Trace Element Micro-Analysis by Transmission X-ray Microtomography using Absorption Edges

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Transmission X-ray microtomography (micro-CT) has been used to obtain 2- or 3-dimensional distribution of the linear absorption coefficient which is the product of the mass absorption coefficient and the density. When the components of the object are two or more elements, it is difficult to determine the density distribution of the specific element from the data obtained with only one particular X-ray energy.

We propose a new method which utilize two x-ray energies bellow and above the absorption edge of the specific element. We suppose that the linear absorption coefficient of a material at a particular X-ray energy can be written as the sum of the linear absorption coefficient of the specific element and the other total linear absorption coefficient of all the other elements. The latter value is derived by assuming that it is proportional to the X-ray energies bellow and above the absorption edge. In this paper, we show how to determine the density distribution of Fe and Ni in the synthetic diamond.

The experiments were carried out at the BL3C2 of the Photon Factory. The X-ray energies used were 6.4, 7.47, and 8.4keV which are appropriate for the examination of Fe and Ni. Transmission X-ray images were recorded with a CCD camera which has a fluorescence screen and an expansion optical lens (×10). Microtomographs of the FeCl₂ solution (1.6~0.005 mol/l) and the mixture of FeCl₂ (0.005~0.0625 mol/l) and NiCl₂ (0.4 mol/l) solutions were obtained to evaluate the new method. The density mapping of Fe and Ni in the synthetic diamond was carried out by making use of this method. The results are shown in Fig. 1 and Fig. 2. The arrows A and B indicate some inclusions.



Fig. 1 Reconstructed X-ray micro-CT image of the synthetic diamond Fig. 2 The density distribution of Fe (a) and Ni (b)

Development of high speed microtomography system with high definition detector

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High speed micro-tomography system has been developed at BL20B2 and BL47XU in SPring-8. A high definition and high speed CCD camera is used as an image detector. The properties are shown in the table. Using the CCD camera in 2×2 binning mode the total scanning time for 1500 projections was reduced to 30 minutes or less while the scanning time of previous system was a few hours. This enables us to image soft materials and to do time resolved observations in three-dimension. They deform or change during the scanning.

At BL20B2, the image detector consisted of Beam Monitor 4 (f=105mm) and C4880-41S. The effective pixel size was 11.7 μ m x 11.7 μ m. The field of view was about 23.4 mm x 15.3 mm. The three-dimensional image of non-fixed dead animal is shown in figure. The scanning time was about 5 minutes therefore the total scanning was done before deformations of the animal.

At BL47XU, the image detector consisted of Beam Monitor 3 (x20) and C4880-41S. The effective pixel size was 0.47 μ m x 0.47 μ m. The field of view was about 0.94 mm x 0.61 mm.

The CT images of multilayer test patterns showed that the spatial resolution of the system achieved about $1\mu m$. This system has achieved a large field of view and high spatial resolution. It is very suitable for many areas of materials science.

At the conference the detail of the system and some experimental results are presented.

pixel pitch	5.9µm
format	4000×2624
full well	13,000 e ⁻
frame rate	1.7Hz
ADC bits	12bits
cooling temp.	-50°C

 Table. Properties of high definition CCD

 camera (C4880-41S, Hamamatsu.)



Figure. Three-dimensional image of bones of body of rabbit's pup taken with high speed CT scan (25keV, 5minutes). Width of the image is about 25mm.

High-Resolution X-Ray Imaging Microtomography with Fresnel Zone Plate Optics at SPring-8

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A high-resolution CT system using a Fresnel zone plate (FZP) objective has been developed at SPring-8 [1]. The system consists of an in-vacuum type undulator light source of SPring-8, a Si 111 double crystal monochromator cooled with liquid nitrogen, a beam diffuser, high precision sample stages, a Fresnel zone plate objective with the outermost zone width of 100 nm and a high spatial resolution x-ray imaging detector. Resolving power of the system was evaluated from the CT images. For the precise characterization of resolving power, an artificial concentric multilayer is used as a resolution test pattern. Schematic drawing of the sample is shown in Fig. 1(a). A varied period Cu/Al multilayer with line/space of 0.3 μ m - 0.1 μ m is deposited on an Al core by DC magnetron sputtering at AIST Kansai [2,3]. Figure 1(b) shows a CT image of the test pattern at an x-ray energy of 7.1 keV, and Fig. 1(c) shows a line profile between AB in the image. All of the layers up to line width of 0.3 μ m are clearly resolved. Some 3-dimensional image data obtained with this system will be presented.



Fig. 1. Cu/Al multilayer resolution test pattern. (a) schematic drawing, (b) CT image , (c) line profile between A-B in the CT image.

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CT reconstruction by diffraction correction in soft X-ray projection microscopy

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A computer tomographic microscopy (CT microscopy) has been studied by installing a rotary stage into a soft X-ray projection microscope at KEK-PF in Tsukuba. The aim is CT observation of a living tissue. Projection microscopy should eliminate fringes caused by the Fresnel's diffraction in contrast with imaging microscope^[1]. In this trial, the microscopy was evaluated from the viewpoint of the correction of the fringes on a projection image and the comparison between the resolution on the reconstructed CT image and that determined by the microscope's magnification and CCD pixel size. The experiment was accomplished at the beam line 11A or 12A at KEK-PF. The system is illustrated in Fig.1. The soft X-rays of wavelength 15 and 25 angstrom were used as a light source to utilize the penetration and absorption characteristics in the water-window region for a living tissue. The projection image was obtained at the fixed magnification of 50 by a back-illuminated X-ray CCD camera with the pixel pitch of 24.8µm. The diffraction fringes were observed on the projection image when the post-pinhole's aperture was smaller than 5µm^{\u039} as shown in Fig.2. The image of the absorption coefficient just behind the specimen was calculated by the iteration process^[2], which repeated the calculation between Fresnel and inverse Fresnel transformations under some restricted conditions such as illumination intensity. The projection images were obtained at the interval of 1 or 5 degrees in all directions. Each image was integrated its intensity for a few minutes to improve the signal-to-noise ratio. It took 3-4 hours to obtain a set of projection images. Figure 3 shows the reconstructed CT image. The sample was a taper glass capillary of the diameter of 5µm at the cusp. The figure is a cross- sectional image at the position of 30µm below the cusp. The outside

diameter and its thickness were calculated as $8\mu m$ and $1\mu m$, respectively. The pipe structure was clearly reconstructed with the resolution of $0.5\mu m$, while the blur and the distortion due to the eccentricity of the rotary stage should be considered.

For the future study, precise control of the sample stage is essential to realize the high-magnified and high-resolutive CT measurement.

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Fig. 1 Projection microscope with rotary stage.



Fig. 2 Fringes of Fresnel's Diffraction on a projection image (Glass capillary of $10\mu m^{\phi}$).



Fig. 3 Reconstructed CT image of the glass capillary.

Depth of Field Techniques for 3-Dimensional Imaging

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Abstract. Soft x-ray tomography has been used to obtain high-resolution 3-D biological images at the cellular and sub-cellular level. However, the small depth of field for a high-resolution micro zone plate limits the sample thickness that can be reconstructed. Since the lateral resolution is directly proportional to the outermost zone width and the depth of field is directly proportional to the outermost zone width squared, an improvement in lateral resolution corresponds to an even greater decrease in depth of field. For example, at a wavelength of 2.4 nm, the depth of field for a 40 nm zone plate is 2.7 microns, but for a 15 nm zone plate, the depth of field is 375 nm. Therefore, obtaining 3-D images of specimens significantly thicker than the depth of field at higher resolution would require a different data acquisition and reconstruction method. A method of combining optical sectioning and tomography is proposed as a means of obtaining high-resolution 3-D images of weakly absorbing, thick samples. Utilizing the small depths of field, optical sectioning, which is performed by obtaining a series of through focus images and then deconvolving the images, can be used to form a 3-D data set. Combining optical sectioning with tomography allows improvement in the axial resolution of the 3-D image. This method involves performing optical sectioning at each angle, forming a projection image from the deconvolved set of images, and then reconstructing them using a tomographic reconstruction algorithm. A zone plate and a two-layer test sample have been fabricated using ebeam lithography to test this technique, and deconvolution results will be presented.

X-ray Fluorescence Scanning Microscope and Micro-Tomography with a Zone Plate

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X-ray fluorescence micro-tomography is one of the promising applications of an x-ray fluorescence scanning microscope. For micro-tomography, a specimen must be rotated and raster scanned to an incident x-ray beam, which is difficult because a precision rotary stage is usually large and heavy. Then, a scanning microscope by scanning a microprobe instead of a specimen was developed.

Figure 1 shows the optical system. The x-ray beam-line BL20XU of SPring-8 was used. This beam-line has an x-ray undulator and a double-crystal monochromator cooled by liquid nitrogen. X-rays at 9.8 keV were focused by a zone plate. The diameter and the outermost zone width were 155 microns and 0.1 microns, respectively. The zone plate was placed about 250 m downstream from the undulator. This long distance enabled us to obtain the focused spot size of about 0.2 microns. The first order focus was used and the other order x-rays were eliminated by an order sorting aperture (OSA). The zone plate and the OSA were raster scanned synchronously to obtain a 2D image. Transmission and fluorescence x-rays were recorded by an ionization chamber and silicon drift detector (SDD) respectively.

A test sample of iron, zinc, and copper layers evaporated on a tungsten wire (Fig. 2(a)) was used for evaluation of the micro-tomography system. Figure 2(b) shows the section image of iron and copper x-ray fluorescence. The zinc layer (120 nm) could be distinguished, which can be seen as a gap of the two lines.



Fig. 1 Optical system of the microscope





Fig. 2 (a) Test sample, (b) X-ray fluorescence section image of iron and copper of the test sample.

Preliminary Results of Hard X-ray Phase-contrast Imaging

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Phase-contrast x-ray imaging is an emerging imaging technique that can be implemented at synchrotron radiation sources or by a microfocus x-ray source. The new technique is also developed and applied at Beijing Synchrotron Radiation Facility (BSRF). We present some results of X-ray phase-contrast imaging using synchrotron radiation at BSRF. The samples being imaged include gold fish, insects, SD rat and artificial objects made of plastic. Phase-contrast X-ray computed tomography is also explored.

Following figure is the pictures of gold fish with phase-contrast x-ray imaging. Soft tissues such as intestine, intestinal structures and spinal cord of the fish are seen clearly.



Fig. 1 phase-contrast x-ray imaging of gold fish

Refractive Contrast in X-ray Diffraction Enhanced Imaging

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X-ray Diffraction Enhanced Imaging (DEI) is a promising technique for the future medical application. However, the contrast mechanism of the DEI is still not clearly expressed and understood, although there have been many considerations related on the refractive contrast for DEI. In this paper, the deflecting angles of X-ray beams caused by refractive objects are deduced according to the refractive process at interface of different refractive indexes. The spacial resolution and the electron density resolution of the DEI method are discussed relating to the experimental parameters. Appearances of refractive contrast in the DEI method are discussed and experimentally confirmed. Vivid images of blood vessels in human liver sample are obtained by DEI technique using X-rays with different energies. The contributions of the refractive contrast in improving the image contrast are easily understood.

Contrast Enhance Imaging with Micro-focus X-ray Generator and CCD

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X-ray imaging of materials with low Z elements is difficult through conventional photo-absorption contrast method while much higher contrast can be achieved through phase-contrast imaging.

Previous experiments showed high contrast images by using moderate size (20 micron) X-ray generator (e.g., Wilkins, et al. 1996) or synchrotron orbital radiations (e.g., Kagoshima, et al. 1999).

In the current work, we plan to make use of very small (1 micron) micro-focus X-ray generator for the X-ray source and photon counting direct imaging X-ray CCDs for the imaging device. For the CCDs, we are developing large format (1 inch x 2 inch) 2 Mega-pixel chips. These CCDs are buttable and we can combine multiple chips to cover large spatial area. For example, we will be able to image 5 cm x 10 cm area with 8 chips.

Furthermore, we can improve the spatial resolution of the CCD better than the pixel size by referring to the split events. We are planning to develop a compact imaging system with a combination of our CCD and a micro-focus X-ray generator.

In the current poster, we will present the design and initial experimental results.

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Recent development on X-ray phase contrast imaging in China

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Abstract

The latest development in hard X-ray phase contrast image in China is presented. The main principle and methods of phase contrast imaging is introduced generally. Some important experimental results in diffraction-enhanced imaging and X-ray phase contrast tomography are given in the paper.

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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.



Fig.1 Silicon number distribution of (a) the model specimen, of the reconstructed image for (b) $F_N = 1$ and (c) $F_N = 10$. The sample size and the pixel size are $4.4 \times 4.4 \mu m^2$ and 50 nm.

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Digital Phase Difference Amplification in X-ray

Interferometer

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Early in 1965 U. Bonse and M. Hart suggested X-ray interferometry methods. In recent years, A. Momose and colleagues have been extending the techniques to the applications in biological and medical, and introduced the techniques into computed tomography as well. By taking a series of interferometry patterns at different rotational orientations of a sample, a three-dimensional picture of the refractive index of the sample can be reconstructed. With this method, they have studied the cancerous tissues of human breast, liver, and kidney and rabbit cancer lesions as well as a rat cerebellum using radiation at the Photon Factory in Tsukuba. Their results illustrate the potential advantages of phase contrast imaging and the sensitivity to minute density variations—on the order of 10^{-9} g/cm³.

In this paper, a method concerning digital phase difference amplification for X-ray interferometers is suggested, aims at improving the sensitivity of X-ray interferometers for the measurements of biological samples. Phase information is amplified digitally by phase difference amplification by means of Fourier transform from two different interference patterns, wherein one includes the information of the object, and the other only the background of the system.

As we know the interference pattern contains the image from the object and, overlapping the carrier wave fringes in the X-ray interferometer, so the pure phase information can be extracted from interference patterns by eliminating the carrier wave component. In this method, phase difference can be amplified up to 100~1000 times, it means the sensitivity to minute density variations—can be on the order of 10^{-12} g/cm³.

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Development of an apparatus for speckle image observations near the X-ray diffraction spots using focusing beam.

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We have developed a new apparatus for speckle image observations using high coherent beam from a synchrotron radiation beam. The experiment was carried out at the undulator beamline BL-20XU of SPring-8. Figure 1 shows optics layouts for speckle measurements. A cross-slit with opening size of 100 micron x 100 micron was used as a pseudo-point source, and a Fresnel zone plate (FZP) (diameter=100micron, focal distance=16cm at 8keV, diffraction limited focal spot size =0.25micron⁽¹⁾, fabricated at NTT-AT.) was set about 200m downstream from the cross-slit. Sample was set at the focal plane of the FZP. A visible-light converted type two-dimensional image detector coupled with a CCD camera (Hamamatsu. 1024x1024 pixels) was set at 1.2m downstream from sample. Temperature of the sample was controlled from the 473K to below 10 K with a Liquid He cooling system. Sample rotation range was $-3<20<61^\circ$. We observed the diffraction spot speckle image of NbSe₂ single crystal (006). The difference of periodicity between the

speckle pattern of $NbSe_2$ at the room temperature and that below the superconductive transition point was observed. We will show a possibility that this method reveals periodic and/or random structures which is difficult to be observed with the conventional diffraction methods.

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speckle measurement at BL20XU

Study of Matrix Effect with a Full-Field Imaging X-ray Fluorescence Microscope

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When two or more elements in a specimen are analyzed simultaneously by x-ray fluorescence with a microprobe, a matrix effect causes not only a degradation of quantitative analysis but also the broadening of the apparent spot size of the microprobe. A full-field imaging method has potential to solve this problem because it can specify the point of the specimen where the x-ray fluorescence photon generates. In addition, the effect of secondary excitation should be corrected by considering 3-dimensional elemental distributions of the specimen. Therefore, we have been developing a full-field imaging microscope and performing x-ray fluorescence microtomography for the quantitative observation of elemental distributions of Fe and Ni in a synthesized diamond [1]. For more accuracy of the quantitative analysis, the matrix effect and other influential effects were observed and discussed in this presentation.

A full-field imaging x-ray fluorescence microscope system was constructed at BL3C2 in the Photon Factory (KEK). A quasi-monochromatic beam from a double multilayer monochromator was used as an excitation beam. X-ray fluorescence from a sample was imaged by the Wolter mirror (\times 10) onto a CCD camera. The mixture solution of FeCb (II) (3.2 mol/l) and NiCb (0.8 mol/l) in a glass capillary tube (ϕ : 300 µm) was used as the sample. Its x-ray fluorescence image was acquired with the limited irradiation area of the sample. A photon counting image of Fe Ka line is shown in Fig. 1. Its exposure time was 0.8 sec×1000 and the excitation energy was 8.40 keV. The x-ray fluorescence image can be seen outside of the irradiation area. These tails were caused by the excitation due to the matrix effect and the scattered incident beam.



Fig. 1: Photon counting image of Fe Ka line of the mixture solution sample.

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Integrated Image Projection and Detector Array for Real-time Quantitative Synchrotron XRF Elemental Imaging using the X-ray Microprobe

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The *Dynamic Analysis* (DA) method enables the generation of quantitative proton induced Xray emission (PIXE) elemental images using a matrix transform that lends itself to real-time projection [1]. PIXE and synchrotron X-ray fluorescence (SXRF) display many similarities, such as non-destructive trace element analysis, deep penetration and similar X-ray spectra. These similarities have enabled the adaptation of the DA method to generate real-time elemental images using the X-ray Fluorescence Microprobe (XFM). DA for SXRF has been implemented in the GeoPIXE software using recent fundamental parameter compilations and a treatment of scatter peaks. Tests of the method using the 2-ID-E XFM at the APS, and samples with demanding multi-element overlaps, demonstrate the potential of the method.

The aim is to combine the DA imaging approach with an advanced 384 element silicon detector array being developed at Brookhaven National Laboratory (BNL) [2]. The detector system will combine the BNL array and a CSIRO pipelined parallel processing engine to yield a detector with 1-2 steradian solid-angle for high detection sensitivity and a maximum total count rate exceeding 10^7 counts per second, tightly coupled to sample stage control for fast scanning at ~ 10^3 pixels per second.

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Acknowledgments

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REPRODUCTION OF HOLOGRAM IMAGE USING A ZONE PLATE FOR HARD X-RAY RADIATION

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The images of the silicon test object have been studied. An image for in-line hologram for hard X-ray (8 - 18 KeV) is presented. The transmission of a hologram image for hard X-ray radiation using Fresnel phase zone plate has been investigated. The experimental investigations have been conducted on the station BL29XU, Spring-8.

Abstract

X-ray waveguide optics for x-ray in-line holo-microscopy

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We present recent advances of x-ray point source production by two-dimensional lithographic x-ray waveguide nanostructures. A pre-focussed synchrotron beam is coupled in from the front side of a polymer channel embedded in silicon, which acts as a waveguide and blocks the unwanted (super-illuminated) part of the beam, leaving a well defined hard x-ray beam of around 30 x50 nm2 the end of the 1-5 mm long device. The field propagation and efficiency of the combined KB mirror and waveguide optics are discussed. A waveguided beam with a flux on the order of 106 photons per second has been achieved in a first ESRF undulator experiment using this coupling scheme. The divergent and coherent beam exiting from the quasi-point source can then be used to magnify the wave front distorted by the object. The scheme of in-line holographic imaging, the image formation and object reconstruction will be discussed.

Phase Imaging with X-ray Talbot Interferometer Using Gratings Fabricated with LIGA Process

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X-ray phase imaging attracts increasing attention in this decade. X-ray Talbot interferometry (XTI) has been proposed as a novel X-ray phase imaging and phase tomography [1, 2]. In this paper we demonstrate a phase measurement and tomographic image reconstruction with high-energy X-rays, which was realized by using gratings fabricated by the Lithographie, Galvanoformung, Abforming (LIGA) process. Higher-energy XTI makes it possible to capture wide-area phase images for large and thick biological and polymer samples.

The XTI uses two transmission gratings. When a transmission grating is illuminated by coherent X-rays a periodic pattern (self-image) is generated at specific distance from the grating, which is known as the 'Talbot effect'. If a sample is placed in front of the grating, the self-image is correspondingly deformed, where differential phase shift by the sample is involved. In the XTI the deformation is then depicted as a moiré pattern, which is formed by the second grating placed on the self-mage.

For high-energy XTI the gratings are required to have a period of less than several microns and a high aspect ratio (more than 10 for an amplitude grating) to generate high-contrast moiré fringes. It is, however, difficult to fabricate such a grating with conventional techniques, especially when the period approaches one micron. The LIGA process is a promising method for fabricating such a grating. The LIGA allows us to make not only high-aspect-ratio but wide-area gratings, which is favorable for large and thick materials. We fabricated a grating using the LIGA process, and phase imaging and phase tomography of biological samples was successfully demonstrated.

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Biomedical phase imaging using a grating interferometer

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The imaging of biological or medical tissue samples in the hard x-ray range is problematic due to the low absorption coefficient of light elements. We have recently developed a grating interferometer that allows us to visualize the phase shift gradient, which can greatly increases the contrast of such samples. As example the figure below shows x-ray images of an animal organ which was put in a container with water. We have used synchrotron radiation of 17 - 18 keV photon energy for imaging. Due to the limited size of the synchrotron beam of 3mm, the image has been stitched together from 20 sub-frames. In absorption contrast, only air bubbles and some fatty tissue are visible, whereas the complete organ with many details can be seen in the differential phase contrast image. We expect that the technique can be useful to reduce the dose applied in medical examinations, especially in mammography.



X-ray micrographs of a rat heart in water taken with polychromatic radiation of 17-18 keV photon energy.

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Extreme Ultraviolet (EUV) lithography is the most likely candidate for adoption by the semiconductor manufacturing industry as the next generation lithography technique. In this technology, reflective optics coated with Mo/Si multilayers that provide high reflectivity in a narrow band of wavelengths near 13-nm are used for imaging. One of the remaining issues to be solved before EUV lithography can be commercialized is the reduction of defects on multilayer-coated EUV masks. Zone plate microscopy at 13-nm wavelength is an ideal technology to find and understand multilayer defects not only because it can provide very high spatial resolution (30 nm or possibly below), but also because it can relatively easily provide phase contrast imaging if a phase plate is placed in the back focal plane. In this talk we will present the design of and preliminary results from a phase-contrast zone plate microscope at the Advanced Light Source at Lawrence Berkeley National Laboratory that will be used for reflection imaging of EUV masks at 13-nm wavelength.

Development of a Large Angle X-ray Spreading Element for Projection Xray Microscopy with Undulator Light Source

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X-ray diffuser has been used for the purpose of eliminating image noises from various optical elements, and for the purpose of getting noiseless image. Therefore X-ray diffuser is now widely used in various imaging techniques such as laser optics, X-ray computed tomography (X-ray CT) and X-ray Topography. Though X-ray diffuser has been used for the purpose of noise elimination, a new possibility of X-ray diffuser as a large angle X-ray spreading element for use in projection X-ray microscopy with undulator light source is discussed. Figure 1 shows the experimental setup for the measurement of angular spread of 30keV X-rays. The X-ray spreading elements used in this experiment are made of Al₂O₃ and SiO₂ powders. Figure 2 shows the measured angular spread in related to the average grain size of Al₂O₃ powders. It was measured that the maximum angular spread was 104µrad at 2µm grain size, and the angular spread was depressed at smaller average grain sizes. As the reason for the rapid decrease of the measured angular spread at grain sizes of 0.05 and 0.35µm was guessed that is due to coagulation of each Al₂O₃ particle, a SiO₂ slurry whose SiO₂ particles does not coagulate each other, was tested as an X-ray spreading element. Figure 3 shows the measured intensity profile of the spreaded beam from the mixed SiO₂ slurry (72nm grains mixed with 8.2nm grains). The angular spread of about 1000µrad. was achieved.



Figure 1 Scheme of the experimental setup



Figure 2 Relation between Al₂O₃ grain size and angular spread



Figure 3 Intensity profile of the field of vision using the mixed grain size SiO_2 slurry

Submicron-Resolution X-ray Topography Using Fresnel-Zone-Plate Magnification

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We have developed an image magnification method using a Fresnel zone plate (FZP) to obtain submicron resolution in X-ray diffraction and topography [1, 2]. This paper describes the FZP method for local strain analysis of microstructurefabricated silicon materials.

The experiments were performed at BL16XU in SPring-8. The FZP was a phase modulation type, whose zone structure was made of a 2-micron-thick Ta layer on a 2-micron-thick SiN membrane. The diameter was 22 microns, and the width of the outermost zone was 0.2 micron. The experimental setup is shown in Fig. 1. Si-400 reflection was used at a photon energy of 8.5keV (Si-111 monochromatized), at which the focal length of the FZP was 31mm. The diffracted X-ray image was expanded 27 times by the FZP (50mm from the sample) and focused on a slit (900mm from the FZP), through which the X-ray intensity was measured. The slit aperture was located at a 200-micron-offaxial position to avoid the zeroth-order direct beam.

The sample used was a Si{100} wafer upon which an oxide pattern was fabricated (Fig. 2A, 2B). The topograph shown in Fig.2C was obtained by moving the sample in two dimensions. The lines of high X-ray intensity, which correspond to the regions between the oxide stripes, represent surface damage caused by etching the oxide. Figure 2D shows the contour map of rocking curves obtained at intervals of 0.25 micron along Y = 0. The peak positions P Of the rocking curves were estimated by parabola fitting (Fig. 2E). The variation of P corresponds to d/d~ $\pm 1 \times 10^{-5}$, indicating that tensile strains were caused under the oxide layers. The spatial resolution was estimated to be less than 0.5 micron.

This work was performed as a part of research proposal number C04A16XU-3111N.



Fig.1. Experimental set-up



Fig.2. Strain analysis of an oxidepatterned silicon wafer

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Crystallinity Estimation of Strained-Si Wafers by Using Highly Parallel X-Ray Microbeam

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Strained-Si (s-Si) wafers are expected as the next generation high-speed electronic devices. In order to estimate the crystallinity of s-Si wafers, we developed a high flux X-ray microbeam with a small angular divergence and a narrow energy bandwidth [1]. The X-ray microbeam is formed at SPring-8 by combining the Si single crystals and an X-ray mirror.

We estimated three commercially available s-Si wafers. The structures of these samples are s-Si/SiGe/Si, s-Si/SiGe/SiO2/Si and s-Si/SiO2/Si. The thicknesses of s-Si layers of these samples are 17.5 nm, 17.0 nm and 15.0 nm, respectively. The high flux X-ray microbeam enable us to obtain the reciprocal lattice maps of these extremely thin s-Si layers.

The intensity distributions in reciprocal lattice space maps reveal that the lattice parameters of s-Si layers are almost the same as expected values. However, the crystallographic directions normal to s-Si lattice planes greatly distribute about 500 micro radian.



Fig.1. Reciprocal lattice map of s-Si/SiO2/Si wafer.

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High Resolution X-Ray Inspection Microscope equipped with a Field Emission Gun and its Application

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Until now, the spatial resolution of Projection X-ray Microscope Inspection Systems using thermal electron emitters such as those made of lanthanum hexa-boride (LaB₆) has been achieved to a level just better than 0.4μ m[1]. Now, we have developed an X-ray Microscope Inspection System using a thermal field electron emitter coupled with a new type of electron condenser lens. This system is capable of 0.1μ m spatial resolution.

Our currently achieved spatial resolution has been measured with a 2000 lines/mm, 0.2µm thick gold grid. Figure 1 shows an X-ray image of the gold (Au) grid taken with photographic film, using a 0.4µm thick Cr-target, at applied voltage 25kV, where we can clearly see Fresnel fringes. We estimated the resolution as half the width of the first maximum of the Fresnel fringes, as suggested by Cosslett and Nixon[2]. The width of the first maximum of the Fresnel fringe is 0.2µm which is expressed as $2d_F$ in the micrograph. By substituting b=50µm(the spacing between the target) and = 0.23nm(wavelength of Cr K -line) to the relation $d_F = (b_{-})^{1/2}$, we have $d_F = 0.107$ µm. So it is reasonable to infer that the Fresnel fringes are formed by Cr K -line.

Emulsion layers of photographic films were examined with the X-ray microscope before and after development and independent particles postulated to be AgBr and Ag were observed. Some other examples will be reported.



Fig.1 X-ray micrograph of gold transmission grating (2000 lines/mm) taken with photographic film, Cr target 0.4µm thick, 25kV. From the width of the first maximum of Fresnel fringes, spatial resolution of about 0.1µm is proved.

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XFH study of dilute system using cooled APD

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X-ray fluorescence holography (XFH) is a relatively new experimental tool for determination of a local atomic structure around a specific element. Analysis of local structure around impurity in single crystal is one of most important applications of XFH. Thus, we have developed XFH setup for the dilute systems, which enables us to record a hologram within few hours. A cooled avalanche photo diode (APD) is one of suitable detector for this purpose, because it has 10 % energy resolution and fast counting system at the countrate of 10^8 cps. In the present study, we carried out the XFH measurement of dilute system using the cooled APD.

The measured sample was $Si_{0.999}Ge_{0.001}$ single crystal. The incident energies were

14.5 - 17.5 keV with 0.5 keV steps. Radiations from the sample were detected by the cooled APD. Photon signals were processed by fast amplifier, signal divider, discriminator and scaler. By scanning the discriminator's threshold, we determined lower and higher levels of pulse heights of Ge fluorescent X-rays and elastic scatterings. We collected the intensity of Ge fluorescence and elastic scatterings simultaneously. Figures 1 (a) and (b) show the 2D maps of the fluorescence and elastic scattering intensities measured at the incident energy of 15.0 keV. Comparing these, it is found that the pattern in Fig.1 (a) is overlapped by that in Fig. 1 (a). This is due to a long tail at the energy region below the main peak of the elastic scattering. But, it is possible to calibrate true hologram pattern from the patterns in Figs. 1 (a) and (b). Figure 1 (c) shows the calibrated hologram pattern. From the observed holograms, atomic images around Ge were successfully reconstructed.



Figure 1. 2D maps of intensity variations.

Spectroscopic Photoemission and Low Energy Electron Microscope (SPELEEM) at MAX-Lab

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We describe a SPELEEM microscope (Elmitec, GmbH) which will be initially installed as a second experimental station at a high resolution, undulator based soft X-ray beamline (BLI311) [1] working in the photon energy range of 30–1500eV at the MAX II synchrotron radiation source (E=1.5GeV). The general design of the beamline is based on a horizontally focusing premirror, an SX-700 type of plane-grating monochromator and Kirkpatrick-Baez refocusing optics. The beamline is equipped with an experimental station consisting of separate analyzer and preparation chambers. The photoelectron microscope will be installed after the existing experimental chamber and the photon beam will be refocused with two (vertical and horizontal) refocusing mirrors. The output flux from the monochromator is 10^{11} - 10^{13} ph/sec depending on the photon energy used and beamline settings. The flux will be delivered to the $100 \times 100 \mu m^2$ spot size on the sample through the beam separator and the objective lens of the microscope at normal incidence. Two main advantages of having normal incidence illumination as compared to grazing incidence are the absence of shadowing effects for samples with surface topography and more even beam spot on the surface (grazing incidence stretches the light spot in the horizontal direction by a factor of 4 at a 15⁰ incidence angle). The preparation chamber for the photoelectron microscope is designed to house a sample manipulator with e-beam heating, ion sputtering gun as well as LEED optics and Auger Electron Spectrometer (AES) for sample characterization. We aim to reach the spatial resolution ~30nm in photoemission mode of operation and ~10nm in the LEEM mode. The possible application examples - spectroscopy of single quantum dots and nanowhiskers, phase separation in strongly correlated systems, magnetic structures, phase transformations in overlayers etc. will be presented.

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Scanning Transmission X-ray Microscopy with Momentum Analyzer

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X-ray topography is widely used for characterizing dislocations and defects in single crystal samples. The spatial resolution of the topographic image is usually a few μ m that is limited by both the Fresnel diffraction and detector's spatial resolution. The scanning microscopy with diffracted-X-ray-detection method, so called "scanning topography", may be applied to high-spatial-resolution topographic imaging, because the spatial resolution of the image is determined by the spot size of the focused beam.

However, when a convergent beam is diffracted by a nearly perfect crystal, only a small portion of incident beam is propagated to the detector, because the diffraction occurs within a narrow angular range that is smaller than the angular width of the incident beam. The spatial resolution of scanning microscopy image is limited by the diffraction theory of light, that is expressed by $\Delta x = 0.61\lambda/NA$, where the NA (Numerical Aperture) corresponds to the convergent angle of the focused beam. Therefore, it may be probable that the spatial resolution of the topographic image may be limited by not only the NA of the beam-focusing optics but also by the angular width of diffraction at the sample. In order to confirm this assumption, we have tried scanning microscopy experiment in which the transmitting beam through the test object is filtered by a crystal analyzer that selects a portion of the transmitted X-ray beam. The schematic diagram of the experimental setup is shown in Fig. 1. The experimental setup is essentially the same as that of the conventional scanning microscopy experiment, except the analyzer crystal (Si 111 Bragg reflection). Scanning microscopic images of resolution test patterns were taken in order to characterize the imaging properties.

In the measured images, strong artifacts (edge-enhancement) appear in the horizontal direction, and some deterioration of contrast is observed in the vertical direction. Therefore, it is considered that the imaging properties are determined not only by the primary focused beam but also by the acceptance for the transmitting beam.



Fig. 1. Schematic diagram of experimental setup and measured image of resolution test patterns by scanning transmission X-ray microscopy with crystal analyzer. X-ray energy is 10 keV.

Scanning µ-x ray excited luminescence in semiconductors

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A new microprobe approach based on x-ray excited luminescence is introduced, demonstrating that luminescent mapping and spectroscopic analysis on semiconductor layers can be also coupled with elemental sensitivity on a microscopic scale.[1] We have applied this new method principally to the study of nitride based films: Mn doped GaN and freestanding GaN.[2] Owing to their wide-bandgap, polarization-based effects, and superior mechanical, magnetic and thermal properties, group III nitrides provide a promising material for optoelectronics devices. Basically in the UV optical range, their luminescence presents astonishing properties with potential applications in new and sophisticated technologies (laser diodes and light emitting diodes). However, a deep understanding of the phenomena involved is still required to allow their full exploitation in the generation of systems with preestablished properties. By imaging and dispersing simultaneously x-ray fluorescence and luminescence excited by synchrotron radiation, this novel approach opens new facilities to the semiconductor research community. Investigation of multiexciton states in quantum dots by non-linear excitation, site and chemical environment in quantum wires by x-ray absorption spectroscopy and x-ray excited optical luminescence, elemental homogeneity by x-ray fluorescence [3] are just a few examples of the potentialities of this technique. Despite the complexity of the energy relaxation and transfer processes, which limit its potential as a detection scheme, there remains a lot of exciting physics to be investigated.

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Evaluation of Hard X-Ray Con-Focal Optics

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Con-focal optics is widely applied in optical microscopy since the method has advantages in the sensitivity, spatial resolution, and depth resolution. However, the con-focal optics has never been applied as microscopy in x-ray region. We will present about evaluation of hard x-ray con-focal optics constructed by using two Fresnel zone plates (FZPs) with identical characteristic.

Experimental work has been carried out at beamline 20XU of SPring-8. The 20XU has an undulator source, and monochromatized 8 keV x-rays were used. The "end station" is about 200 m apart from the monochromator, and long propagation length is available at the 20XU.

The con-focal optics consists of an illumination system confugured with FZP1 and a detection system with FZP2 having same characteristics with FZP1 (Fig.1). In a general situation, these two systems are required to be symmetric with respect to an object point. For our setup, however, magnification factor of the detection system is set to be much smaller than demagnification factor of the illumination system. It is because a quite long source-to-FZP1 distance (~200 m) is required for a fully-coherent illumination to the FZP1. Therefore, a small aperture (1/100 of source size) is needed at detector for configuring a con-focal condition. A high-spatially resolving imaging detector coupled with optical

microscope is used instead of the aperture. Spatial resolution of the detector is about 1 micron and it is enough for experimental condition (magnification of detection system: 20, diffraction-limited focusing size of FZP: 120 nm)

The con-focal optics is evaluated by knife-edge response at con-focal point, and by scanning profile of a test pattern. The con-focal optics shows different characteristics from the optics without FZP2.



Figure 1: Schematic diagram of x-ray con-focal optics constructed at BL20XU of SPring-8

Towards table-top time-resolved soft x-ray microscopy imaging with a laboratory high-harmonic source at 100 eV

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Nowadays soft x-ray microscopy is well established at synchrotron radiation sources [1] as well as at laboratory scale [2]. Many different techniques for specific applications and contrast mechanisms have been developed. However, these x-ray microscopy techniques are rarely connected to time-resolved measurements, i.e. combining a high spatial with a temporal resolution by employing visible-pump x-ray-probe measurements. One reason for this is the lack of brilliant soft x-ray sources delivering sufficiently short pulses in the fs-domain. In this contribution we demonstrate imaging with a laser driven table-top soft x-ray microscope. By combining a high-harmonic light source, optimized for having a maximum brightness at around 100~eV, a multilayer-mirror setup as a condenser and a zone plate as microscope objective, we were able to resolve 200~nm structures of a diatom sample [3]. Due to the pulsed nature of the high-harmonic radiation the microscope offers the possibility of adding a temporal resolution to established microscopy techniques like, e.g. high-resolution imaging or spectromicroscopy. The pulse duration in the order of a few femtoseconds or even below [4] allows for studying ultrafast processes not easily accessible with conventional synchrotron sources. Further development of the high-harmonic sources will extend the wavelength range into the water window region [5] enabling time-resolved microscopy at the carbon K-edge.

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Hard X-ray Interference Microscope with Two Zone Plates

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A hard x-ray interference microscope with two zone plates was designed and tested at SPring-8 BL20XU. This was the first on-axis interference microscope with zone plates on the hard x-ray region.

The optical system is shown in Fig. 1. A zone plate is a circular diffraction grating so that there are various diffraction orders. If parallel x-rays are incident on a zone plate, the first order x-rays are focused at the focal length f downstream the zone plate, the minus first order x-rays diverge as if they emerge from the point f upstream the zone plate, and the zero-th order x-rays are simply transmitted in the forward direction. Then, the two zone plates were placed twice of the focal length apart from each other. The (+1, 0) order x-rays of the beam illuminating the specimen had the same path as the (0, -1) order of the other beam after the two zone plates. Then, the two beams interfered with almost the same phase difference over the image plane.

The two zone plates had the same specifics. The diameter and the outermost zone width were 155 microns and 0.1 microns, respectively. The beam-line BL20XU has an x-ray undulator source. The distance between the source and the experimental hatch is 245 m, so that the full area of the zone plate could be illuminated coherently. The image contrast could be adjusted by a phase shifter shown in Fig. 1 (rotating quartz sheet of 62 microns in thickness). Figure 2 shows the bright and dark contrast images of a diatom at 10 keV.



Fig. 1 Optical system.

Fig. 2 Bright (left) and dark (right) phase-contrast images of a diatom at 10 keV.

Reflection Mode Imaging with High Resolution X-ray Microscopy

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We report on the first demonstration of imaging microstructures with soft x-ray microscopy operating in reflection geometry. One of the limitations of soft x-ray microscopy has been the requirement that samples be prepared thin enough for x-ray transmission. With the reflection geometry, thin multilayered samples on thick substrates can be imaged. For the demonstration experiments, the sample was illuminated with 500 eV x-rays at an incident angle of 6 degrees. The image formed from the reflected light was magnified by a zone plate onto a CCD. In transmission mode, this geometry would have provided a 10 by 10 micron field of view. With the shallow angle reflection geometry, the image is squeezed in one direction, so the resultant field of view was approximately 10 by 100 microns, but only approximately 10 by 3 microns was within the depth of focus. Future experiments with this geometry will include tuning the incident angle to obtain depth resolution in grazing incidence geometry. In combination with XMCD as magnetic contrast mechanism this mode will allow studies of deep buried magnetic interfaces with respect to the local variation of magnetic roughnesses and to obtain a magnetization depth profile with lateral resolution. This will provide important information to characterize the magnetization behaviour at interfaces.

Projection-type micro X-ray fluorescence and diffraction imaging

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This paper concerns the recent instrumentation for projection-type imaging for X-ray florescence and diffraction. The method is quite different from normal scanning type imaging, which has been widely used at many synchrotron beamlines all over the world. The present instrument uses quite a wide beam (typically 8mm (H) \times 0.2mm(V)), which illuminates the whole sample surface in a low-angle-incidence arrangement (0.5~1.5 deg). The detector used is a CCD camera based on TC281 (Texas Instruments) working at 30 fr./sec, equipped with a collimator inside, and the distance between the sample surface and the detector is set extremely close, in order to enhance both spatial resolution and efficiency. In order to distinguish elements effectively, most of the experiments were performed with monochromatic or quasi-monochromatic X-rays. The experiments at BL-16A1, a multipole wiggler beamline at the Photon Factory, indicate that the typical exposure time is 30-300 msec for one XRF image (1000×1000 pixels) corresponding to 0.64 mm² area with a spatial resolution of ca. 15-20 micron. Furthermore, in addition to the normal XRF, one can perform X-ray absorption fine structure (XAFS) and X-ray diffraction (XRD) imaging by repeating exposures with a synchronized scan of the primary X-ray energy. Therefore one can analyze the distribution of the chemical composition, the crystal structures, the orientation, the lattice strains, chemical states, and inter-atomic distances simultaneously for inhomogeneous sample within very short time. From a viewpoint of the application to the materials science, in particular to combinatorial research, not only the use of synchrotron radiation but also new X-ray microscopes with laboratory source would be demanded. Recent developments will be introduced at the conference.

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Nanometer Focusing using Diffractive X-ray Optics

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Significant attention has been paid recently to the spatial resolution that can be achieved with x-ray optics. While it is widely accepted that a spatial resolution approaching 10 nm is possible using both far-field optics and near-field optics, the feasibility of focusing below 10 nm has only recently begun to be explored.^{1,2}

We will discuss the focusing properties of diffractive optics designed to achieve a spatial resolution below 10 nm. The diffraction properties of these optics are characterized by dynamic diffraction effects.³ This results in diffraction efficiencies well above 40% for the diffraction order used for focusing, and to correspondingly small efficiencies for all other diffraction orders. Dynamic effects also lead to radial changes of the phase of the diffracted wave, which will change the focal length and cause spherical aberrations. Increased sensitivity to deviations from the Bragg condition with increasing numerical aperture requires tilting of the zones with respect to the optical axis, and limits the energy range for which high-resolution diffractive optics can be used.

A technical concept for achieving sub-10 nm focusing is the Multilayer Laue Lens.^{4,5} An MLL consists of two crossed linear zone plates fabricated by deposition of a graded multilayer on a plane substrate. The diffraction and focusing properties of this system will be presented.



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The focusing limits of Fresnel Zone Plate x-ray optics

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With this contribution we will present the results of a theoretical work in which we investigated the limitations of Fresnel Zone Plate (FZP) x-ray optics. We will in particular address the question: what is the smallest spot size to which an x-ray beam can be focused ? Based on previous work [1,2], where the focus limit has been investigated in the specific case of a narrowly tapered x-ray waveguide capillary, we have further generalized our approach and developed a theoretical treatment for solving directly Maxwell's equations for more complex boundary conditions, e.g. a linear FZP array. We will show how such a problem can efficiently be reduced to the solution of an Eigenvalue problem similar to frequently occurring problems in quantum mechanics [3]. The solution of the problem, i.e. the complex value of the electrical field as a function of the space coordinates in- and outside of the FZP, is then found by applying basic matrix diagonalization schemes in combination with Fourier space propagation methods.

Based on the results of our calculations we will discuss the dependency of the minimum achievable focal spot size with FZP optics as a function of several parameters. Just like in the case of a single narrowly tapered waveguide we find a minimum focal spot size for hard x-rays of the order of 10 nm (FWHM), the exact value depending only on the electron density of the confining material. Finally, we will discuss whether this limit applies to all x-ray focusing devices.

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Hard x-ray micro focusing with a single bounce multilayer optic

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The spatial resolution of many x-ray investigation techniques (microscopy, micro diffraction, micro absorption spectroscopy, micro fluorescence) is determined by the use of an x-ray beam with a small cross section in both directions. Several focusing techniques (Fresnel Zone Plates, Kirk-Patrick Baez mirror systems, or bent crystals) can be used to provide a beam size of typically a few square microns. The practical use, i.e. the alignment, of the latter micro focusing optics is, however, quite difficult and usually time-consuming. Some of the problems are caused by the fact that most existing focusing optics consist of two separate components, with which the focusing is achieved in the two different directions one after the other.



Fig. 1: Schematic drawing of the investigated x-ray optical device. It consists of a curved mirror substrate with a specially designed, graded multilayer coating.

With this contribution we present a novel x-ray optical device which focuses the beam simultaneously in two directions and therefore eliminates some of the problems mentioned above. It consists of a curved mirror substrate with a specially designed, graded multilayer coating (see Figure 1). The grading compensates for the variation of incident angles across the mirror surface. It was fabricated by XENOCS by DC magnetron sputtering [1].

The focusing properties of the optics were tested at the Material Science beamline at the Swiss Light Source (SLS). We will show that this new type of optics can efficiently compress a hard x-ray beam of a width of several hundred microns in both directions into a spot size of a few square microns. Since the device only requires a single reflection, the alignment of the optics is much faster and easier than in case of most currently used micro focusing devices.

[1] For further information, please refer to www.xenocs.com (FOX-2D-CU 12_INF).

demodulation method.

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Any type of microscopy imaging (such as x-ray scanning microscopy, x-ray fluorescence microscopy, SNOM, SEM, STM, and AFM) can be expressed as the convolution process between the object to be investigated and the point-spread-function (PSF) of the imaging system. The spatial resolution is mainly determined by the PSF. Most efforts to improve the spatial resolution are focused on how to reduce it, such as using Fresnel Zone Plate or Kirkpatrick-Baez mirror with several nanometer resolutions. This way is common, and full of difficulties.

An alternative promising new approach is using the propriety deconvolution arithmetic to obtain higher resolution reconstructed image.

In this presentation, we will report the theory and experiment work on improving the spatial resolution of the oversampling microscopy image via the direct demodulation method, which is invented by astronomer, and solve the convolution equation under physical constraints. We can achieve the higher resolution (than the intrinsic resolution) reconstruction of complicated object from the oversampling data, and can greatly depress the influence of noise. Some examples will be discussed. Sub-nanometer spatial resolution of x-ray microscopy can be achieved via this methods.

Design and Performance of Multilayer Coating on the Blazed Grating in 25 – 80 nm Region

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A new beam line for high-resolution angle-resolved photoemission study on solids and surfaces has been constructed at BL13 of the Saga Light Source, which is the first synchrotron light facility in Kyushu Island. In order to realize high resolution, high energy reproducibility and wide energy rage, the monochromator is composed of the combination of grazing incidence and normal incident mounts. The grazing incident mount has two varied line spacing plane gratings and three spherical mirrors, while the normal incidence mount involves a plane grating and a spherical mirror. It is found that resolving power better than 10,000 and photon flux of 10^{10} – 10^{12} photon/sec can be obtained from ray-tracing calculations. To cover the energy gap between the grazing and normal incident mounts, the multilayer-coating on the plane grating has been designed and the performance was examined.

The multilayer-coated grating was designed for the 25–80 nm regions. As the grating substrate, a gold-coated blazed plane grating with 1200 grooves/mm (Spectra-Physics) was selected. Its nominal blaze angle and blaze wavelength are 1° and 29 nm, respectively. Three different multilayer, i.e. SiC/Mg multilayer, Y_2O_3/Mg multilayer and SiC-coated Y_2O_3/Mg multilayer, were coated on each third of the grating by magnetron sputtering. The SiC/Mg multilayer is expected to have a sharp peak around 26 nm, while the Y_2O_3/Mg multilayer covers broad region from 27 to 80 nm. The SiC-coated Y_2O_3/Mg multilayer has high reflectivity in long wavelength region more than 45 nm. The SiC coating is also expected to be strong for heat load. The absolute efficiencies of the +1 order diffraction were measured in a constant deviation configuration (deviation angle: 15°) at BL5B of UVSOR facility, IMS, OKAZAKI.

Synchrotron infrared microspectroscopic imaging of biological sample

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The advantage of the synchrotron source is high throughput at high spatial resolution compared to a conventional thermal source. And the infrared output of the synchrotron beam line was fed into a IR microscope as an alternate infrared light. The coupling of infrared microscope and synchrotron source produces the highest signal-to-noise ratio spectrum with the highest spectral resolution from the smallest sample area. The unapertured beam size of the synchrotron infrared radiation is about $10 \times 13 \text{ micron}^2$. The size of aperture is continuous changed to 5 micro by a commercial motorized aperture. IR spectroscopic imaging uses a single element detector associated with an imaging spectrometer to produce an array of spectra over a sample.

High Sensitivity Chemically Amplified Resist for EUV Lithography

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Extreme ultraviolet lithography (EUVL) is now planned to address the 32 nn node. The top three issues of EUVL resist are sensitivity, LER, and outgassing. It is required that sensitivity of less than 2 mJ/cm², line edge roughness of less than 3 nm (3σ), and total pressure of outgassing less than 10⁻⁶ Pa. Especially, to achieve high sensitivity and small LER the acid generation yield have to be increased. As the results of studying chemical behaviors of many kinds of PAGs such as sulfonium salts and iodonium salts, we succeed to obtain the PAG chemical structure which goes along way toward the sensitivity under EUV exposure. Exposure was carried out at BL3 beamine in NewSUBARU. The resist evaluation system can simulate six-mirror imaging optics. Furthermore, to measure the photodissociation species under EUV exposure, the high sensitive quadrupole mass (Q-mass) spectrometer was connected to the resist evaluation system. FT-IR was utilized to measure chemical structure changing under EUV exposure. Table 1 shows the sensitivity of Resist A and B under EUV, KrF. and EB exposure. The anions of PAG of resists A and B are cyclo(1,3perfluoropropanedisulfone) imidate and nonaflate, respectively. The cation of PAG is same. The sensitivity of resist A and B under KrF exposure and EB exposure have no change. However, resist A is about four times higher under EUV exposure. Both the results of mass spectroscopy and those of FT-IR indicate that more than two acid is generated by one photon under EUV irradiation in Resist A. The internal reaction of PAG is very effective to obtain the high yield of acid generation reactions.

	Resist A	Resist B
EUV exposure	1.1 mJ/cm^2	3.8 mJ/cm^2
KrF exposure	14.5 mJ/cm^2	14.0 mJ/cm^2
EB exposure	14.3 μ C/cm ²	14.6 μ C/cm ²

Table 1. Sensivitity of resists A, B under EUV, KrF, and EB exposures.