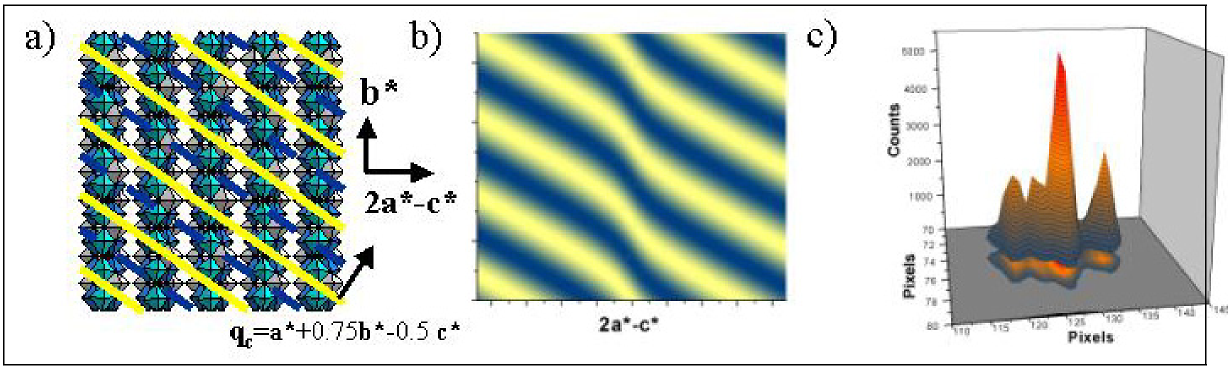

Figure 1: a) Schematic representation of the perfect CDW phase fronts in the Blue Bronze. b) Sections of the CDW screw-like dislocation c).Typical diffraction profile of the satellite reflection **Qs.**

In Fig.1c is displayed a typical diffraction pattern of the incommensurate satellite reflection $\mathbf{Q_s}$=(6,-0.252,-3.5), associated to the CDW, obtained at the ID20 beamline at the ESRF, under coherent illumination. For some beam positions on the sample, regular fringes appear while no fringe has ever been observed on the (6 0 -3) fundamental Bragg reflection. From elastic theory and knowing the experimental elastic constants, we show that this diffraction pattern is consistent with a single screw dislocation with the Burgers vector along the CDW modulation and the screw axis along **b*** (Fig.1b) [4]. Note that this screw-like dislocation does not lead to any compression or dilatation of the CDW along the **b*** direction which would be expensive in terms of Coulomb energy.

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A Method to Reconstruct the 3D Non-crystalline Sample from the X-Ray Diffraction Intensities Only

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The image reconstruction of non-crystalline objects from the X-ray diffraction oversampling phasing method has been carried out by Miao et.al [1,2]. However, the problem of the missing near-forward data caused by the beam-stopper blocking makes the reconstruction by using only the diffraction intensities very difficult. Recently a modified hybrid input-output (MHIO) algorithm was proposed to resolve this problem by demonstrating a few examples of applying to some 2D samples [3]. By borrowing a concept of isomorphous replacement in X-ray crystallography, we propose a different method that resolves this problem with a much larger loss of near-forward data allowed. The results for several 2D and 3D samples will be presented and compared with other methods.

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Monolithic Fresnel Bi-Mirror for Hard X-Rays

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We report about recent experiments with different types of monolithic X-ray interferometers. We used them to measure the degree of spatial coherence of hard X-rays in the energy interval of 5 keV to 50 keV at synchrotron beamlines of BESSY, ESRF and SLS.
A modified Young's two slit experiment for hard X-rays is realized using a '*monolithic Fresnel bi-mirror*' operating in total external reflection on a polished substrate. Compared to the conventional double-pinhole set-up the 'monolithic Fresnel bi-mirror' is simply to build and easy to align. The effective "slit size" and the "slit separation" can be modified by changing the incidence angle of the X-rays. The effective slit width was a few micrometer and their separation was up to some ten micrometers. We could observe interference fringes with high contrast in the whole spectral range.
The experiments were done with monochromatic X-rays as well as with 'white beam'. The interference pattern of a monochromatic beam was recorded using a high resolution area detector. In the 'white beam' set-up an energy dispersive solid state detector was used for energy discrimination at each point of the observation plane. The experimental results obtained for different modifications of this set-up are discussed and compared to similar experiments measuring the spatial coherence of X-rays. The advantages and drawbacks of experiments in the monochromatic and energy dispersive experimental regime and some applications are discussed.

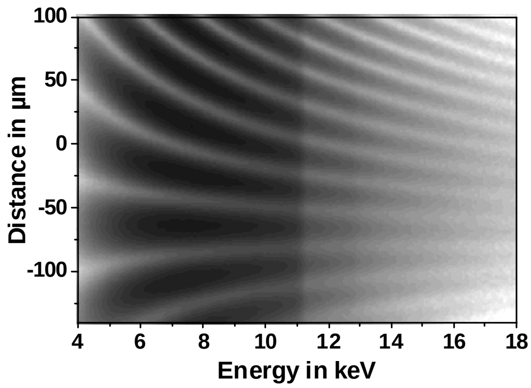

Figure 1: Typical diffraction pattern of a Fresnel bi-mirror equivalent to a pair of 0.4 µm slits in 6 µm distance (energy interval 4 - 18 keV). In the range of ±100 µm from the optical axis, 120 energy spectra were recorded and displayed in a grey scale map. Each horizontal line represents an energy spectrum measured through a 5 µm pinhole (black is high intensity).

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Non-destructive Testing with Neutron Phase Contrast Imaging

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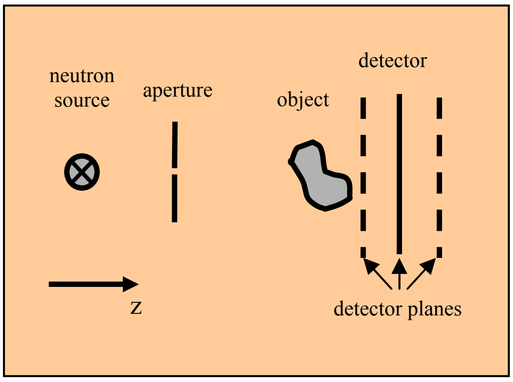
The basic setup for neutron phase contrast imaging is quite simple as shown in figure 1. To get the phase contrast in addition to the conventional absorption contrast, the utilized neutron beam has to have a sufficient lateral coherence and the detector plane must be in a suitable distance from the object. In cooperation with Dr. Eberhard Lehmann's group at the PSI in Villigen, a great variety of objects was investigated at the NEUTRA facility (SINQ), for instance prostheses and metal foams. Those measurements were very helpful in the final design of ANTARES, the new facility for neutron radiography and tomography at the FRM-II in Garching near Munich.



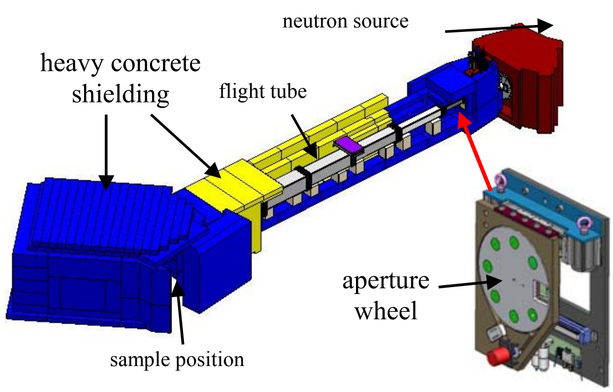


Figure 1: Schematics of a setup for neutron phase contrast imaging (left side) and the realization of such an experimental setup at the new neutron source FRM-II in Munich (right side).

The required lateral coherence for phase contrast imaging is achieved by an aperture and a sufficient distance between aperture and sample (~14m at ANTARES, see figure 1). At ANTARES, besides simple pinholes also coded apertures and slits are arranged in an aperture wheel, that allows to change aperture plates quickly.

Up to now, the phase contrast effect has mainly been used qualitatively as a contrast enhancing effect at edges and interfaces. The next consequential step is to do phase retrieval [1] to be able to use the real part of the refractive index $n=1-\delta-i\beta$ of a material to separate different materials in radiographies and tomographies [2]. Doing this procedure with neutrons and x-rays (a 320keV x-ray tube is installed next to the aperture wheel) will make it possible to separate materials with the help of four different values δ_x, δ_n, β_x, β_n.

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Characterization of Nanometric Grain Boundary Wetting Layers by Different X-Ray Imaging Approaches

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When liquid Ga is put into contact with Al polycrystals one can observe a very intriguing and spectacular phenomenon: within minutes the liquid metal will penetrate along the grain boundaries of the host material and lead to the formation of nanometric wetting layers. In-situ characterization of this penetration process by means of X-ray projection microscopy allows for simultaneous measurements of the layer thickness (via quantitative absorption and phase contrast measurements) and elasto-plastic grain deformations (via image correlation techniques)[1]. The layer thickness values determined from X-ray absorption measurements will be compared to results obtained from simulations of the Fresnel diffraction pattern (direct approach) and holographic phase reconstructions (inverse approach). The temporal evolution of the layer thickness and deformation values can be consistently interpreted in terms of a crack propagation process and have allowed to gain new insight into the mechanisms of liquid metal embrittlement.

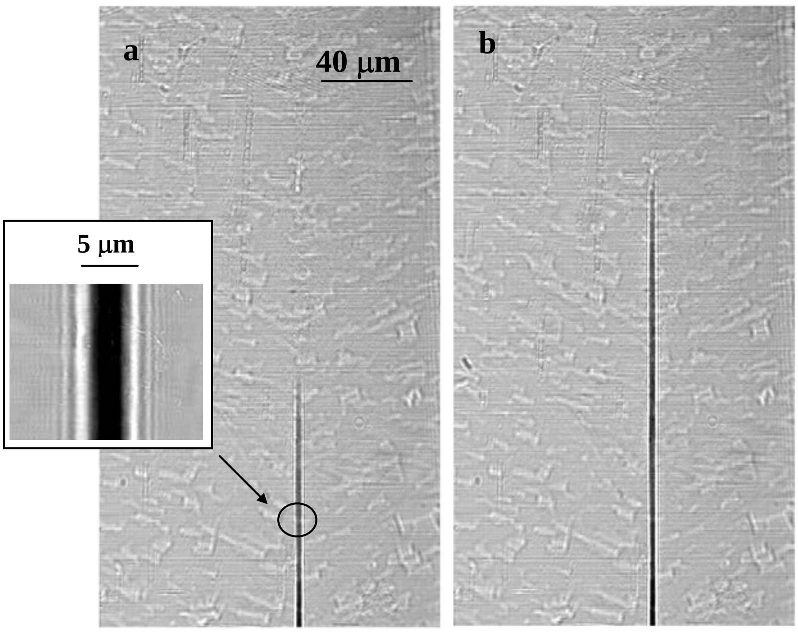

Figure 1: Coherent X-ray projection microscopy images showing grain boundary penetration of an Al bicrystal by liquid Ga.

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A Unified Evaluation of Iterative Projection Algorithms

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Imaging by phase retrival of a diffraction pattern is a promising new microscopy technique. This technique should enable for example to image objects at a resolution limited only by radiation damage (5-10 nm). The challenge of this imaging technique is transferred from being able to produce the best lens to developing the best algorithm to recover the lost phases from the measured diffraction intensities. Iterative projection algorithms for phase retrieval are tested on simple 'toy' models (arXiv:physics.optics/0404091). By studying the behavior of these algorithms, it is possible to predict the main components of future steps, increasing the speed of convergence and escape from local minima.

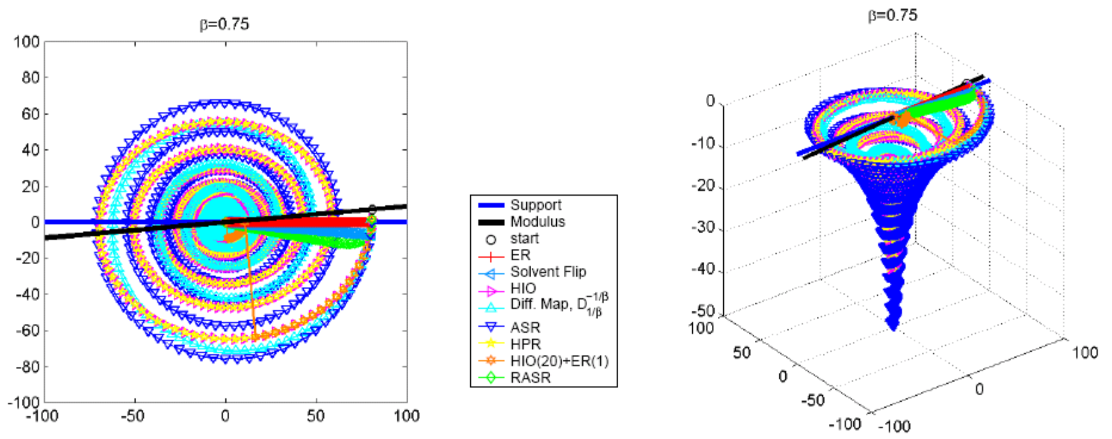

Figure 1: The basic features of the iterative projection algorithms can be understood by this simple model of two lines intersecting (1(a)). The aim is to find the intersection. The ER algorithm and the Solvent flipping algorithms converge in some gradient type fashion (the distance to the two sets never increases), with the solvent flip method being slightly faster when the angle between the two lines is small. HIO and variants move slightly in the direction where the gap between the two projections decreases, but at the same time in the direction of the gap, following a spiral path. When the two lines do not intersect (1(b), HIO and variants keep moving in the direction of the gap. ER, Solvent Flipping and RASR converge at (or close to) the local minimum.

Direct Phasing by Fourier Transform X-Ray Holography

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X-ray holography is a promising method for imaging biological and materials science specimens with intrinsic phase and absorption contrast at nanometer-scale resolution [1,2]. The spatial resolution in Fourier transform holography is limited only by the precision with which the reference wave is known. While in practice this is limited by the optic used to form the reference wave, the object wave phase is directly determined upon reconstruction of the hologram. Due to this solution to the phase problem at lower resolution and its compatibility with coherent diffraction methods, Fourier transform holography has been proposed to aid iterative phase retrieval in lensless coherent diffraction microscopy [3,4]. To investigate this approach we recorded Fourier transform holograms of lead micro- and nano-crystals with coherent 1-2 keV x-rays at the 2-ID-B undulator beamline at the Advanced Photon Source. A zone plate lens with a finest zone width of 50 nm formed the spherical reference wave. The third diffraction order of the zone plate was used for its potentially higher resolution. Hologram exposures were typically 10 s using a direct-detection CCD camera; series of 100-200 exposures were acquired to improve the hologram signal to background ratio. Hologram series required only a few seconds to reconstruct numerically on a PC-class computer. Fine features in the samples were resolved to ~50 nm in the reconstructed holograms. These results give impetus to development of atomic resolution x-ray microscopy using ultrafast coherent x-ray laser sources.

*Work at APS is supported by the U.S. Department of Energy, Office of Science, Basic Energy Sciences, under contract W-31-109-ENG-38.

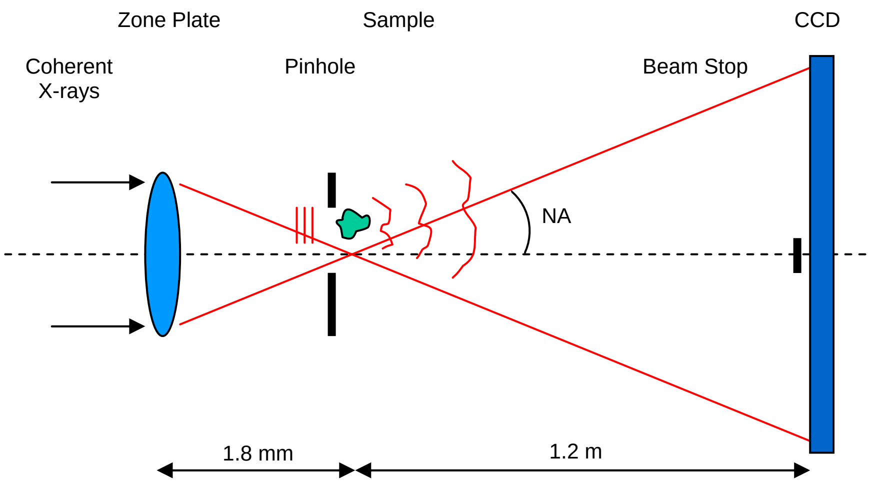

Figure 1: Schematic of the Fourier transform holography experiment (1.1 keV x-rays).

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Application of ALICE Diffractometer for Coherent Resonant Soft X-Ray Scattering

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In this contribution we present the possibility to use the ALICE scattering chamber for coherent resonant (magnetic) soft X-ray scattering (SPECKLE) [1,2] and spectro-holography, a new lensless imaging technique that has been developed recently [3,4]. In the past we have used coherent soft X-rays to obtain finger print like scattering patterns from magnetic stripe domains at remanence as well as in externally applied magnetic fields. Last year we extended our studies towards real space imaging via soft X-ray spectro-holography [4]. Furthermore we expect to obtain detailed information about the magnetic reversal behavior of such advanced magnetic systems. Such information could be obtained via a real space reversal movie while sweeping an externally applied magnetic field from negative to positive saturation.
The SPECKLE and spectro-holography patterns can be collected in a transmission and scattering geometry using the ALICE scattering chamber [5]. During the last year we have upgraded the ALICE setup with an additional set of pinholes (10, 20 and 50 μm) upstream of the sample for coherence filtering and a CCD camera for 2D detection of the coherent scattering (Fig. 1). Additionally the external magnetic field range accessible in the ALICE scattering chamber has been extended to 6.5 kOe via a new set of magnetic poles with a smaller gap (4 mm), so that it is now possible to completely saturate a large range of magnetic systems.

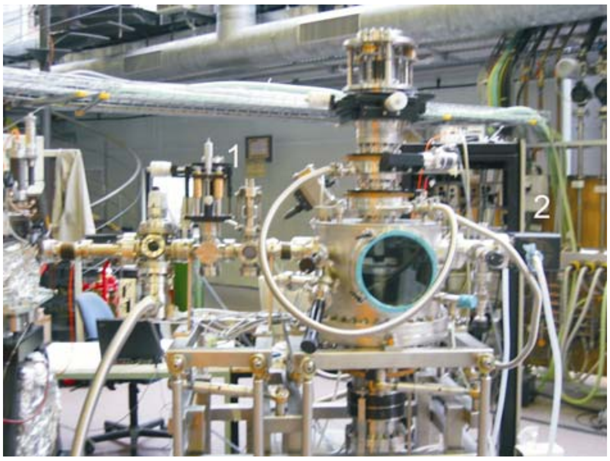

Figure 1: ALICE chamber with the additional set of pinholes (1) and the CCD camera (2).

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Two Dimensionally Confined Hard X-Ray Waveguides

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Two dimensionally confined hard x-ray waveguides provide a highly coherent, slightly divergent beam with a cross section of a few ten nm at the waveguide exit [1,2]. We have recently shown [2], that such two dimensionally confined x-ray waveguides exceed an integral flux of some 10^6 photons/sec in the the farfield, probably sufficient to perform experiments like scanning x-ray fluorescence, inline x-ray holography or micro diffraction experiments.

We present results of recently performed experiments with two dimensionally confined x-ray waveguides showing good agreement with numerical simulations of the waveguide properties.

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Fresnel Diffraction at a Circular Aperture-Determine the Illumination Function for Coherent Reflectifity

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As shown [1], coherent x-ray reflectivity pattern can be used to reconstruct the height-function of coherently illuminated sample area. One of the important free parameters of this procedure is the so called illumination function B(x), usually assumed to be a Gaussian. In some cases the approximation of B(x) by Fermi-integrals gives reasonable results [2]. In cases of reflectivity experiments this is an incorrect approximation.
In this talk we will present the white beam illumination function B(x, Energy) (Figure 1 a)) measured at the coherent reflectivity set up at the EDR-beamline at BESSY II [3]. It shows pronounced oscillations reflecting the spatial modulation of the coherently diffracted beam by the incident pinhole. We calculated the measured intensity with the formalism of Fresnel diffraction from a circular aperture (Figure 1 b)) using the formalism described in [4]. The results show a good agreement with the measurements. One advantage of this formalism is the possibility to calculate the phase of the incoming x-rays propagating through the pinhole. The results clearly show the importance of knowledge of the incident beam intensity distribution for static speckle experiments.

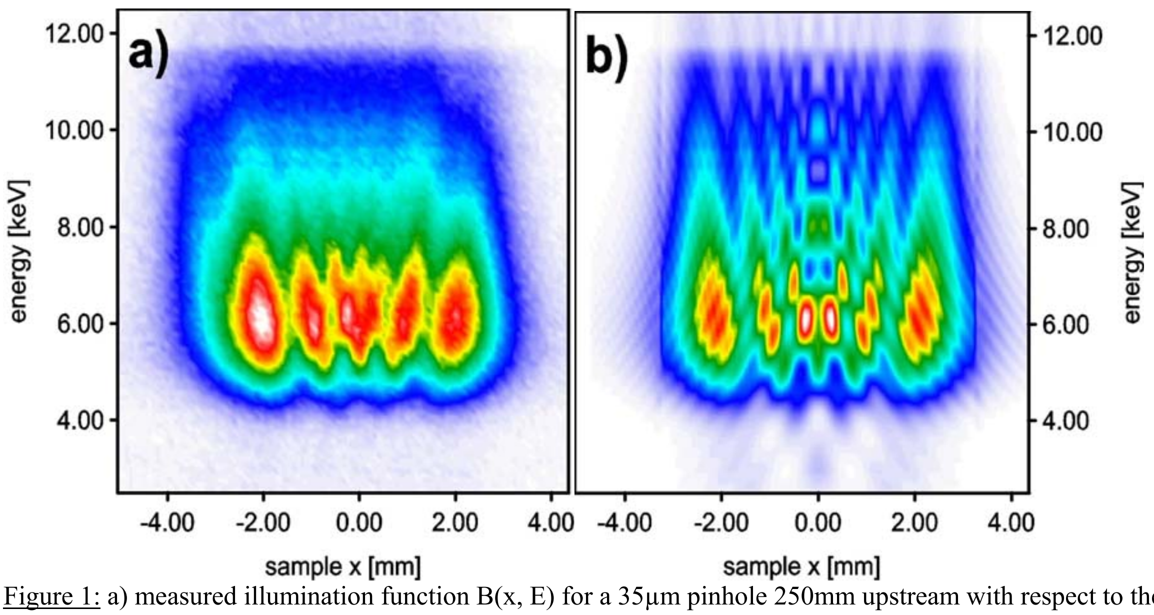

Figure 1: a) measured illumination function B(x, E) for a 35μm pinhole 250mm upstream with respect to the sample (simulated incident angel α_i = 0.3 degree); b) calculated illumination function B(x, E) for the given experimental condition using the Fresnel formalism.

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Characterization of Medium-Range Order in Noncrystalline Systems by Fluctuation X-Ray Microscopy

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Materials research has increasingly focused on developing a better understanding of the disordered state of matter. Many x-ray techniques exist to probe long- and short-range order in matter, in real space by imaging and in reciprocal space by diffraction and scattering. However, the characterization of medium-range order (MRO) is a long-standing problem that current x-ray techniques can not effectively probe.

We have developed fluctuation x-ray microscopy (FXM) based on fluctuation electron microscopy [1,2]. This novel approach offers quantitative insight into medium-range correlations in materials at nanometer and larger length scales. FXM examines spatially resolved fluctuations in the intensity of x-ray speckle patterns. Measuring the speckle variance as a function of scattering vector and illumination size produces a fluctuation map that reveals MRO and correlation lengths. FXM can explore MRO and subtle spatial structural changes in a wide range of disordered materials from soft condensed matter to nanowire arrays, semiconductor quantum dot arrays and magnetic materials.

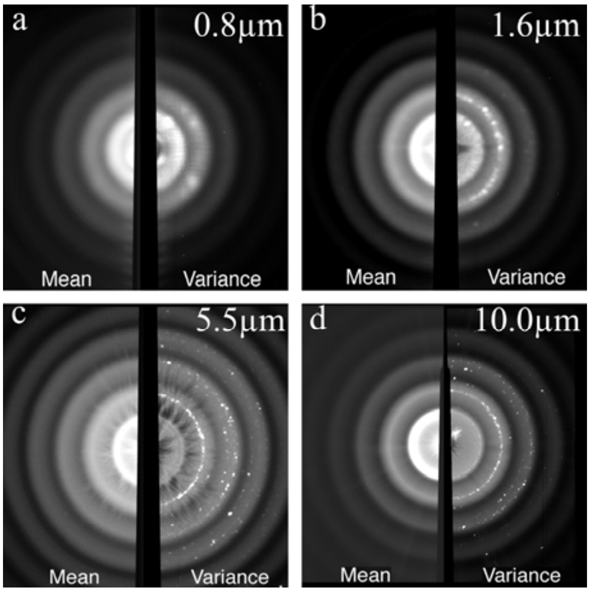

Figure 1: Mean and variance data from ~4000 speckle patterns from a thin film of latex spheres, using 1.83 keV radiation and differing illumination diameters as labelled.

FXM has been demonstrated at micron correlation length scales in studies of a model system comprising polystyrene latex spheres. The theory underlying FXM [3], the data analysis and the quantitative determination of MRO correlation lengths are discussed. Efforts to develop FXM to study MRO with correlation lengths of 50 nm are discussed.

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Practical Considerations for the Use of Structured Illumination in Coherent Diffractive Imaging

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Oversampling methods of inverting far-field x-ray diffraction patterns have received considerable attention recently as a result of some impressive experimental demonstrations [1]. However, the iterative approaches that have been adopted as a means of performing that inversion can be slow to converge. Additionally, stagnation points can make it difficult to assess when the optimal solution has been reached. We have shown that introducing a scheme of appropriately structured illumination greatly improves the time to convergence and reduces stagnation [2]. Using structured illumination can produce additional benefits. These include removing the need for *a priori* knowledge of the object support; allowing for objects that are larger than the beam (including periodic objects); and providing a unique solution for certain cases [3].

Figure 1: Example of a homometric pair and their identical plane wave diffraction pattern. Structured illumination methods consistently recovered the correct structure. Conventional coherent diffraction methods were unable to reliably retrieve the correct structure.

We have begun a series of experimental tests of the structured wavefield technique. Here, we describe some of the experimental limitations on the method. In particular, we consider the effect of imperfect knowledge of the illuminating beam on the quality of the reconstructed object. Different test objects are considered, including that most pathological of objects for these methods – the x-ray vortex.

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Coarsening Dynamics in Elastically Anisotropic Phase-Separating Alloys with XPCS

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In recent experiments [1] we have shown that X-ray photon correlation spectroscopy (XPCS) is a proper method to investigate the very slow dynamics of coarsening in phase-separating alloys at late stages of phase separation. In order to analyse temporal correlations in the XPCS data the fluctuation analysis technique was applied. Thereby, the so-called fluctuation exponent α is computed, which specifies the degree of correlation. Since the investigated systems in [1] are isotropic with spherical precipitates α was only dependent on the norm of the scattering vector **Q**. By comparing this dependence with Monte Carlo simulation results [2] it was possible to distinguish between different predominant coarsening mechanisms [1].
Here we present first results for Ni-Al-Mo alloys [3] that belong to the group of so-called nickel-based superalloys. By varying the Mo content different lattice constants of the matrix and, hence, different lattice misfits between the precipitates and the matrix can be realised, causing elastic anisotropy. Because of this anisotropy the precipitates are no longer spherically shaped, but are cuboidal or plate-like and aligned along the elastic soft directions ([001]) of the material. Thus, the fluctuation exponent α is expected to be additionally dependent on the direction of **Q**.

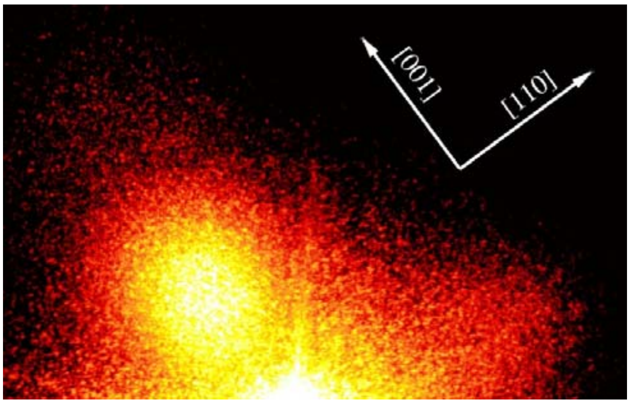

Figure 1: SAXS pattern of Ni-10at.%Al-5at.%Mo exhibiting pronounced speckle, logarithmic intensity scale.

XPCS measurements were performed at the ESRF beamline ID10A in small-angle X-ray scattering (SAXS) transmission geometry with four Ni-Al-Mo single crystals with different Mo content at temperatures up to 975°C. Figure 1 shows a typical SAXS pattern with the main crystallographic directions indicated. Due to the preferred alignment of the precipitates along [001] a strong intensity maximum appears in this direction.

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Shearing Interferometer for Quantifying the Coherence of Hard X-Ray Beams

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Coherence is the common denominator of many of the most exciting x-ray research techniques that exploit the radiation produced by third generation synchrotron sources. X-ray Photon Correlation Spectroscopy (XPCS), Coherent X-ray Diffraction (CXD), lensless imaging, or phase contrast radiography/tomography are only a few examples of recently developed techniques. While it is widely recognized that coherence-based experiments demand characterization tools to monitor the stability, shape, and size of the x-ray beam, the intrinsic coherence properties are equally important. Since coherence is readily modified by the beamline optics, this can have a dramatic impact on the quality of measured data.

With this contribution we report a quantitative measurement of the full transverse coherence function of the 14.4 keV x-ray radiation produced by an undulator at the Swiss Light Source. An x-ray grating interferometer consisting of a beam splitter phase grating and an analyzer amplitude grating has been used to measure the degree of coherence as a function of the beam separation out to 30 microns. Importantly, the technique provides a model-free and spatially resolved measurement of the complex coherence function and is not restricted to high resolution detectors and small fields of view. The spatial characterization of the wavefront has important applications in discovering localized defects in beamline optics.

cSAXS – The New Coherent Small Angle X-Ray Scattering Beamline at the Swiss Light Source

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With this contribution a review about the current status of the coherent Small Angle X-Ray scattering (cSAXS) beamline at the Swiss Light Source (SLS) will be given.
Based on the scientific case for this project, the beamline targets mainly three experimental x-ray techniques, namely: Small Angle X-Ray Scattering (SAXS), Coherent X-ray Diffraction Imaging (CXDI), and X-Ray Photon Correlation Spectroscopy (XPCS).

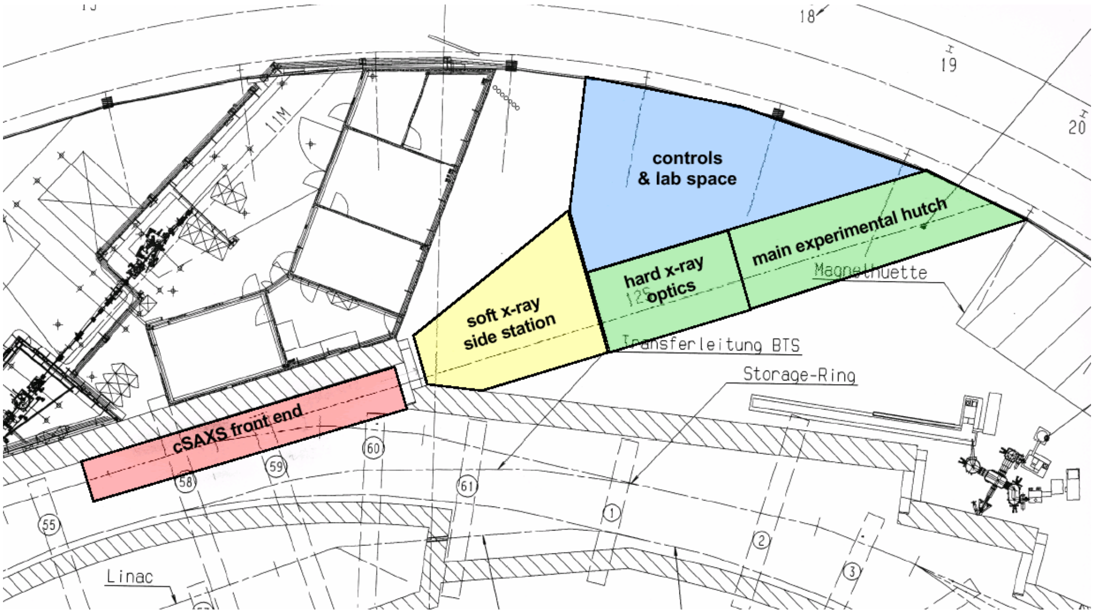

Figure 1: Layout of the coherent Small Angle X-Ray Scattering (cSAXS) beamline X12SA at the Swiss Light Source SLS.

Based on the requirements of the three main future activities, the conceptual design and layout of the beamline has been finalized. The general layout, the beamline specifications, and results of specific feasibility studies concerning the key components of the beamline will be presented. Finally, personnel issues and a preliminary schedule will be discussed.

Coherent Diffractive Imaging Algorithms: New Points of View

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In iterative phase retrieval methods, real-space constraints impose low-resolution information about the size and shape of the object to be reconstructed, and may involve the use of independent techniques such as microscopy or holography. Some additional high-resolution information about the composition of the target object may also be encoded by imposing constraints on the real and imaginary parts of the function that describes it. Iterative algorithms of this type exploit the interrelations

$$f(\vec{x}) = \hat{F}[F(\vec{u})] \quad (1)$$

$$F(\vec{u}) = \hat{F}^{-1}[f(\vec{x})] \quad (2)$$

$$I(\vec{u}) = F^{*}(\vec{u})F(\vec{u}) \quad (3)$$

$$g(\vec{x}) = \int f^{*}(\vec{r})\, f(\vec{r}+\vec{x})\, d\vec{r} \quad (4)$$

where $f(\vec{x})$ denotes the target function, $F(\vec{u})$ is its Fourier transform, $\hat{F}$ denotes the Fourier transform operator, $\hat{F}^{-1}$ is the corresponding inverse operator, $I(\vec{u})$ is the measured intensity in Fourier space, and $g(\vec{x})$ is the spatial autocorrelation of $f(\vec{x})$. The functions $g(\vec{x})$ and $I(\vec{u})$ are, like $f(\vec{x})$ and $F(\vec{u})$, related as a Fourier transform pair. Iterative schemes based on Eq (1) and Eq (2) with constraints imposed on $f(\vec{x})$ and $F(\vec{u})$ form the basis of the Gerchberg-Saxton algorithm and its subsequent elaborations. In this paper we explore the direct use of Eq. (4), a non-linear integral equation of Volterra type that may solved iteratively for $f(\vec{x})$. Alternatively, computational techniques borrowed from quantum chemistry may be adapted to form the basis of an efficient gradient-based scheme that determines $f(\vec{x})$ by minimizing the error in $g(\vec{x})$. The computational implementations of these two approaches is presented in this paper, using representative examples of a realistic dimension. The advantages imparted by illumination with phase curvature on these algorithms and the insights gained into the familiar problem of iterative stagnation will be discussed in the context of our target applications, which are nanoscale localized electromagnetic wavefields and biomolecular systems.

Displacive Transition Revisited by Coherent X-Ray Diffraction

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The perovskite oxide $SrTiO_3$ undergoes a prototypical example of structural phase transition, which stabilizes below T_c ~100-105 K an antiferrodistorsive modulation at the (1/2,1/2,1/2) reduced wave vector. However, this transition has been the first example where, in addition to the usual Lorentzian component in the critical x-ray scattering profiles, a sharp Lorentzian-squared component has been observed close to T_c[1]. This sharp component was later found to be quite general in the class of structural phase transitions[2], and believed to correspond to a surface phase transition (10 to 100 µm depth), exhibiting a different critical behavior from the bulk (a second length scale) but the same transition temperature.
We show that the use of Coherent X-ray Diffraction (CXD) allows one i) to separate the different critical behaviors ii) to give evidence of the static character of the second length scale fluctuations and iii) to confirm it takes place in the near surface close to defects.
Beyond this experiment, we show that CXD is a valuable new tool to study phase transitions and defects in the low temperature ordering[3].

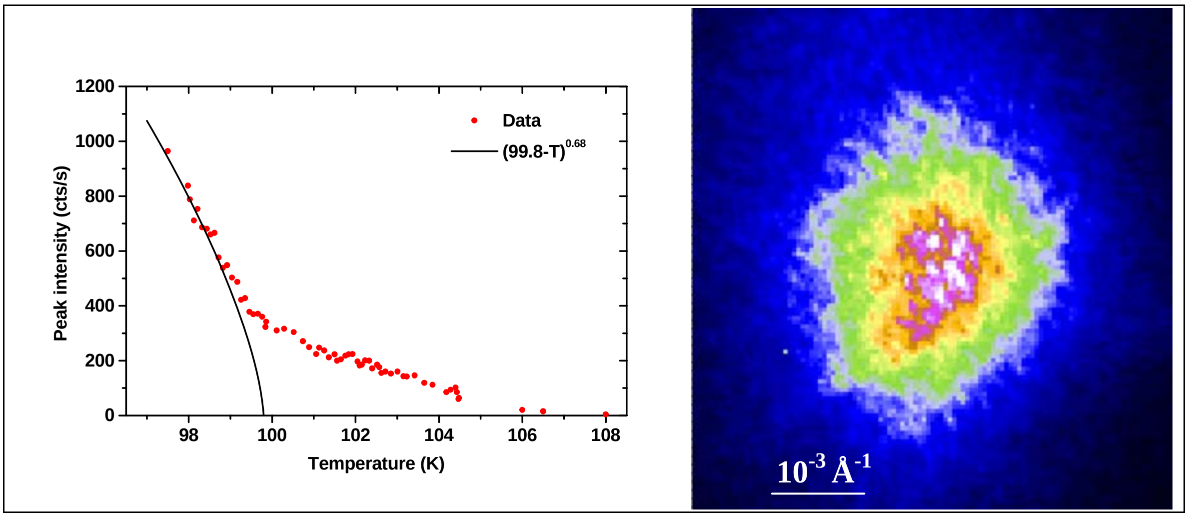

Figure 1: Left: Temperature dependence of the (3/2,1/2,1/2) superstructure reflection. The intensity above the phase transition is due to the sharp component. The transition temperatureas been obtained by fitting the data with a power Right: X-ray diffraction pattern of the sharp component (3/2,1/2,1/2) reflection at T=108 K. Speckles are clearly visible.

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Computational Simulations and Characterization of a CCD-Detector in Preparation of an Experiment with Coherent X-Rays

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Lensless imaging experiments with coherent X-ray radiation can be used to determine the structure of objects down to a nanometer length scale. Lithographically structured samples of a few micrometers size have been prepared and are to be exposed directly to a partially coherent X-ray beam in an upcoming experiment. A detector positioned in the Fraunhofer regime of this imaging system records the diffraction image of the sample. Using methods of phase retrieval, the shape and structure of the sample can be reconstructed from that image [1, 2].

In preparation of this experiment various computational simulations modeling the experimental setup were carried out. These simulations are based on the general formalism of wave propagation in the mathematical frame of the Fresnel approximation. Thus it is possible to calculate the complex amplitude of an X-ray beam in any plane perpendicular to the propagation path. Various objects like slits, pinholes, lenses and arbitrary apertures, e.g. the lithographically prepared samples, can be inserted in such a path. Furthermore, the computer program permits to model the more realistic situation of partially coherent illumination. These results help to determine the components of the setup that substantially influence the outcome of the experiment and give additionally an estimate of the absolute photon flux that can be expected on the detector. The latter parameter is highly important because the strong decay of the diffracted intensity around the forward direction of the beam limits the spatial resolution in this experiment.

The demands on the detector can therefore be formulated in terms of high dynamic range as well as single photon counting capabilities.

The results of the simulations together with the characterization of the CCD-detector are summarized.

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Coherent X-Ray Diffraction Imaging of Biological Specimens

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Coherent X-ray diffraction imaging was used to investigate nano-scale structural information of biological specimens such as cytoskeletal actin filaments and mineralized fish bone at different developmental stages. Reliable phase retrievals obtained solely from oversampled diffraction patterns [1] show successful application of this method to imaging of biological systems at a modest spatial resolution of ~ 30 nm. While there still are practical limitations to employing coherent X-ray diffraction to visualize biological specimens, we have demonstrated that optimal sample preparation protocols and experimental configurations can help to circumvent some of the difficulties.

Experiment details, image reconstruction procedures, and the potential for exploring biological systems with coherent X-ray diffraction imaging will be addressed.

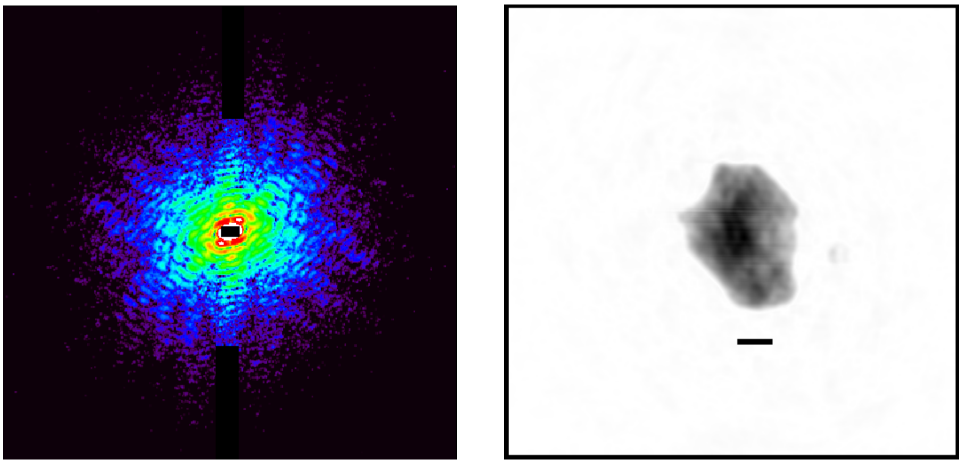

Figure 1: Coherent diffraction pattern from a fish bone particle at a low mineralization stage (left) and its reconstructed image (right). The scale bar corresponds to 500 nm.

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Sector 8: A Dedicated Undulator Beamline for Performing XPCS and GISAXS Studies

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Two experimental stations share the x-ray beam produced by an undulator at Sector 8 of the Advanced Photon Source, Argonne National Laboratory. The end station, 8-ID-I, is a dedicated instrument for performing XPCS in transmission and reflection geometry and at the side station, 8-ID-G, a new GISAXS setup has been implemented.
X-Ray Photon Correlation Spectroscopy (XPCS), the x-ray analog of dynamic light scattering, probes sample dynamics in a range of wave-vector and frequency space that is generally inaccessible to other light- and neutron-scattering techniques. XPCS experiments are performed by illuminating the sample with a sufficiently coherent beam so that the scattered x-rays produce a speckle pattern. Time auto-correlation of a speckle pattern yields the characteristic decay time(s) of a dynamic process of the sample. 8-ID-I has successfully performed measurements of dynamics on nanometer length scales of e.g. polymer melts [1] and aging gels [2]. The implementation of a dedicated sample reflectometer allowed it to extend the XPCS capabilities to include also reflection geometry. The new sample stage has been used to probe the dynamics of polymer films in the near-surface region [3].
Grazing-incidence small-angle scattering (GISAXS) is becoming an increasingly important structural-characterization technique for nanostructures and nanocomposites at surfaces and interfaces. GISAXS can be used *in situ* and in real time to monitor the formation of these. This capability makes it especially suitable for studying the kinetics of nano-assembly processes. We have built a dedicated GISAXS beamline in 8-ID-E station at APS. This beamline employs a large 4-cycle diffractometer equipped with high-precision X-Y-Z-stages and a high vacuum chamber capable of controlled in-situ heating and cooling processes. Both upstream and down stream beam paths are connected with sample chamber through bellows, thus making one vacuum body without x-ray windows. Thin film samples as well as liquid samples can be investigated. The sample to detector distance is up to 3m. The beam energy is usually fixed at 7.5 keV, however the third harmonic of the undulator (~22.5 keV) is also accessible. An overview of layout and capabilities will be presented.

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XTM – Beamline at the SLS: A Novel Facility for X-Ray Tomographic Microscopy and Real Time Coherent Radiology

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Synchrotron-based X-ray Tomographic Microscopy (XTM) is nowadays a powerful technique for the non-destructive, high-resolution investigation of a broad kind of materials. High-brilliance and high-coherence of modern third generation synchrotron radiation facilities allow micrometer and sub-micrometer, quantitative, three dimensional imaging within very short time and extend the traditional absorption imaging technique to edge-enhanced and pure phase-sensitive measurements.

The XTM station of the Swiss Light Source (SLS) is presently serving more than 20 users groups working in very different research areas. Due to the continuous increase of demand, SLS is currently constructing a beamline fully dedicated to tomography and coherent radiology.
The new beamline will be located at the X02DA port of the SLS and get photons from a 3.1 T superbend with a critical energy of 11.9 keV. This makes energies above 20 keV easily accessible. To guarantee the best beam quality (stability and homogeneity), the number of optical elements has been kept to a minimum. A Double Crystal Multilayer Monochromator (DCMM) covering an energy range from 8 to 45 keV with bandwidth of a few percent down to 10^{-4} will be installed [1]. The beamline can also be operated in white-beam mode, providing the ideal conditions for real-time coherent radiology [2].

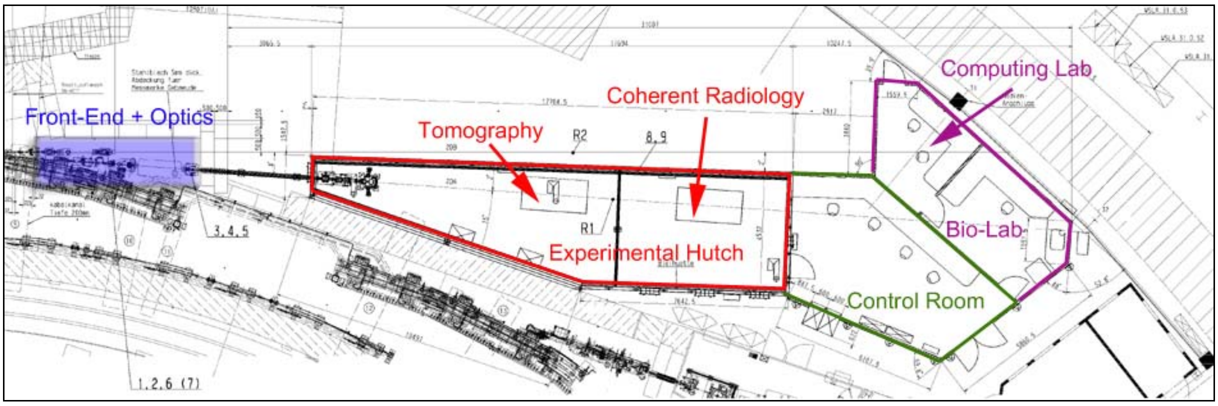

Figure 3: Layout of the XTM-beamline. There will be two, independent experimental stations for tomography and coherent radiology.

Available techniques will be traditional absorption tomography for small (1-5 mm) and large (up to 40 mm diameters) samples. Phase contrast imaging (differential phase stepping, free propagation and *Bronnikov* methods) will be also accessible to the general user.

The poster will present the scientific goals and the main technical aspects of the novel beamline.

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Probing Surface Dynamics of Poly-Propyleneglycol near the Glass Transition by 2D-XPCS

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By CCD-based photon correlation spectroscopy experiments with coherent X-rays performed under grazing incidence conditions surface dynamics of Poly-Propyleneglycol was studied near the glass transition ($T_G \approx 200$ K). The experiment was performed at the ID10A Beamline at the ESRF. In the high-viscosity regime the surface dynamics is governed by so-called overdamped capillary waves [1] which are characterized by a linear relation between the scattering vector $q_{||}$ and the relaxation rate. This prediction seems only valid in the low $q_{||}$ regime where the slope is inversely proportional to the viscosity (see Figure 1), because for larger $q_{||}$ a decrease of the slope is observed. Beyond the capillary wave model this can be explained by a theory of J. Jäckle [2] yielding a saturation of the relaxation rate for large $q_{||}$.

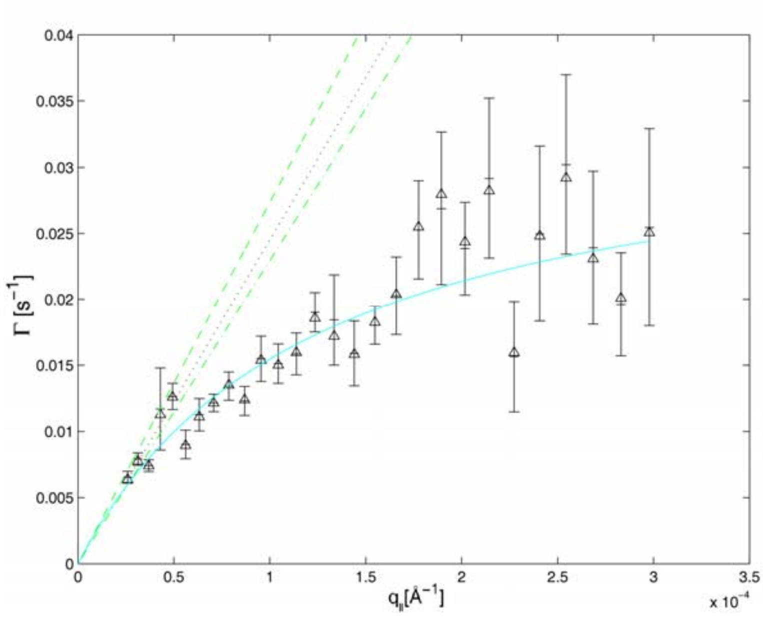

Figure 1: Relaxation rates determined by XPCS as a function of momentum transfer $q_{||}$ parallel to the surface (triangular symbols) at 214 K. The dashed lines indicate the expectation for overdamped capillary waves as determined by previous experiments. The solid line is a fit with the predicted dispersion relation in [2] by J. Jäckle.

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Investigation of the Dynamics of Colloidal Thin Films at the Nanometer Scale with Surface XPCS

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The surface dynamics at the nanometer scale of thin polymer films decorated with gold clusters acting as markers was investigated using x-ray photon correlation spectroscopy (XPCS) in grazing incidence geometry. While at the micrometer scale it is expected that fluctuating matter behaves according to the continuum classical hydrodynamic theory, strong departures from this theory are predicted at nanometer scales [1]. Kim et al. have already studied the dynamics of thin polymer films on micrometer scales with surface XPCS [2].
In our experiment, the scattering signal from polystyrene films with different molecular weights decorated with gold clusters was measured using a CCD setup as a function of time in a temperature range of 290 to 450 K. A low gold coverage is necessary to prevent an altering of the properties of the polymer films [3]. $q_{||}$ values from 0.1 to 1 nm^{-1} were measured simultaneously. As can be seen in Figure 1, images taken at low temperatures look more “grainy” than those at high temperatures. This can be explained by static speckles while the “fuzzy” aspect of the high temperature measurements is attributed to “dynamic speckles” moving faster than the total accumulation time.
Capillary waves motion at the polymer surface as well as surface diffusion of the nanoparticles have already been observed for length scales of about 10 nm, leading to viscosity values much larger than those already known for the bulk [4]. In this experiment we confirm the large surface viscosity and investigate the $q_{||}$ dependence of the relaxation time for different temperatures.

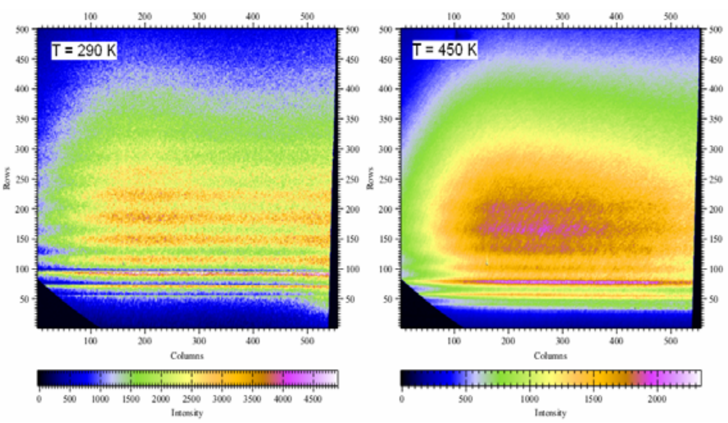

Figure 1: GISAXS pattern measured on a 100 nm thick, 220 kg/mol polystyrene film decorated with 2 nm gold at T = 290 K (left) and 450 K (right), respectively. Each picture results from an accumulation of 400 frames of 10 s. The oscillations along the exit angle direction (vertical axis) correspond to interference fringes of the polymer film, whereas the out-of-plane intensity maximum (horizontal, $q_{||}$) is due to the mean particle-particle distance of the gold clusters.

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Phase Retrieval and Tomographic Reconstruction of the Refractive Index Distribution from Diffraction Enhanced Imaging

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Diffraction enhanced imaging (DEI) as a phase contrast imaging technique was introduced in the late 1980ies and mid of 1990ies for neutrons [1] and x-rays [2] respectively. The instrumental set-up used for DEI corresponds to a double crystal diffractometer (DCD) i.e. a Bonse-Hart camera [3] as conventionally used for small angle scattering (SAS). At a perfect crystal monochromator only the fraction of an incident beam fulfilling the Bragg condition as described by the dynamical theory of diffraction is reflected. The same condition is valid for the perfect crystal analyser. Hence smallest beam deviations (down to 10^{-7} rad) due to diffraction, refraction or SAS can be resolved and measured. The phase difference at a sample edge causes a beam deviation and therefore a loss (or increase, depending on the analyser angle) of intensity in the beam behind the analyser. Hence an enhancement of contrast in a radiographic image recorded at this position is detected. If the sample geometry is well known the refractive index (and for neutrons the scattering length, which is not available from theory) of the penetrated material can be measured and calculated accurately from Snellius´ law. However, up to now DEI was mainly used as a radiography technique with a wide range of applications in materials science and medicine. In some other cases tomographic projections have been recorded and reconstructions were done, but the diffraction enhanced contrast was simply used straightforward as an intensity effect. Neither phase retrieval nor refractive index reconstruction have been taken into account so far. Of course the results are helpful to image additional structure details and to improve contrast compared to pure attenuation contrast. On the other hand these results lack a correct physical basis because the reconstruction is due to the attenuation law, which does not fit DEI. Therefore a mathematical algorithm involving phase considerations for the tomographic reconstruction of the sample structure i.e. the refractive index distribution from diffraction enhanced projection images will be introduced. This includes necessary treatment for DEI data in order to calculate the beam deviation angles from the intensity patterns and the adaptation of the Radon transformation with respect to beam deviation and phase shift for straightforward filtered back projection (FBP) algorithm reconstruction. Furthermore some examples of successful reconstructions from neutron DEI tomography will be presented.

Acknowledgement: This work is part of the BMBF project 03TR6TFH

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One-Dimensional Phase Problem of High-Resolution Fourier Transform X-Ray Spectroscopy

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A one-dimensional phase problem was discussed for high-resolution Fourier transform X-ray spectroscopy [1], where the phase of interferogram cannot be measured. A numerical iterative method [2] and an analytic logarithmic Hilbert transform method [3] were tested for recovery of the missing phase information from the modulus of interferogram. These methods were applied to measured data of the Si 14 6 0 back reflection and a calculated interferogram of an X-ray Fabry-Perot interferometer. The iterative method had ambiguity of reconstruction, however, the ambiguity was relatively small and might be acceptable. The logarithmic Hilbert transform method gave poorer reconstruction for the measured data due to lower signal-to-noise ratio, whereas it recovered the original spectrum from the calculated data without noise.

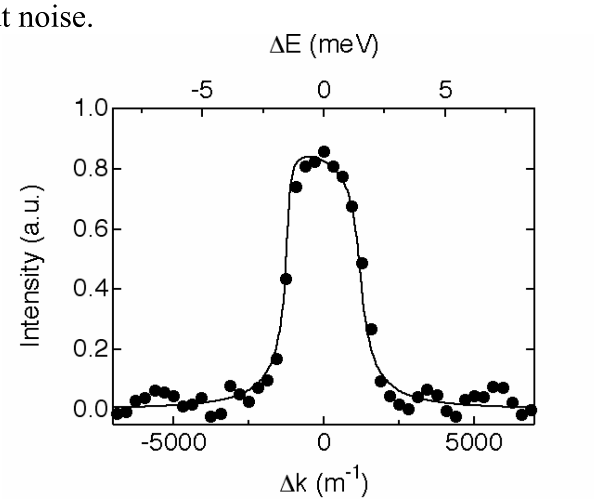

Figure 1: Typical reconstruction of the 14 6 0 back diffraction spectrum by the numerical iterative method (closed circle) and the theoretical one (solid line).

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Imaging of a Yeast Cell by Diffraction Microscopy: The Algorithmic Part

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High quality diffraction patterns of a 3 micron, freeze-dried, yeast cell have recently been produced at the Advanced Light Source, Lawrence Berkeley National Laboratory [1] with 750 eV X-rays. A total of 9 diffraction patterns, forming a tilt series of views separated by 1 degree, were phased successfully to at least 30 nm resolution. The consistency between the reconstructed exit waves assesses the success of the method used. This presentation gives the details of the algorithmic part of this X-ray Diffraction Microscopy experiment, using the difference map [2].

The reconstruction was particularly challenging for many reasons. First, wavefield simulations show that Born approximation is not valid because of the cell's large optical thickness. This brings many complications, among which our inhability to use simple constraints on the values of the reconstruction, such as positivity in the case of real-valued objects.

Second, missing data in the center of the diffraction pattern (due to the beam-block) introduces unconstrained degrees of freedom in the reconstructions. It will be shown how the impact of this missing data can be greatly reduced. The relation between the round shape of the cell and the acuteness of the missing data problem will also be explained.

Finally, averaging has been found to be useful in order to have unique solutions and to help evaluate the resolution of the reconstruction. The averaging method used in this reconstruction will be described.

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Complete Wavefield Recovery using Phase-Space Tomography and Its Applications in X-Ray Imaging

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Current imaging techniques are incapable of utilizing the information capacity of partially incoherent wavefields. For thermal sources, the second-order correlation function (or the mutual coherence function) gives all extractable information, of both the source and the object, contained in the beam. We will discuss our recent investigations of coherence properties and of complete reconstructions of one-dimensional x-ray beams produced from a third generation synchrotron using the phase-space tomography technique [1,2]. The results are far more complete and accurate compared to those obtained from alternative techniques. With the measured coherence of the beam, we present experimental demonstrations of the use of partially coherent diffraction data to recover fully coherent information. Recent results of our investigations of complete recovery of two-dimensional wavefields and future opportunities in optical imaging will also be discussed.

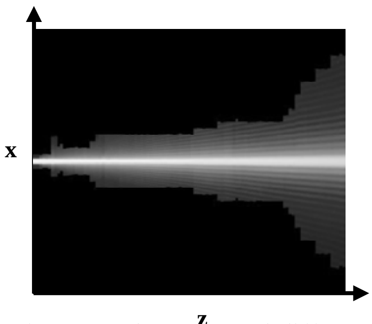

Figure 1: The measured diffracted intensity I(x,z) of a1.5 keV x-ray beam defined by a single-slit aperture of 25 μm width (logarithmic scale).

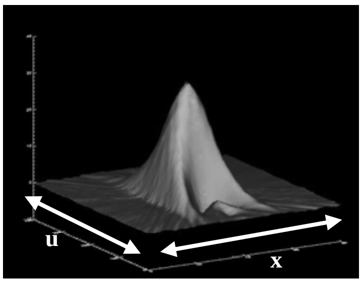

Figure 2: The phase-space density function B(x,u) reconstructed from the measured intensity distribution I(x,z) in Figure 1.

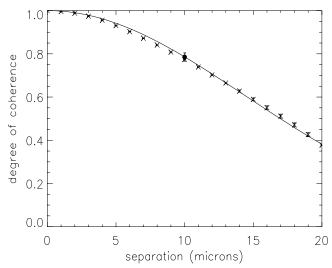

Figure 3: Deduced degree of coherence as a function of slit separation. The circle indicates the fringe visibility deduced from independent Young's fringe measurements.

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Ultra Small Angle Scattering vs Refraction – A Phase Based Problem

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To work with phase contrast one has to illuminate the object or the region of interest with a coherent wave front having a coherence width w_c. This quantity can either be calculated from the set up parameters or it must be measured by means of diffraction by defined gratings or slits. The interaction of the coherent wave with matter, however, is sometimes more difficult due to different interactions that cause overlapping phase contrasts in a signal (or image) [1], [2]. A Si crystal may have, in spite of well polishing, a surface roughness that can be displayed as a line profile as shown in Fig. 1. A simplified model of it is shown in Fig. 2. Illuminated with a plane wave, due to Babinet's theorem, one will always get USAS contrast if the surface is not plane. The specific phase based contribution to an amplitude (or intensity) depends on the ratio w_c/w_{Obj}, i.e. it determines whether USAS or refraction dominates the phase contrast. If the coherent wave front w_c is smaller than the effective size w_{Obj} of the object, refraction occurs, if $w_c > w_{Obj}$ USAS is present. To calculate the specific contributions one can simplify matters by assuming the surface to consists of e.g. a set of different triangles having a "width $w_{1,2,3,..n}$" and different depths as shown in Fig. 2.

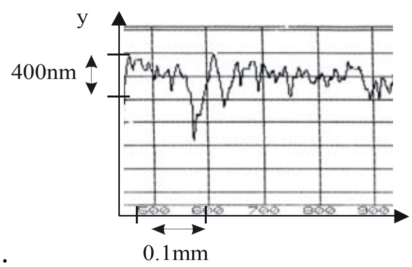

Figure 1: Line profile across a polished Si surface

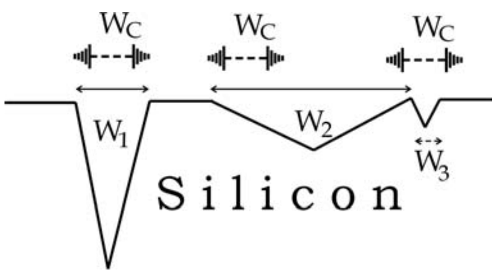

Figure 2: Coherence width w_c and wedge shaped surfaces $w_{1,2,3}$

For samples containing structures with sizes of $(100\mu m)^3$ that are investigated with highly coherent radiation (SR) or with special instruments, such as a double crystal diffractometer (DCD, Bonse-Hart type), w_c can be up to 100µm and more, so samples might show both, USAS and refraction phase contrast. It can be shown, that from scattering curves one can distinguish USAS from refraction and that in the case of "rough" Si surfaces the dominating phase based contrast is indeed USAS.

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Precise Estimation of Atom Positions from the Phase of a Reconstructed Electron Exit Wave

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In this study, exit wave reconstruction combined with statistical parameter estimation has been used to precisely determine the atom positions of the new compound $Bi_4Mn_{1/3}W_{2/3}O_8Cl$. Using exit wave reconstruction [1-2], the amplitude and phase of the complex electron wave function at the exit plane of the TEM (transmission electron microscopy) sample has been reconstructed. In a sense, exit wave reconstruction inverts the effect of lens aberrations which in general complicate direct interpretation of the images. In contrast to a conventional high resolution TEM image, the reconstructed exit wave is more closely related to the object structure. Moreover, since the light atom columns are revealed in the phase of the exit wave [3], it is possible to image oxygen atoms in the presence of heavier atoms. The phase can thus be used as a starting point for quantitative refinement of the atom positions. This has been done using statistical parameter estimation theory [4]. By adapting a parametric model to the experimental phase in the least-squares sense, it is possible to determine the atom positions. Using the so-called Cramér-Rao lower bound as a measure of the precision, it can be shown that sub-Ångstrom precision is within reach. In figure 1, the experimental phase is compared with the model evaluated at the estimated parameters [5].

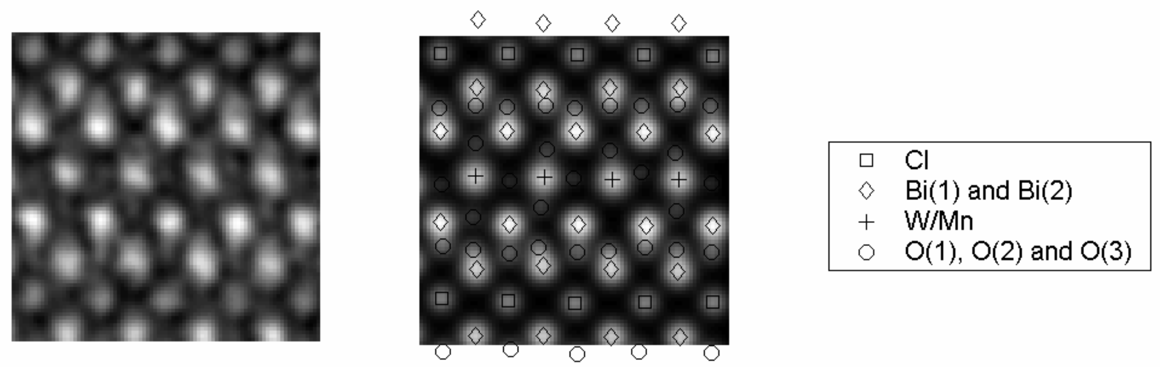

Figure 1: the experimental phase of the exit wave is shown at the left, whereas the model evaluated at the estimated parameters is shown at the right. The atom positions are indicated by the overlaying symbols.

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Combined Analyser-Based and Propagation-Based Phase-Contrast Imaging of Weak Objects

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Recently a new method combining analyser-based (AB) and propagation-based (PB) hard X-ray phase-contrast imaging has been investigated both theoretically [1] and experimentally [2]. A theoretical model of the combined imaging method was constructed under the assumption of slow variation of the individual transfer functions. In this work we present a complementary theoretical model using the assumption of a weak scatterer (analogous to the first Born approximation) [3,4]. We also show that, similarly to the case of PB imaging [5], the requirement for the phase variations in the transmitted beam to be small can be replaced by the less restrictive requirement for the phase to change slowly over a characteristic distance that is determined by the parameters of the crystal reflection (extinction length) and the free-space propagation (size of the first Fresnel zone). We derive an explicit expression for the combined AB/PB transfer function and present analytical and numerical examples solving related inverse imaging problems. Unlike the previously-obtained expressions [1], our results are not limited to the case of short propagation distances or low-resolution imaging. While our analytical expressions for the image intensity are valid in the case of a monochromatic plane incident wave, we also consider the effects of partial coherence due to the polychromaticity and finite size of the source. With the help of computer simulations using realistic experimental parameters we demonstrate that a small degree of polychromaticity of the source ($\Delta\lambda / \lambda \sim 10^{-3}$) is not an obstacle for observing the analyser-based phase-contrast image but, on the contrary, is necessary in order to observe it. We also show that the intensity distribution in the image obtained with a finite incoherent source in the combined AB/PB mode can be presented as a convolution of the image obtained with the polychromatic point source located on the optical axis with the rescaled intensity distribution function of the source.

The obtained analytical expression for the combined AB/PB transfer function can be used for quantitative analysis of the image contrast. It can also be used to solve the inverse problem of retrieval of the object-plane complex amplitude from collected image intensities as a first step in the reconstruction of the 3D spatial distribution of complex refractive index by means of computed tomography in the combined AB/PB regime.

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Imaging Nanocrystals with Curved Beam Diffraction

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The emerging field of coherent diffractive imaging with X-rays provides exciting prospects for structural determination on length scales down to the atomic. It is currently possible to image small crystals and nonperiodic objects composed of or decorated by high Z materials. An obvious strategy for imaging smaller objects is to place one or more focusing optics in the beam to increase the flux incident upon the sample. The presence of these optics also provides the opportunity to illuminate the sample with a curved X-ray beam. Since it has been shown that this curvature eliminates the so-called trivial ambiguities typically present in iterative fitting schema, the reconstruction of curved beam data is expected to proceed more quickly to the final solution. As the focus spots of Fresnel zone plates and Kirkpatrick-Baez mirrors are falling below 100nm, we are presented with the prospect of illuminating nanocrystals—of order 10^3 to 10^6 unit cells—with very bright, strongly curved beams. In this poster, we explore the feasibility of reconstructing a nanocrystal illuminated by such a beam. We will address considerations such as required flux, the validity of the projection approximation, and the effect of the magnitude of the curvature of the beam, as well as presenting the result of simulations reconstructing a small protein crystal.

Phase Sensitive Imaging with X-Rays and Neutrons - Parallels and Differences -

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Phase sensitive imaging is based on the phase-to-intensity variations by the propagation of a planar wave through a defined medium. The phase shift in the wave depends on the real decrement of the refractive index of the medium and the effective propagation path (medium thickness). The use of different types of radiation for imaging purposes changes the phase-contrast performance due to different interaction of the radiation with material. From a physical point of view x-rays and neutrons interact with matter in a completely different way – while the x-rays undergo an interaction with the electronic shell of the atoms, the neutrons interact mainly with the atomic core. This causes different absorption and refraction properties of the materials for these two kinds of radiation. On the other hand a neutron, independently of its mass, can be regarded like x-rays as a propagating wave with corresponding complex amplitude and wavelength. Therefore, similar to phase radiography and holotomography based on partially coherent synchrotron radiation, phase sensitive imaging with neutrons is also possible. The first phase contrast images with thermal neutrons were reported recently [1].
Furthermore the quantitative phase retrieval established for electrons and synchrotron light is now being extended to neutron phase contrast images. For holotomography the interferences due to the Fresnel-propagation could be used in a direct retrieval of the two-dimensional phase-maps using the weak-phase approach. The relatively low spatial resolution of neutron images (approx. 100 µm) as well as the cone-beam geometry prohibits the visibility of any interference fringes. Despite these obstacles refraction enhanced contrast is observed and can be used as input for phase retrieval methods, e.g. using the transport of intensity equation.

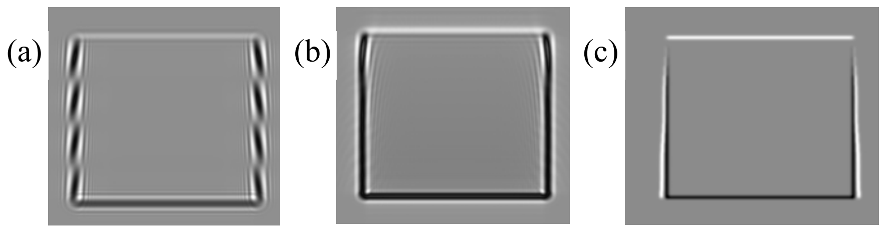

Figure 1: Simulation of radiographs of a Si wedge using the propagation method. (a) Wedge seen at 36 cm sample-to-detector distance by high-resolution synchrotron camera (2 keV bandwidth) and parallel beam geometry, (b) same picture with 20 keV bandwidth, (c) simulation of neutron phase contrast with low-resolution and cone-beam geometry with 4 m source-to-sample distance and 1 m sample-to-detector distance.

We present parallels and differences in phase contrast experiments at synchrotron sources compared to those at neutron facilities. Interpretation and analysis is done per simulations.

Reference
[1] - B. E. Allman, P. J. McMahon, K. A. Nugent, et al. , Nature, vol. 408, p. 158-159, 9 November 2000.

Optimisation of Phase Imaging with Hard X-Rays

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X-ray radiography and tomography are important tools in medicine as well as in life- and material-science. Not long ago, a new approach, called in-line holography based on the propagation method became possible via partially coherent synchrotron beams like the ones available at the European Synchrotron Radiation Facility ESRF. Theoretical and experimental work by Cloetens et al. has shown that quantitative retrieval of the optical phase, from a set of radiographs, taken at different sample-detector distances, is feasible. Mathematically speaking we are dealing with a direct method based on linearization in order to solve an "inverse" non-linear problem. In order to optimise the image-contrast for the numerical phase retrieval process, we have carried out calculations resulting in an optimised choice of value and number of the sample-detector distances as well as for the X-ray energy [3]. These results were verified by experiments on the "long" ESRF beamline ID19 (some results are depicted in figure 1).

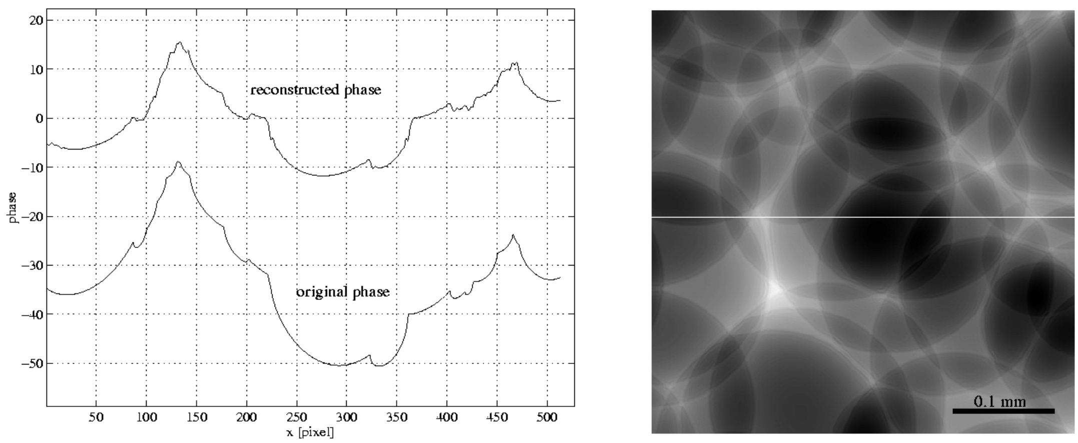

Figure 1: (Right) retrieved 2D phase map from four simulated radiographs (depicted objects correspond to a set of small polymer balls), (left) comparison between retrieved phase profile (white line in the 2D picture) and the original phase profile from the simulation data.

The present work focuses on the slowly-varying-phase (SVP) approach, more commonly known as the weak-phase approach. SVP gives a linear relationship between the Fourier transform of the optical phase-shift and the Fourier transform of the recorded intensity distribution on the radiographs thus permitting a straightforward phase retrieval [1,2]. The prior optimisation of SVP that we present here allows to improve the quality of the retrieved phase maps and can be extended to other linear methods.

References

[1] - J.-P. Guigay , Optik, Vol. 49, p. 121ff, 1977

[2] - P. Cloetens, W. Ludwig, J. Baruchel, D. Van Dyck, J. Van Landuyt, J. P. Guigay et M. Schlenker, Appl. Phys. Lett., Vol. 75, p. 2912-2914, 1999

[3] - S. Zabler, P. Cloetens, J.-P. Guigay, J. Baruchel, M. Schlenker, Rev. Sci. Instrum.

Hard-Sphere Colloids in the Fluid Phase Probed by X-Ray Photon Correlation Spectroscopy

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Colloids represent a large class of materials widely present in our every-day life (milk, paints, smokes, …). On the other hand they can be easily tailored to “simulate” model systems to study the role of well-define particle-particle interactions on the dynamic behavior of a condensed system. Here we present an X-ray study of colloidal suspensions made of spherical PMMA particles suspended in cis-decalin. We show measurements of the static and dynamic properties of the system at various volume concentrations up to the limit of the freezing concentration Φ=49% well beyond the limit in momentum transfer Q that can be accessed by Dynamic Light Scattering. The static structure functions were derived from Small-Angle X-ray Scattering measurements and the Q-dependence of the dynamics by the classical Photon Correlation Spectroscopy technique used with the highly intense coherent X-ray beams available at the ID10A beamline of the European Synchrotron Radiation Facility. The results are interpreted in the frame of the present theories for the hard-sphere potential.

International Workshop on

Phase Retrieval and Coherent Scattering

List of Participants

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Notes

Notes

Notes

	Tuesday June 14	Wednesday June 15	Thursday June 16	Friday June 17	Saturday June 18	
		Phase Retrieval Methods	X-ray Photon Correlation Spectroscopy	Coherent Diffraction Imaging		
08:00-08:30		**Breakfast**	**Breakfast**	**Breakfast**	**Breakfast**	*08:00-08:30*
08:30-09:00		Keith Nugent	Gerhard Grübel	Ian Robinson	*Ferry boat from Porquerolles Island to La Tour Fondue every half an hour from 8.30am to 11.30am*	*08:30-09:00*
09:00-09:30		James Fienup	Karl Ludwig	Henry Chapman		*09:00-09:30*
09:30-10:00						*09:30-10:00*
10:00-10:30		Dirk Van Dyck	Christian Gutt	Lorenz Stadler		*10:00-10:30*
10:30-11:00		**Coffee Break**	**Coffee Break**	**Coffee Break**		*10:30-11:00*
11:00-11:30		Timm Weitkamp	Frank Scheffold	Stefan Eisebitt		*11:00-11:30*
11:30-12:00		Andrei Bronnikov	Aymeric Robert	Chris Jacobsen		*11:30-12:00*
12:00-12:30		Qun Shen	Peter Falus	Ivan Vartaniants		*12:00-12:30*
12:30-13:00	*Ferry boat from La Tour Fondue to Porquerolles Island every half an hour from 12:30 to 18:30*	**Lunch**	**Lunch**	**Lunch**	**Lunch**	*12:30-13:00*
13:00-13:30						*13:00-13:30*
13:30-14:00		**FREE TIME**	**FREE TIME**	**FREE TIME**		*13:30-14:00*
14:00-14:30					*Ferry boat from Porquerolles Island to La Tour Fondue every hour from 14:00 to 19:00*	*14:00-14:30*
14:30-15:00						*14:30-15:00*
15:00-15:30						*15:00-15:30*
15:30-16:00						*15:30-16:00*
16:00-16:30		Stephen Wilkins	Mark Sutton	Yoshinori Nishino		*16:00-16:30*
16:30-17:00		Rajmund Mokso	Frédéric Livet	John Spence		*16:30-17:00*
17:00-17:30	Welcome and Registration	Helen Faulkner	Irakli Sikharulidze	Jian-Min Zuo		*17:00-17:30*
17:30-18:00		**Coffee Break**	**Coffee Break**	**Coffee Break**		*17:30-18:00*
18:00-18:30		Xizeng Wu	Laurence Lurio	Karine Chesnel		*18:00-18:30*
18:30-19:00		Discussion Rick Millane	Discussion Sunil Sinha	Discussion Janos Kirz		*18:30-19:00*
19:00-19:30	**Dinner**	**Dinner**	**Cocktail Workshop Banquet**	**Dinner**		*19:00-19:30*
19:30-20:00						*19:30-20:00*
20:00-20:30						*20:00-20:30*
20:30-21:00						*20:30-21:00*
21:00-21:30	Welcome and Registration	**Poster Session**		**Highlights and Summary John Spence**		*21:00-21:30*
21:30-22:00						*21:30-22:00*