The Transmission X-Ray Microscope Project at NSRL

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Synchrotron radiation facility of NSRL has updated through the second stage project and its performance is better than ever. Also a full-field transmission x-ray microscope (TXM) project was proposed in 2003 and was put into practice in 2004. According to the schedule, the microscope will operate by the begining of 2006. It will employ the radiation from bend magnet and be installed on a newly-built beamline. The spatial resolution theoretically is about 50nm.

Main parameters of the TXM are as follows.
Condenser zone plate (KZP7): diameter 9mm; outermost width 50nm
Objective zone plate (MZP): Diameter 80microns; outermost width 40nm
Pinhole: Diameter 15microns
Spectrum resolution ($\lambda/\Delta\lambda$): about 600
CCD (Andor Ltd Co.): 13microns $\times$13microns/pixel, 1024pixel$\times$1024pixel
Magnified : $\times$800
Work wavelength: 2.4nm

X-ray Microscope with a Gas-puff Plasma X-ray Source and Grazing Incidence Mirrors

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We have developed a laboratory-size X-ray microscope that consists of a gas puff plasma Z-pinch X-ray source, Wolter-type1 mirrors, and a back-illuminated CCD camera.

To realize a compact and high resolution X-ray microscope, we designed an optical system that includes a condenser and an objective mirrors. We also fabricated special 100x and 300x objective mirrors whose sample-to-objective distances are 20 and 15 mm, respectively. Resultantly, the overall length of the optical system is less than 6 m. Moreover, ray-tracing simulations showed that the magnification was variable by changing the sample-to-objective and objective-to-detector distances. For example, in the case of the 100x objective, the magnification factor is varied over a range between 60 and 300. Thus, we can obtain both coarse and detail images at suitable magnifications without exchanging objectives.

Figure1 shows a schematic of the X-ray microscope. In Fig.1, the gas-puff plasma X-ray source equips capacitor banks that store 1.2kJ at charged voltage of 30kV. The X-ray wavelength is determined with a combination of the working gas (Ne, Ar, N_2) and filter (Ti, Al, C) materials.

Imaging tests were carried out using the 300x mirror. We confirmed that zones of about 300 nm width were clearly resolved from the image of a zone plate at wavelength of 2.9 nm (strongest radiation line from N_2 plasma). Figure2 shows the image of a glomerulus of a rat, which was embedded in epoxy resin and sliced at 200 nm thickness. It was stained with uranyl acetate and lead citrate. Several images were put together to obtain a large field of view. Each image was taken with a single pulse X-ray source of the wavelength ranging mainly from 10 nm to 20 nm using Ne plasma and a 100nm-thick C filter. Tissues, podocytes, mesangial cells, etc., are clearly observed in the image.

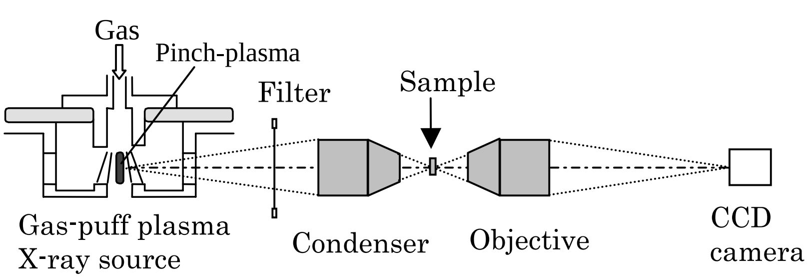

Fig.1. Schematic of the X-ray microscope system

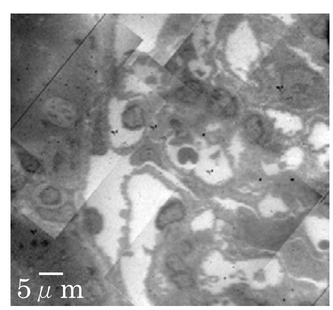

Fig.2. X-ray image of a glomerulus of a rat

Sample: Dr.Y.Muranaka and Dr.I.Ohta
(Hamamatsu University School of Medical)

The Magnetic Transmission X-ray Microscopy Project at BESSY II

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Magnetic transmission x-ray microscopy (MTXM) allows imaging magnetic domains and magnetization reversal processes in externally applied magnetic fields with a lateral resolution below 20 nm, and with a time resolution of 70-100 ps, in a quantitative and element-selective way [1,2]. A transmission x-ray microscope dedicated to this technique and to spectromicroscopy is currently being built at the beamline ID-10 of the synchrotron BESSY II in Berlin. The setup of this beamline, its expected features and possibilities will be presented.

The helical undulator UE46 provides photons with circular-, horizontal-, vertical, and linear polarization under various angles and enables thus x-ray magnetic circular dichroism (XMCD) as well as x-ray magnetic linear dichroism (XMLD) as a magnetic contrast to study ferromagnetic and antiferromagnetic domains. Special attention has been given to enable spectromicroscopy: An off axis condenser zone plate (OTZ) generates a spectral resolution E/Delta(E) of 4000, much higher than in conventional zone plate linear monochromators. To conserve this high value, condenser mirrors with slope errors below 0.05 arc seconds as well as computerized controlled ultra high precision movement of all axes are necessary. The energy can be tuned within 24% by shifting the OTZ with a stage on a polished granite plate. Within the total travel range of 1 m the measured vertical deviations are below 3 microns, ensuring a high linearity of the energy which is necessary for spectromicroscopy.

A condenser with dynamical aperture synthesis allows rotating two condenser mirrors with a speed of up to 800 rpm in ultra high vacuum. A new direct drive has been designed, using a divided stator coupled through a stainless steel vacuum tube to a permanent magnet at the rotor [3]. The measured ripples are below 0.1%, ensuring a homogeneous illumination of the object.

The sample will be in ultra high vacuum and can be transferred by a load lock system to a preparation chamber, which enables in-situ sample preparation. A telescope has been designed which allows simultaneous optical and x-ray imaging of an object. This is not only very helpful for alignment but enables excitation of the sample by femtosecond laser pulses and thus studying magnetization dynamics in pump-probe experiments.

[1] T. Eimüller, P. Fischer P., M. Köhler, M. Scholz, P. Guttmann, G. Denbeaux, S. Glück, G. Bayreuther, G. Schmahl, D. Attwood, G. Schütz, Appl. Phys. A **73**, 697-701 (2001)

[2] H. Stoll, A. Puzic, B. V. Waeyenberge, P. Fischer, J. Raabe, M. Buess, T. Haug, R. Höllinger, C. H. Back, D. Weiss, G. Denbeau, Appl. Phys. Lett., **84**, 3328-3330, 2004

[3] J. Merwerth, A. Oswald, M. Hörz, H.-G. Herzog, T. Eimüller, R. Vatter, H. D. Carstanjen: *Developing a brushless permanent magnet synchronous drive for an x-ray microscope condenser in ultra-high vacuum,* Proceedings of the International Electric Machines and Drives Conference, San Antonio, TX, May 15-18 (2005)

Low Energy-Electron-Excited X-ray Microscopy

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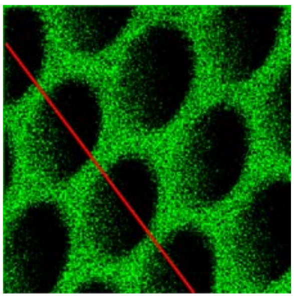
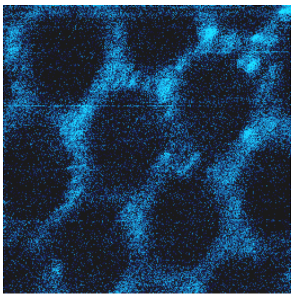
Recently a development of low-energy X-ray microscope have been desired having a spatial resolution of 100 nm in order to investigate living bodies. While a low-energy X-ray microscope using X-ray laser generated by multi-ionization has not yet been accomplished. Very recently a low-energy electron-excited X-ray microscope has been developed by Ueda[1]. When a low energy electron irradiates a specimen surface, X-ray is generated as "bremsstrahlung" and valence band excitation up to few tens electronvolts.

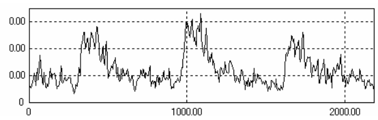
In order to separate the X-ray from secondary electrons and emitted ions for X-ray imaging, a time-of-flight techniques have been adopted where a primary electron beam is pulsed with 220 ns pulse-width. Pulsed primary electron is scanned on the surface. A repetition of pulse beam irradiation accumulates X-ray signal intensity from one position as a pixel for imaging. Since whole signals excepting reflected electron are measured in a TOF spectrum, X-ray image and ion-images are obtained simultaneously from the same position as shown in a figure shown below. Fig.(a) is X-ray image from microchannel plate of which hole diameter is 12 μm, and (b) hydrogen image on the same surface. Fig.(c) is a line profile of X-ray intensity on the oblique line on fig.(a).



(a)



(b)



(c)

A setup for full-field soft x-ray microscopy at the Pohang Light Source

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A test setup for full-field soft-x-ray microscopy has been installed at the Pohang Light Source. The setup has a condenser and an objective zone plates as focusing optics. The outermost zone width of the objective zone plate is 40 nm and the diameter of the condenser zone plate is 2 mm. The zone plates and a sample holder are inside a vacuum chamber and a soft x-ray CCD is used as a detector. The setup has been tested at the 8A1 U7 undulator radiation beam line that has refocusing mirrors with variable radii of curvatures. The test image on 2000 mesh shows a diffraction-limited space resolution; the space-resolution is 50 nm at the photon energy of about 400 eV. The setup has been also tested at the 7B1 bending magnet beam line. In this report, we will present the performance of the setup and some application images obtained at both beam lines.

A new optical design for compact soft x-ray microscope

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The lab-scale x-ray microscope reported so far mostly uses the laser plasma source which contains limited brightness and bandwidth of wavelength comparing with the synchrotron radiation source. The lab-scale system has been not enough to examine the live cells because exposure time is too long to get reasonable resolving power. In this reason, we introduce that high spatial resolution (50 nm) the compact soft x-ray microscope can be performed with reasonable exposure times and contrast. In the present paper, we describe the new optical system design and development of a compact soft x-ray microscope that have high reflective condenser optics and objective micro zone plate (OZP). This x-ray microscope operates in wavelength region of the water window (2.3~4.4nm), where natural contrast between carbon (protein) and oxygen (water) allows imaging of unstained cells under the natural, hydrated environment.

Our compact soft x-ray microscope consists of the laser plasma as an x-ray source, doubled ellipsoidal reflective condenser optics, diffractive zone plate optics and MCP-coupled CCD to record x-ray image. A liquid-jet laser plasma target system was used for the x-ray generation due to its debris free and high average power operation. The doubled ellipsoidal condensing mirror will increase the photon density in the object plane more than one order of magnitude compared to the zone plate condenser adjusting the numerical aperture of the optical system between condenser mirror and objective zone plate. The spatial resolution of the system is determined by a ~12% efficient gold zone plate with an outmost zone width of 35nm which generates an enlarged image of the object in the image plane of detector. The enlarged x-ray image of the object is recorded with CCD coupled with a Chevron-typed MCP.

Recent Progress of X-Ray Microscopy in Ritsumeikan SR Center

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Since 1996, we have been operating a transmission X-ray microscope at BL12 of Ritsumeikan SR center ([575MeV, 300mA], Kusatsu, Japan) [1]. The achieved resolution upto now is 45nm, judged from the edge analysis [2]. The SR center is supported by research projects on nanotechnology and materials of Ministry of Education, Culture, Sports, Science and Technology in Japan from 2002 to 2006. The X-ray microscope beam line is open to researchers from universities, government research centers and companies with this support.
In this conference, we will report some recent results after XRM2002. Most interesting topic is an application of vanadium probe for bio-specimens. Dictyostelium discoideum were cultured and labeled by $VOSO_4$ solution. All cell imaging was done with BL12 transmission X-ray microscope. At the conference, we are going to show the details of an effect of the vanadium labelling.

References

[1] Hirai, A., Takemoto, K., Nishino, K., Niemann, B., Hettwer, M., Rudolph, D., Anderson, E., Attwood, D., Kern, D. P., Nakayama, Y., and Kihara, H. *Jpn. J. Appl. Phys.* **38** 274-278 (1999)
[2] K. Takemoto, A. Hirai, B. Niemann, K. Nishino, M. Hettwer, D. Rudolph, E. Anderson, D. Attwood, D.P. Kern, Y. Nakayama and H. Kihara, in "X-ray Microscopy (eds. W. Meyer-Ilse, T. Warwick, & D. Attwood)", American Institute of Physcis, pp.446-451(2000)

Development of X-Ray Microscope at Ritsumeikan University Synchrotron Radiation Center

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Both CZP and OZP positions were accurately regulated by high performance stepping motors, which is the first step of automatically controlled station. These enable high resolution, element-specific imaging of specimens which have an absorption edge around water window X-ray wavelength region. In addition, a sample stage was also designed more suitable for an observation of biological samples. At the conference, details of the improved system will be reported. This is the first step of developing of a user-friendly X-ray microscope beamline at Ritsumeikan University.

Nickel zone plates for compact x-ray microscopy

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We have demonstrated the first compact x-ray microscope with sub-visible resolution.[1] It operates with a methanol-liquid-jet laser-plasma source providing λ=3.37 nm radiation. Our next microscope is designed for λ=2.48 nm operation with a liquid-nitrogen-jet source.[2] In order to optimise microscope system performance for, e.g., different sources and applications, a high degree of flexibility in the choice of design parameters of the optics is necessary. We have therefore started an in-house fabrication effort of high-resolution diffractive optics for compact x-ray microscopy. The optics are fabricated on 50 nm thin Si_3N_4-foils using a three-layer resist scheme and 30 keV e-beam lithography (Raith 150 system) in combination with reactive ion etching and nickel electroplating methods.[3] The emphasis of this program is on process control for fabrication of high-aspect-ratio electro-plated nickel structures with high uniformity and narrow line-widths.

In the present paper we report results based on recent improvements in the fabrication process. Figure 1 shows a micro zone plate with 25 nm outermost zones and a nickel height of 120 nm. The full zone plate has the quality shown in the figure. In order to further improve the process stability for high-aspect-ratio structures we are introducing an in-situ determination of the electroplating rate based on visual-light transmission measurements. By proper fitting of data this improves the accuracy of predicting the final nickel height in the plating mold and, therefore, avoids over or under plating. In addition to micro zone plates we have fabricated a 4.5 mm diameter condenser zone plate based on 656 100×100 μm stitched field with 50-60 nm zone widths.[4] The CZP is intended for the λ=2.48 nm microscope and has an groove efficiency of 11%±2% at that wavelength.

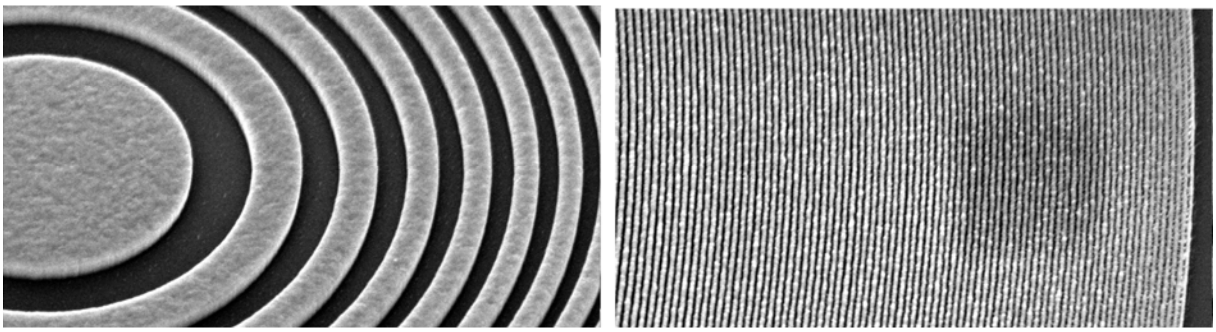

Figure 1. The centre and outermost part a nickel zone plate with 25 nm outermost zone. The innermost part has a nickel height of ~150 nm and the outermost part ~120 nm. (The shaded square in right image is due to SEM viewing contamination)

References

[1] M. Berglund, L. Rymell, M. Peuker, T. Wilhein, and H. M. Hertz, J. Microscopy **197**, 268 (2000)
[2] P.A.C Jansson et. al. Rev. Sci. Instrum., **76** (2005), in press.
[3] A. Holmberg, S. Rehbein, and H.M Hertz, Microel. Engin. **73-74**, 639 (2004).
[4] S. Rehbein, A. Holmberg, G. Johansson, P.A.C Jansson, and H.M Hertz, JVST B **22**, 1118 (2004)

Scanning Transmission X-ray Microscope with Three Axes Laser Interferometer at the NSLS

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Soft x-ray spectromicroscopy frequently involves the acquisition of a large number of images of the specimen over a spectroscopic energy range [1] which places strong demands on the reproducibility of the scanned image field. Previous approaches for ensuring scan field reproducibility have included the use of custom-built laser interferometers [2] and capacitance micrometers [3]; more recently, Kilcoyne *et al.* have shown excellent results using a commercial 2-axis laser interferometer with improved precision [4]. We describe here the retrofit of a 3 axis laser interferometer system onto the hardware of an existing scanning microscope [5], and an all-new scan control system. The use of an interferometer on a third axis allows one to correct for thermal drifts of the focus position (of special importance in differential phase contrast using a segmented detector [6]), and to maintain the beam at the proper focus while acquiring point spectra.

Our system uses an Agilent differential interferometer system to measure the position of the scanning stage relative to the zone plate. The 'actual' position information from this system as well as the 'commanded' positions – also used as pixel advance clock - are streamed as parallel data to a digital servo controller (PMAC2 by Delta Tau). In closed loop the positions are controlled to coincide within 0.3 nm resolution. High resolution scans using piezos are acquired by digitally streaming a set of (*x*,*y*) positions from the scan control computer to the digital servo controller; the streamed positions can incorporate the entire scan field, thus eliminating any computer delays that would otherwise occur at the end of every scan line. The scan control computer communicates using a client-server protocol with a graphical user interface on a separate computer which can include a microscope user's own laptop.

Recent applications of the new microscope will be shown, highlighting its improved performance. We gratefully acknowledge support from NASA, DoE, NSF, and NIH, and many helpful conversations with A. Kilcoyne and T. Tyliszczak.

[1] - C. Jacobsen *et al.*, *J. Microscopy* **197**, 173 (2000).
[2] - H. Rarback *et al.*, *Rev. Sci. Instr.* **59**, 52 (1988).
[3] - C. Jacobsen *et al.*, *Opt. Comm.* **86**, 351 (1991).
[4] - A. Kilcoyne *et al.*, *J. Sync. Rad.* **10**, 125 (2003).
[5] - M. Feser *et al.*, XRM 1999 proceedings, p. 367.
[6] - M. Feser *et al.*, *J. de Physique IV* **104**, 529 (2003).

Xradia's nano-XFI X-ray Fluorescence Imager for High Resolution Elemental Mapping

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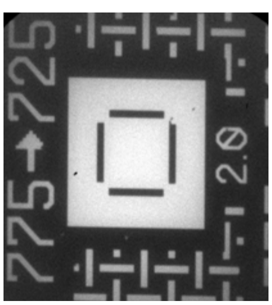
We present a novel approach to map characteristic x-ray fluorescence using an x-ray camera that can image selectively different x-ray energies. The camera employs a Fresnel zone plate as the x-ray imaging optic and achieves currently sub-100nm spatial resolution, ultimately limited by the quality of zone plates available. The camera can be used with electron or x-ray excitation beams and therefore is suitable for attachments to standard scanning electron microscope (SEM) instruments as well as synchrotron-based experimental end stations.

The advantages of this camera compared to traditional energy dispersive (EDS) or wavelength dispersive (WDS) mapping techniques are that the spatial resolution is not limited by the excitation spot, that a whole map of one element is collected simultaneously in a full-field image, that the spatial resolution does not degrade with sampling depth, and that the method has high efficiency and energy resolution for sub-2kV radiation. The imaging is also insensitivite to sample charging experienced with the SEM, and has the ability to perform thin film measurements on small patterned structures.

The X-ray fluorescence imager can be used for both x-ray and electron beam excitation. For example, it can easily be integrated into an existing scanning electron microscope, in a similar way to EDS and WDS equipment.

In contrast to EDS/WDS mapping, it is preferred for the x-ray fluorescence camera to excite a larger spot on the sample corresponding to the field of view of the fluorescence map. High spatial resolution is achieved by directly imaging the fluorescence x-rays on an array detector (CCD), rather than compiling a map by varying the position of the excitation spot. The high spatial resolution of the x-ray imaging camera is also preserved with sampling depth, enabling high-resolution x-ray maps of buried structures and recovery of 3-d information. Since a whole map is collected simultaneously, x-ray fluorescence maps can be collected in a short amount of time compared to WDS/EDS maps.



X-ray fluorescence maps of backend copper integrated circuits are presented as an example of non-destructive imaging of sub-surface structures. The x-ray fluorescence camera enables the in-situ location of voids and defects in backend copper ICs without physical deprocessing or ion beam milling preparation and can be performed on whole silicon wafers.

Left: nano-XFI image of a copper IC sample. The shown field of view is approximately 70x90um in size.

Nano-Positioning Control of Condenser Mirror for Soft X-ray Microscopy System Using 5-axis Manipulator

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This paper presents a method of a nano-positioning control for the high precision focusing of a doubled ellipsoidal condenser reflective mirror using 5-axis manipulator. We have developed the compact vertical type of soft X-ray microscopy system with 50nm resolution for biomedical application. This microscopy system is composed of a laser plasma x-ray source, doubled ellipsoidal condenser reflective optics, diffractive zone plate optics and MCP coupled with CCD to record an x-ray image. The X-ray source was focused on a sample by a doubled ellipsoidal condenser reflective mirror. X-ray source focusing will increase the photon density in the object plane and is very important to approach high resolution imaging. Required degree of freedom (DOF) of optics aligner in X-ray microscope is dependent on the kind of optics, but generally 5-DOF is needed. We used 5-axis manipulator that consists of three linear motions (X, Y and Z) and two tilting motions (R_x, R_y). A linear translation stage is adopted a kind of DC motor with a linear resolution 50nm and travel range of 5mm. The mechanism was controlled with PID controller augmented with closed feedback loop for precision control. A two axis tilt stage is employed a design resolution of 0.23μrad and tilt range of ±7deg. We have designed 5-axis manipulator for the precision position control of condenser mirror optics and have developed to control algorithm by inverse kinematics. The performance of the proposed 5-DOF manipulator is evaluated by using a laser interferometer system with two plane mirror reflectors. The experimental results are depicted in this paper.

References

[1] K. W. Kim, K. Y. Nam, Y. M. Kwon, S. T. Shim, K, G. Kim and K. H. Yoon:, “Conceptual Design of Soft X-ray Microscopy for Live Biological Samples”, J. Optical Society of Korea, 7(2003) 230.

[2] S. H. Kang, D. W. Kang, D. G. Gweon, G. H. Yoon and J. Y. Min, “Design of an Optics Alignment Stage in X-ray Microscopy”, 2th ISNM 2004

High resolution x-ray absorption spectroscopy using a laser-plasma radiation source

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The rapid development of laboratory sources for extreme ultraviolet (EUV) and soft x-ray radiation like laser produced plasmas, high harmonic radiation or x-ray lasers paves the way for applications which before mainly took place at synchrotron light sources. Although the average photon brilliance is normally higher at storage rings, the advantage of laboratory laser produced plasma sources is the potential for time-resolved experiments by pump-and-probe techniques [1,2]. The same laser pulse can be used for the generation of the visible pump pulse and x-ray probe pulse which makes the synchronization of the two pulses very easy. According to the method for x-ray generation time resolution in the range of few ns [3] down to few fs [4] or probably even less could be achieved. In this contribution we present results on near edge x-ray absorption fine structure spectroscopy (NEXAFS) at 284 eV (carbon K-edge) using a compact laser-plasma x-ray source [5]. The spectrometer works with a single x-ray optical element in grazing incidence configuration called off-axis reflection zone plate. Using different sample foils we were able to demonstrate a spectral resolution of E/(DeltaE)=600. The positions of specific absorption peaks are in agreement with data measured at synchrotron sources. Since the off-axis zone plate is a focussing system, the x-ray flux in the detector plane is high enough to record an absorption spectrum on a CCD detector with a single shot. In the single shot modus a spectrum can be recorded before a sample degradation takes place. This will enable time-resolved experiments using pump-probe techniques.

[1] F. Raksi, K. R. Wilson, Z. Jiang, A. Ikhlef, C.Y. Cote, J.-C. Kieffer, *J. Chem. Phys.* **104**, 6066 (1996).
[2] H. Stiel, D. Leupold, M. Beck, I. Will, H. Lokstein, W. Sandner, *J. Biom. Biophys. Meth.* **48**, 239 (2001)
[3] B. C. Larson, J. Z. Tischler, D. M. Mills, *J. Mater. Res.* **1**, 144 (1986).
[4] K. T. Phuoc, A. Rousse, M. Pittman, J. P. Rousseau, V. Malka, S. Fritzler, D. Umstadter, D. Hulin, *Phys. Rev. Lett.* ***91***,195001 (2003).
[5] U. Vogt, T. Wilhein, H. Stiel, H. Legall, *Rev. Sci. Instrum.* **75**, 4606 (2004)

Development of Algorithm for Automatic Alignment of Soft X-ray Microscope Condenser Mirror using Response Surface Method

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Nowadays, many kinds of microscope for living cell are researched and developed. Especially, A X-ray microscope is popular among them. At the first time, we introduce a Vertical Soft X-ray Microscope (VSXM) with a Double Ellipse Condenser Mirror (DECM) and a Zone Plate (ZP) as its focusing unit. Concretely, the VSXM is composed of a target chamber, a mirror chamber, and an image detection part. In the VSXM, the DECM is used to illuminate a specimen. In order to get clear image of a specimen, it must be illuminated uniformly and in maximum intensity (broadly). However, at the integration of the VSXM, the DECM is decentered, tilted, and defocused. Due to the errors, the DECM doesn't illuminate a specimen uniformly and in maximum intensity (broadly). In addition to the requirement, because the quality of images acquired lastly in CCD sensitively reacts to the errors and the DECM cannot be manually aligned by hand in a few micron, an automatic alignment algorithm and system are required. In result, this paper proposes algorithm for automatic alignment of DECM. The automatic alignment algorithm is based on Response Surface Method (RSM) that is a kind of experimental design methods. RSM is a statistical analysis method of response surfaces that variations of responses make, when several explanatory (independent) variables have complicate relationship between them and influences on a response (dependent) variable. In addition, because the VSXM is incongruent to be used in general laboratory environment, this paper suggests a simulator to verify the proposed alignment algorithm. The simulator includes an alternative light source (635nm Fiber Optic Diode Laser), an alternative DECM, an alternative ZP and an alternative detector (CCD). In result, decreasing the errors of decenter, tilt, and defocus, we could align the DECM to illuminate uniformly and broadly a specimen. And for an ideal state (cost function value = 1) that no errors exit, we could reduce CM errors within 5%, that is, Cost function value is 0.95.

X-Ray Microscopy Activities at ACCEL

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ACCEL Instruments GmbH is a worldwide operating engineering and manufacturing company specializing in custom-designed equipment for research and industry in the fields of particle accelerators, medical applications, and synchrotron radiation instrumentation. We design, manufacture, install, and commission complete beamlines as turnkey systems. We also provide components such as undulators, wigglers, monochromators, mirror systems, slit systems and beam monitors as well as other beamline components. Among other types of experimental end stations, ACCEL Instruments also offers X-ray microscopes.

In this poster, we give an overview of our current activities in the field of X-ray microscopy, comprising both scanning transmission microscopes (STXMs) for spectromicroscopy and full-field microscopes (TXMs) for biological specimens. The microscopes are based on designs developed at synchrotron facilities and are realized in close cooperation with the respective research groups.

Phase Contrast EUV microscope for EUV Mask Defect Inspection

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EUV lithography (EUVL) will introduce as next generation lithography of 32 nm node in 2009. Defect-free mask fabrication is one of the technical issue to achieve EUVL. There are two types of defects in EUVL mask: amplitude defect and phase defect. However, phase defect due to the multilayer fabrication can not be resolved with an existing inspection tool. Thus, we constructed the EUV microscope for actinic mask inspection which consists of Schwarzschild optics and X-ray zooming tube. Furthermore, this microscope has a plan to build a Mirau interferometer which can detect the phase defect (as shown in Fig. 1). Magnification of Schwarzschild optics is 30X, and X-ray zooming tube can change the magnification in the range from 10 X to 200 X. So, the total magnification of the microscope is 300 X to 6000 X. And the numerical aperture of Schwarzschild optics is 0.3, so, it can inspect the defect of 10 nm in size. Figure error of mirrors are less than 0.4 nm and surface roughness of mid-frequency was less than 0.15 nm. These Zerodur mirrors were fabricated by ASML Tinsley. D-graded Mo/Si multilayer was coated on these optics by X-ray, Company in Russia. D-spacing matching of less than 0.01 nm has been achieved at the wavelength of 13.5 nm.

Up to now, the characteristics of optics are evaluated. Using this system, Mo/Si glass substrates are inspected, which defects have been already inspected by DUV inspection system. Inspection results and furthermore information will be presented in conference.

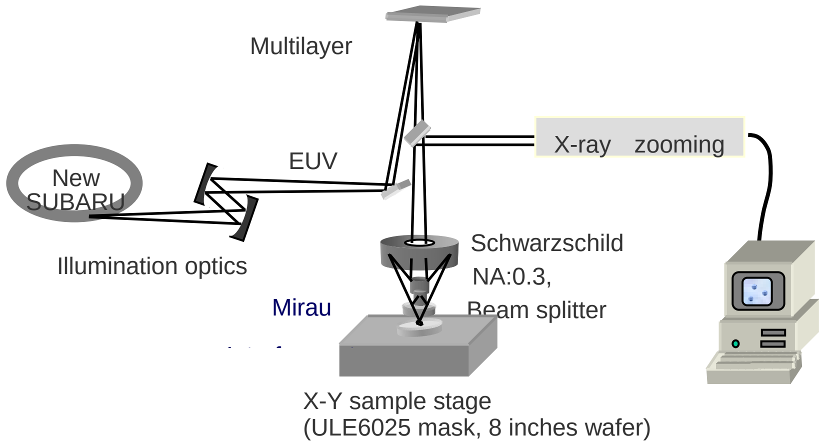

Fig. 1 Configuration of EUV microscope.

Development of a laser plasma x-ray microscope for Living hydrated biological specimens

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Investigating the structure and the function of life object performing advanced life activity becomes important. In order to investigate the life object, it is necessary to observe living specimens with high spatial resolution and high temporal resolution. Since laser plasma x-ray source has high brightness and short pulse duration, x-ray microscope with the laser plasma x-ray source makes possible to observe living specimens. Such as chromosomes, macrophages[1], bacterium[2][3], and so on have been observed by contact x-ray microscopy. The x-ray images obtained by indirect measurements such as the contact x-ray microscopy have difficulty to avoid artificial effect such as irregular due to developing process. Development of an x-ray microscope with laser plasma x-ray source is necessary to avoid such defects. We have been developing an x-ray microscope with laser plasma x-ray source to observe wet live specimens. The detail of the x-ray microscope will be presented.

REFERENCES

[1] M. Kado, M. C. Richardson, J. M. Rajyaguru, M. J. Muszynski, Y. Yamamoto, Proc. of Experimental Biology and Medicine, **220**, 21 (1999).
[2] J. M. Rajyaguru, M. Kado, M. C. Richardson, and M. J. Muszynski, Biophysical Journal, **72**, 1521 (1997).
[3] J. M. Rajyaguru, M. Kado, K. Nekula, M. C. Richardson, and M. J. Muszynski, Microbiology, **143**, 733 (1997).

Hard X-ray Imaging Microscopy and Microbeam with Fresnel Zone Plate and Quasi-monochromatic Undulator Radiation

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Fresnel zone plate (FZP) is widely used for imaging microscopy and for microbeam generation in hard X-ray region. Monochromatic X-ray beam is required for the FZP objective because of its strong chromatic aberration. However, the minimum monochromaticity required for FZP optics is equal to the total number of zone in order to achieve the diffraction-limited resolution. The bandwidth of crystal monochromator is usually around 10^{-4} that is much narrower than the required monochromaticity for FZPs. Undulator coupled with low-emittance storage ring provides quasi-monochromatic radiation with a bandwidth of about 1/100 that is well matched to the FZP optics. So, compared with conventional beamlines with crystal monochromator, the much higher flux is available by using the quasi-monochromatic undulator radiation. When it is applied to imaging microscopy, for example, an very short exposure time is expected.

We have performed imaging microscopy and microbeam experiment with FZP objective using quasi-monochromatic undulator radiation without any monochromators. The experiments were done at SPring-8 helical undulator beamline 40XU where the bandwidth of emitted X-ray beam is 1.2% at an X-ray energy of 8.3 keV. A tantalum FZP with outermost zone width of 0.25 micron and zone number of 100 was used as an objective.

An example of imaging microscopy experiment is shown in Fig. 1. This image was taken at an exposure time of 1.5 ms that is shorter than that in conventional beamlines by a factor of 1000. The spatial resolution of imaging microscope estimated by taking images of resolution test object is better than 0.5 micron [1]. In the microbeam experiment, the spot size of focused beam is measured to be 0.9 micron x 1.0 micron at an X-ray energy of 8.3 keV, and the photon flux density is measured to be 2 x 10^{12} photons/s/micron^2. Typical example of scanning microscopy experiments with this microbeam is shown in Fig. 2. Fine structures up to 0.5 micron are observable in the measured image.

1. Y. Suzuki et al., Rev. Sci. Instrum. **75** (2004) 1155.

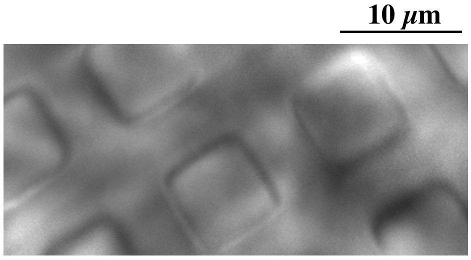

Fig. 1. Result of imaging microscopy.
Sample: copper grid mesh.
X-ray energy: 8.3 keV

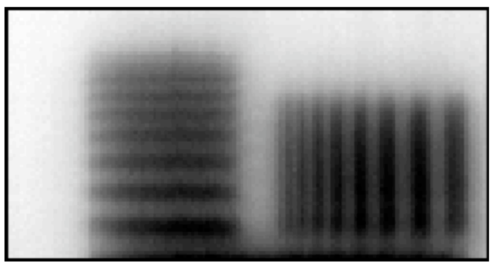

Fig. 2. Result of scanning microscopy experiment with FZP focusing optics.
Filed of view is 12.6 micron x 6.6 micron.
Sample: resolution test patterns.

X-ray Imaging Microscopy at 82 keV with Sputtered-sliced Fresnel Zone Plate Objective

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Fresnel zone plate (FZP) is widely used as an objective lens of X-ray microscope. Most of FZPs are fabricated using micro-fabrication technique developed for LSI technology (electron-beam lithography). However, these techniques are difficult to apply to fabrication of high-aspect-ratio structures. Therefore, the FZPs fabricated by electron beam lithography can not be used in high energy region (typically above 20 keV) because of low diffraction efficiency due to the thin zone structure.

Sputtered-sliced FZP (SS-FZP) fabricated by depositing concentric multilayer on a wire core is one of the optical elements that can be used in high energy region, because the SS-FZP has no-thickness limit in fabrication process. Recently, SS-FZP was applied to micro-focusing optics for high energy X-rays up to 100 keV, and spatial resolution (focused spot size) of 0.5 micron has been achieved [1, 2].

In this report, we describe X-ray imaging microscopy in high energy region (82 keV) using SS-FZP as an objective lens. The SS-FZP used in the experiment consists of 50 concentric Cu/Al multilayer deposited on a Au wire (50 micron in diameter). The outermost zone width is 0.25 micron, and the estimated thickness of the SS-FZP is about 36 micron. The focal length of the FZP is 1.63 m at 82 keV. The experiment has been done at BL20XU of SPring-8. The object and objective FZP were placed at the first experimental hutch located at 80 m from the light source, and a CCD-based imaging detector is placed at 245 m from the light source. Therefore, magnification of the X-ray optics is 100, in spite of long focal length (1.64 m) of the SS-FZP.

Measured image of a test object is shown in Fig. 1. A gold grid mesh with 1500 mesh/inch was used as the test object. The X-ray energy (82 keV) was selected so as to obtain the maximum absorption contrast by utilizing Au K-absorption edge (80.7 keV).

1. M. Awaji, et al., Rev. Sci. Instrum. **74** (2003) 4948.
2. N. Kamijo, et al., Rev. Sci. Instrum **74** (2003) 5101.

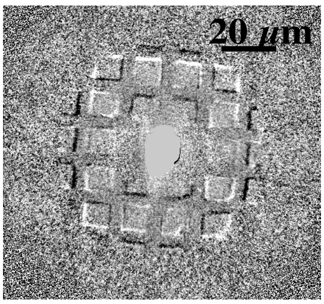

Fig. 1. Result of imaging microscopy at 82 keV with sputtered-sliced zone plate. Magnification of X-ray optics: 100. X-ray energy: 82 keV. Exposure time: 6 min.

Present Status of Hard X-ray Micro-Imaging at BL24XU of SPring-8

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In Hyogo beamline (BL24XU) of SPring-8, we are developing research programs using a hard x-ray microscope for applications to the wide range of science.

MICROSCOPE [1]: The apparatus mainly consists of three parts; one is an X-ray microscope unit, another is an optical microscope unit and the other is a sample stage unit. The two microscope units are interchangeable each other onto the beam axis with the high position repeatability better than 1.0 micron in the three-axis directions. In an X-ray microscope, we can select one of two zone plates (ZP1 or ZP2) for the proper purpose of experiments. The ZP1 has the outermost zone width of 250 nm and the tantalum thickness of 2.4 microns. The ZP2 has the outermost zone width of 50 nm and the tantalum thickness of 800 nm. The ZP1 is used mainly for high-energy experiments, while the ZP2 is used for experiments requiring higher spatial resolution in spite of less intensity. The sample stage unit consists of eight high precision stepping -motor-driven stages including θ-2θ goniometers. The θ goniometer has the very high eccentricity within ±0.25 micron/360°, and a high angular resolution of 0.72 arcsec/pulse can be achieved. The apparatus is also equipped with a SDD for X-ray fluorescence analysis.

PERFOREMANCE: The ZP1 provides the beam size of ~1 μm (10 keV) and ~2 μm (20 keV). The ZP2 provides ~300 nm (15 keV). By adopting a narrow slit in front of the ZP2, a microbeam with a relatively small horizontal angular divergence (~70 μsec) can be also available. This beam is used for strain analysis of various semiconductor devices. Because the diamond crystals used in the two upstream monochromators reduces the beam coherence, the diffraction limited beam size can not be obtained. By putting the ZP2 in BL20XU, where the ZP2 was put 200 m apart from a 20 μm pinhole, the almost diffraction limited beam size of 70 nm can be certainly achieved.

MICROBEAM APPLICATIONS: The microscope is used, for example, for measurements of strain distribution in laser diodes with a θ-2θ diffractometer and structural analysis of polymers with a diffractometer using imaging plate detectors. Scanning differential phase contrast microscope using a wedge absorber detector has been demonstrated [2].

MICRO-INTERFEROMETER [3]: Using the twin zone plate, the micro-interferometer has been successfully demonstrated. Three dimensional phase measurement has been done with the spatial resolution of about 250 nm.

References
[1] Y. Kagoshima *et al.*, in the proceedings of SRI2003 (AIP Conference Proceedings **705**, 2004), 1263.
[2] Y. Kagoshima *et al.*, Jpn. J. Appl. Phys. **43** (2004) L1449-L1451.
[3] T. Koyama *et al.*, Jpn. J. Appl. Phys. **43** (2004) L421-L423 and also presented in this conference.

Development of Scanning Hard X-ray Microprobe for Element-Specific Magnetic Imaging at SPring-8 BL39XU

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A scanning x-ray microprobe has been developed at SPring-8 BL39XU for x-ray magnetic circular dichroism (XMCD) measurement in the hard x-ray region with micron scale spacial resolution. This equipment is based on a micro-focusing method utilizing a Kirkpatrick-Baez (KB) mirror and the x-ray helicity switching technique using a diamond x-ray phase retarder[1] installed in BL39XU. The highly refined KB mirror was fabricated using the plasma chemical vaporization machining and the elastic emission machining[2]. This microprobe allows element-specific magnetometry and XMCD spectroscopy in a particular minute area of a sample. Element-specific magnetization mapping is also available. The intended photon energy is 5-16 keV. These high energy x-rays offer a deep probing depth, which is quite useful for exploring a buried magnetic layer in industrial samples of layered structures, such as magnetic storage media and magnetic random access memories. The following results were obtained through the development process:

1. Focusing property

The focused beam spot size of 1.2(vertical) micron $\times$ 1.0(horizontal) micron was achieved at 8 keV when the source size is minimized. The photon flux in the beam spot was 4.8×10^9 photons/s at that condition. A ten times more intense flux was obtained in a reasonable spot size of 2 micron square for a larger source size.

2. Magnetization mapping

For magnetization mapping, an XMCD image of a hard disk medium CoCrPtB, artificially in-plane magnetized with stripe patterns, was successfully obtained at Pt-L_3 edge (Fig. 1). The XMCD effect was $\sim$ 5 % of the total absorption, and the stripes of 2.4 micron width was clearly resolved.

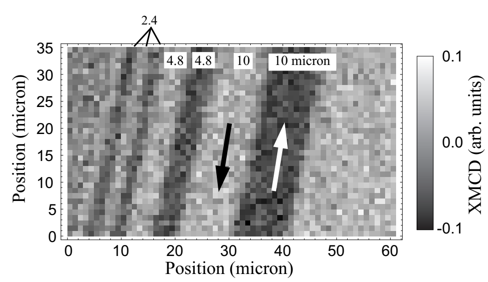

Fig 1: An XMCD image of CoCrPtB taken at Pt-L_3 edge in the fluorescence mode. The incident x-rays make an angle of 45 degrees with respect to the sample surface. In the image, numerals indicate the width of the stripe patterns, and the arrows are the direction of magnetization.

[1] M. Suzuki et al. Jpn. J. Appl. Phys. 37 (1998) L1488.

[2] K. Yamauchi et al. Jpn. J. Appl. Phys. 42 (2003) 7129.

3D-CT with MIRRORCLE-6X.

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The portable synchrotron MIRRORCLE-6X is a novel x-ray source suitable for hard x-ray imaging. The highly brilliant x-rays by MIRRORCLE-6X are generated in the shape of cone from the source point of micron size. The angle of radiation is ±85 mrad, which is determined by the kinematics 1/γ of electron beam in the storage ring. We use this machine for 3-dimentional computed tomography (3D-CT) with high magnification and high resolution.

Fig. 1 shows the 3D-CT system with MIRRORCLE-6X. We have developed the program to control the rotational stage and the flat panel detector (*PAXSCAN2520*, *VARIAN*) by the software, LaboView. This detector has an irradiation area of 240 mm wide × 192 mm height and 125 μm of the pixel size. In this system, when we set the sample at 0.5 m from the source point and the detector at 2.4 m, the projection data can be obtained in the maximum magnification rate, 4.8 times, for samples of 50 mm wide. Consequently, the spatial resolution of about 30 μm is obtainable. The necessary exposure time is 5 minutes at full power operation. As the result, the microscopic 3D images can be obtained by the reconstruction calculation of projection data. The 3D-CT with MIRRORCLE-6X is useful for nondestructive inspection and biological study of small animals.

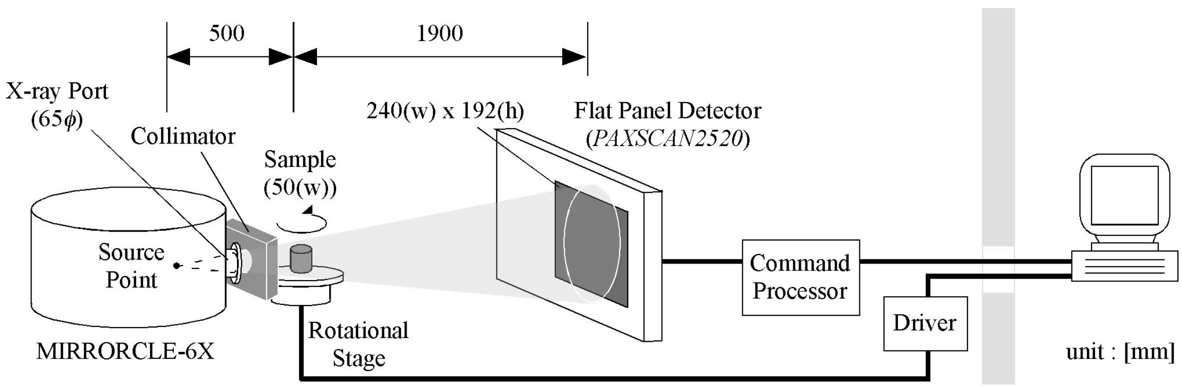

Fig.1. 3D-CT system with MIRRORCLE-6X for seeing 50 mm wide samples.

Precision Mechanical Design for a Hard X-ray Nanoprobe Instrument with Active Vibration Control in Nanometer Scale*

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We are developing a new hard x-ray nanoprobe instrument, which is one of the centerpieces of the characterization facilities of the Center for Nanoscale Materials (CNM) being constructed at Argonne National Laboratory (ANL). This new probe will cover an energy range of 3-30 keV with 30 nm special resolution [1]. Imaging and spectroscopy at this resolution level require staging of x-ray optics and specimens with a mechanical repeatability of better than 10 nm.

Fast feedback for d ifferential vibration control between the zone-plate x-ray optics and the sample holder has been implemented in its design using a DSP-based real-time closed-loop feedback technique. A specially designed, custom-built laser Doppler displacement meter system provides two-dimensional differential displacement measurement with subnanometer resolution between the zone-plate x-ray optics and the sample holder. The entire scanning system was designed with high stiffness, high repeatability, low drift, and flexible scanning schemes.

Precision mechanical design of the hard x-ray nanoprobe, as well as test results from an “Early User Instrument”, which we have developed to test a novel two-dimensional interferometrically controlled scanning stage system [2], are presented in this paper.

References:

1. J. Maser, G. B. Stephenson, D. Shu, B. Lai, S. Vogt, A. Khounsary, Y. Li, C. Benson, G. Schneider, SRI 2003 Conf. Proc., 705, AIP (2004) 470-473.
2. D. Shu**,** J. Maser, B. Lai, and S. Vogt, SRI 2003 Conf. Proc., 705, AIP (2004) 1287-1290.

Key words: scanning stage, differential measurement, active vibration control.

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Hard X-ray Microscopy and its Applications at Shanghai Synchrotron Radiation Facility

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Abstract

A beamline for hard X-ray microscopy is now under construction at Shanghai Synchrotron Radiation Facility (SSRF) and will be commissioned in 2009. Imaging modalities including microtomography, diffraction-enhanced imaging and traditional radiography are to be developed. Imaging mechanisms such as phase contrast and absorption contrast will be employed. The scientific cases and current status of the beamline are to be introduced. An in-house X-ray phase contrast imaging system has been developed at SSRF. The facility and its applications in biomedicine will be brought out. Investigations on X-ray imaging methodology and corresponding applications, carried out at BSRF, Photon Factory and Elettra, will also be introduced.

Scanning hard-X-ray microscope with spatial resolution better than 50nm using K-B mirror optics

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Intensive nanofocused X-ray beams are necessary for enhancing performances in various types of X-ray microscope. X-ray focusing techniques using K-B mirrors are promising in terms of highly efficient and energy-tunable focusing. We have been developing a hard-X-ray focusing system in which the obtainable focus size is less than 100nm. In this presentation, we will report the development of a mirror manipulator and demonstrations of the scanning X-ray microscope. Not only figure accuracy of mirrors but also precise mirror alignments are required for ideal focusing. Alignment tolerances in mirror adjustments such as glancing angles and the perpendicularity between two mirrors were estimated using two types of simulator[1]: a geometrical-optical simulator and a wave-optical simulator. The mirror manipulator was designed and constructed on the basis of the simulation results that the glancing angle and the perpendicularity should be set with ±0.9μrad and ±40μrad accuracies, respectively. In the manipulator, the perpendicularity between two mirrors can be adjusted without X-ray beams by the tilt monitor system using two autocollimators before measuring intensity beam profiles. The glancing angles can be adjusted with an angular resolution of 0.2μrad and no backlash while measuring intensity beam profiles simultaneously. At the 1-km-long beamline (BL29XUL) of SPring-8 the focal size, defined as the full width at half maximum in the intensity profile, was achieved to be 48 x 36nm^2 (V x H) by this manipulator with K-B mirrors having a figure accuracy of 2nm peak-to-valley height[2]. As a result of spatial resolution tests using tantalum test patterns, the scanning X-ray microscope with the focus system could resolve the line-and-space patterns of 80nm line width in a high visibility of 60%. We are planning trace element mapping in cell organelles with a high resolution and a high sensitivity using X-ray fluorescent analysis.

1 S. Matsuyama et al., Proc. SPIE 5533, 181 (2004).
2 H. Mimura et al., Jpn. J. Appl. Phys. submitted.

Combinatorial XAFS Imaging: Application to efficient screening of CO_2 absorbent

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X-ray imaging system, which does not need any scans of sample or X-ray beam and therefore dramatically reduces the amount of time required [1], was employed to evaluate combinatorial libraries [2] efficiently. Combinatorial substrate of CO_2 absorbent, lithium ferrite $LiFeO_2$ was prepared, which has 4x4 array of the ferrites synthesized at different temperatures and exposed to CO_2. 2-D XRF (X-ray fluorescence) images of 8 mm ×8 mm area were observed for the substrate by the exposure time of only 3 sec using synchrotron X-rays from BL-16A1, KEK-PF. Thus XRF signals from a whole substrate could be observed at once in a short space of time. In order to see chemical change accompanied by CO_2 absorption simultaneously for each ferrite synthesized at different temperature, fluorescent XAFS (X-ray absorption fine structure) was measured by repeating the imaging during the monochromator scans across the absorption edge for iron. From the amount of spectral change for each ferrite, the performance as CO_2 absorbent could be evaluated, and the best ferrite was selected [3].

This screening procedure is extremely efficient because XAFS spectra for all materials put on the common substrate are obtained from only single energy scan. One can determine the valence numbers and other chemical environment of the metal included in each material, from the differences in spectral features and the energy shifts. Hence combinatorial libraries can be screened very rapidly therefore efficiently using the X-ray imaging system.

[1] K. Sakurai and H. Eba, Anal. Chem. 75 (2003) 355.

[2] X. –D. Xiang, X. Sun, G. Briceno, Y. Lou, K. –A. Wang, H. Chang, W. G. Wallace-Freeman, S. –W. Chen, and P. G. Schultz, Science 268 (1995) 1738; G. Briceno, H. Chang, X. Sun, P. G. Schultz, and X. –D. Xiang, ibid. 270 (1996) 273.

[3] H. Eba and K. Sakurai, Mater. Trans. 46 (2005) 665.

A High Resolution Hard X-ray Imaging Facility at SSRL

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The Stanford Synchrotron Radiation Laboratory (SSRL) in collaboration with Xradia Inc., the NASA Ames Research Center and Cornell University plans to implement a commercial hard x-ray full field imaging microscope on the 54 pole wiggler beam line at SPEAR3. This facility will provide unprecedented analytical capabilities for a broad range of scientific areas and will emphasize research on nanoscale phenomena and structures in materials science, environmental science, and biology. The instrument itself will be a full-field transmission microscope (TXM) based on zone plate optics. This instrument will enable high resolution x-ray microscopy, tomography, and spectromicroscopy in a photon energy range between 3–14 keV. The spatial resolution of the TXM microscope is specified as 20 nm exploiting imaging in third diffraction order. It will be shown that this imaging facility will optimally combine the latest imaging technology developed by Xradia Inc. with the wiggler source characteristics at beam line 6-2 at SSRL. This will result in an instrument capable of high speed and high resolution imaging with spectral tunability for spectromicroscopy, element specific and Zernicke phase contrast imaging.

Furthermore, a scanning microprobe capability will be integral to the system thus allowing elemental mapping and fluorescence yield XANES to be performed with a spatial resolution of 1 μm without introducing any changes to the optical configuration of the instrument.

State-of-the-Art X-ray Tomography Imaging Using Laboratory Sources

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X-ray computed tomography (XCT) offers powerful non-destructive three dimensional imaging capability that is widely used in diverse fields, including medical diagnosis, biomedical and material research, geology, petrology, and archeology. This powerful imaging capability results from many intrinsic and desirable properties of x-rays: short wavelength for high-resolution imaging, high penetration power for imaging interior structures of large and optically opaque objects, and unique elemental specific interaction for elemental specific imaging and spectroscopic imaging. Since its invention, the capabilities of XCT have evolved rapidly in terms of high-resolution x-ray imaging optics and development of phase contrast imaging. For example, the resolution of commercially available systems has improved from about 1 mu to sub-60 nm. Xradia has developed two product families of 3D x-ray tomography imaging systems for the semiconductor industry: one with sub-60 nm for die level imaging (nanoXCT) and another with micrometer scale resolution for package level imaging (MicroXCT). The nanoXCT operates at an x-ray energy of 5.4 keV and is used to produce transmission images of copper IC's with good contrast and a resolution of better than 60 nm. These images, collected in a few minutes, reveal the internal structure of the IC without destructive cross sectioning and in an ambient air environment. Images of electromigration test structures have revealed the progressive failure of thin, buried copper interconnects and vias. The tool is a powerful adjunct to other diagnostic techniques that require localization of defects, e.g., in the preparation of TEM samples and it may supplant the SEM in some imaging applications. Unlike projection x-ray imaging equipment, the resolution of the x-ray microscope used for these measurements is determined by the imaging x-ray lens, not by the size of the x-ray source. The lens is a circular diffraction grating configured as a simple zone-plate, having a resolution of about 60 nm and a focusing efficiency above 10%. The MicroXCT system operates at high energy x-rays that has sufficient transmission through a fully packaged IC device. Similar to the nanoXCT, the microXCT is used to either image defective structures perform reverse engineering of the packaged IC device. The performance of Xradia's two x-ray imaging systems will be presented and their applications discussed.

Hard X-ray mirrors by multilayer replication: developments and application

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In order to extend the energy range of hard X-rays to handle, multilayer coatings have been introduced in many fields. For astrophysical observations, we have developed Pt/C multilayer, with graded density d-spacing, so called “Super mirrors”, which covers broad energy band up to 60 keV. Here broad band is essential, because line energies are sometimes red-shifted by Cosmological expansion of the Universe.

Another key element of mirrors of X-ray telescopes is high throughput to collect as many photons as possible within sever weight budget of the payload to be onboard a satellite. Since the incident angle of X-ray reflection ranges below 1 degree and should be smaller for higher energies. Therefore mirror substrate has to be much thinner than the pitch of the nested mirror shells, that is about 1 mm. It is impossible to polish such thin mirrors and then we introduced replica mirrors from mandrels of extremely smooth surfaces. On the mandrel, usually gold layer had been deposited as a separation agent. In these days, multilayers deposited on glass mandrel can be copied on thin substrate of 0.1-0.2 mm thick.

The combination of replication technique and multilayer coating allow us various possibility of application, such as hard X-ray microscopes. Though it might be almost impossible to deposit multilayers on the inner surfaces of narrow cones of microscopes, it is rather easier to deposit them on outer mandrel and then to replicate them on the substrate. There might be some technical issues to be solved but seems promising to enhance hard X-ray response of microscopes. In this paper, we intend to present current status of the hard X-ray multilayer replica mirrors being developed in our group and to exchange knowledge and requirements with expert of microscopes.

Multilayer Fresnel Zone Plate at AIST and SPring-8
----- high diffraction efficiency, high-energy x-ray region -----

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A multilayer (sputtered-sliced) Fresnel zone plate (FZP) is one of promising focusing optics with high spatial resolution for high energy X-ray region, especially over 20 keV, because a large "aspect ratio" (the ratio of the FZP thickness to zone width) can easily be realized [1,2]. In addition, it is highly important that the kinoform type zone plate (the theoretical diffraction efficiency is 100 %) can be fabricated by the sputtered-sliced method (Fig.1).

A higher diffraction efficiency is indispensable as well as a higher spatial resolution. In order to realize higher focusing efficiency, a multilevel-type [3] (4-step: quasi-kinoform type) multilayer FZP with the diameter of 70 micron has been fabricated (Fig.2). Such a FZP was composed of concentric multilayer of alternating high-Z (Cu), low-Z(Al) and composite materials. The focusing test of the FZP was performed at the BL20XU undulator beamline of SPring-8. The measured diffraction efficiency has been more than 45 % around 50 keV, which exceed the theoretical limit of the phase FZP.

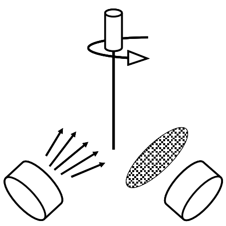
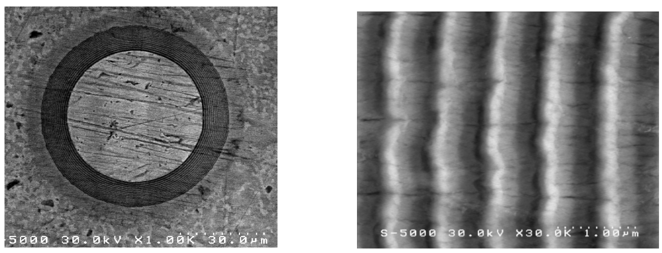


Fig.1 DC sputtering apparatus



(a) (b)

Fig.2 Scanning electron micrographs of Cu/Al 4-step multilayer FZP : (a) Full view, (b) its close-up view.

[1] N.Kamijo, S.Tamura, M.Yasumoto et al: Review of Scientific Instruments、74 (2003.12)5101.
[2] S.Tamura, M.Yasumoto et al.: Journal of Synchrotron Radiation, 9 (2002.5)154.
[3] E Di. Fabrizio et al.: Nature 401(1999)895.

Optical properties of two-lens system on the base of hard X-ray zone plates

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We present results of study of focusing and imaging properties of double-lens system for hard x rays, consisting of such elements as Fresnel zone plates (FZP) made from silicon. As it was shown practically, such FZP has advantages like small absorption, high efficiency and high spatial resolution.

This work demonstrates for the first time a phenomenon of focusing x-ray beam by two FZPs mounted with sufficiently large distance between them. The geometrical point of focus is the same as for the system of two thin refractive lenses. The peculiarities of redistribution of intensity within the focal plane when the second FZP is moved across the optical axis are investigated both experimentally and theoretically (computer simulation). It is shown, the shift of the second FZP leads to a moire pattern, which allows one to adjust FZPs with accuracy up to 50 nm. The intensity distribution along the optical axis is also investigated.

We realise for the first time an image transfer by means of double-lens systembased on FZP. The experiments are performed in ESRF, BM-5 beamline with the x-ray energy 9.4 keV.

We elaborate a computer program for theoretical study of double-lens system based on FZP. The program allows simulation all properties of such system with limited number of zones. The calculation is based on the use of Kirchhoff propagator in a paraxial approximation and fast Fourier procedure. The intensity map inside the plane across the optical axis is calculated for any parameters, including the shift of the second FZP across the optical axis.

Progress of the Fabrication of Soft X-ray Phase Zone Plates at NSRL

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Soft X-ray condenser zone plate is a key condensing and dispersing element in the field of soft x-ray microscopy. The phase condenser zone plates used at the experimental station of soft x-ray microscopy, NSRL (National Synchrotron Radiation Laboratory, Hefei, China) are fabricated. The zone-plate is operated at wavelength between 3.2nm, whose diameter is 2.8mm. The width of its outermost zone is 647nm. The zone plate material is Ge or Ni. The phase zone plate with substrate of Si_3N_4 substrate is fabricated using an x-ray lithographic process and an ion beam etching process or a reactive ion etching process. The zone plate mask is fabricated using holography-ion beam etching and is a condenser amplitude zone plates with polyimide substrate. Zone plates are used under the conditions of high radiation. To overcome the degradation of polyimide by x-ray radiation, we substitute Si_3N_4 for polyimide as zone plate substrate. The x-ray lithography is performed at the x-ray lithography experimental station of NSRL. The optical characteristics of phase zone plates are measured at the soft x-ray microscopy experimental station of NSRL.

Key Words: soft x-ray, condenser zone plate, phase zone plate, fabrication, x-ray microscopy

A Compound Refractive Lens for 175-keV for Magnetic Compton Profile Measurements at SPring-8

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Despite the high intensity of circularly polarized radiation of energy 175 keV at SPring-8 beamline BL08W, for samples with small volume (for example thin layers or material in high pressure cells) the intensity of radiation scattered to the detector in Magnetic Compton experiments is low. Moreover, the sample holder generates a relatively high background. By focusing the radiation it is possible to increase signal from the sample while simultaneously decreasing the background from the holder.

Because, in our case, the focusing is need in the vertical plane only, the planar Compound Refractive Lens (CRL) [1], [2] and Multi Prism Lens (MPL) [3] are considered as the focusing element. They are able to give a focal spot of micrometer size and they are easy to add to an existing setup.

A Monte Carlo program has been written for simulations of plane geometrical optics for X-rays. With this software the performance for different lens parameters was tested. Fig.1 shows example comparison of performance of prism and parabolic lens for 1 micrometer source.

For first tests the 250 steel single parabolic lenses with radius of curvature at vertex of 100 microns will be stacked to form a lens 15 cm long with a focusing length of 4m. Test measurements with a prototype lens are scheduled for this year.

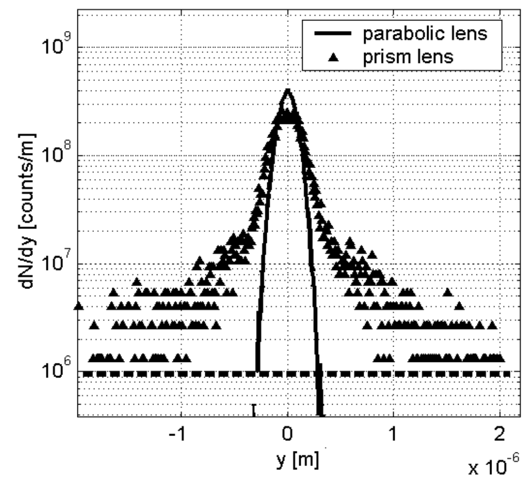

Fig. 1 *The simulated intensity distribution in vertical direction at focus position The focal length of both lenses is 4m The 1 micrometer source is situated 47m far from the lenses. The solid line and triangles represent data for parabolic and prism lenses respectively. The horizontal dashed line shows the intensity at sample without lenses.*

[1] Snigirev A.; Kohn, V.; Snigireva, I.; Lengeler, B., *Nature*, 1996, **384**, 49.
[2] Lengeler, B.; Schroer, C.G.; Benner, B.; Gerhardus, A.; Günzler, T.F.; Kuhlmann, M.; Meyer, J.; Zimprich, C., *J. Synchrotron Rad.,* 2002, **9**, 119.
[3] Cederstrom, B.; Lundqvist, M.; Ribbing, C., *Appl. Phys. Lett.,* 2002, **81**, 1399.

Hard X-ray Microbeam and Scanning Microscopy using Fresnel Zone Plate with 50 nm Outermost Zone Width

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Fresnel zone plate (FZP) with 50 nm outermost zone width has been fabricated, and tested at SPring-8 medium-length-beamline 20XU. The FZP was fabricated by electron-beam lithography at NTT Advanced Technology. The zone material is tantalum with a thickness of 0.5 micron. The diameter of the FZP is 250 µm, and the focal length is designed to be 80 mm at an X-ray energy of 8 keV. Focused beam profile for the first-order diffraction measured by knife-edge scan is shown in Fig. 1. Focused beam size defined as full-width at half-maximum (FWHM) is 58 nm that is very near to the diffraction-limited resolution. The measured diffraction efficiency for the first-order diffraction is 5% at 8 keV. Scanning microscopy experiment was also done for the purpose of confirming the spatial resolution. An example of measured image is shown in Fig. 2. The test object is resolution test patterns with 70 nm line and space. The nanometer-scaled structure is clearly observed in the measured image. Focused beam profiles for the third order diffraction was also tested for investigation of ultimate performance of the FZP. The measured spot size was 31 nm at an X-ray energy of 8 keV [1]. We consider that this value is present technical limitation of FZP fabrication, while the theoretical limit of spatial resolution of FZP is about 10 nm in the X-ray region [2]

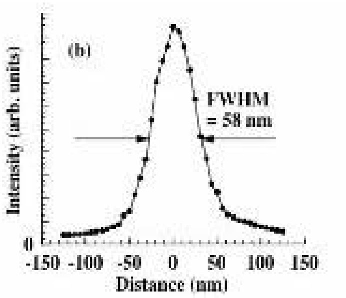

Fig. 1. Focused beam profile measured by knife-edge scan. X-ray energy: 8 keV.

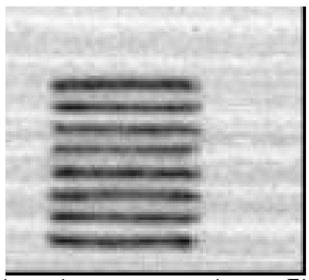

Fig. 2. Scanning microscopy experiment. Field of view: 950 nm x 950 nm, X-ray energy: 8 keV, sample: resolution test pattern

1. Y. Suzuki et al., Jpn. J. Appl. Phys. in press.
2. Y. Suzuki, Jpn. J. Appl. Phys. **43** (2004) 731.

High-resolution Zone Plates Made with Wideband Extreme-Ultraviolet Holography

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Fresnel zone plates (FZP) made with electron-beam lithography (EBL) are used in x-ray microscopy applications providing resolution down to about 20 nm. The fabrication of high-resolution FZP with EBL poses certain challenges including zone placement errors due to thermal drift, resolution limitations due to the proximity effect and low-throughput due to the serial writing scheme. We have developed a new holographic fabrication technique using extreme ultraviolet radiation in order to address these challenges. In the scheme shown in Figure 1, a mask bearing two concentric FZP is illuminated by a spatially coherent EUV beam. The two FZP on the mask are written with EBL and they have the same outermost zone width. At a certain distance from the mask the diverging and converging spherical beams created by the mask overlap and form an interference pattern, which is suitable for recording a “daughter” FZP with half the zone-width of the “parent” FZP on the mask. This technique is achromatic since the two interfering beams travel equal optical path lengths before arriving at the image plane. Analytical and simulation results showing the formation of the FZP pattern was confirmed experimentally with the production and testing of a lens with 60 nm outermost zone width. Holography with extreme ultraviolet light has the potential to produce lenses with sub-10 nm resolution.

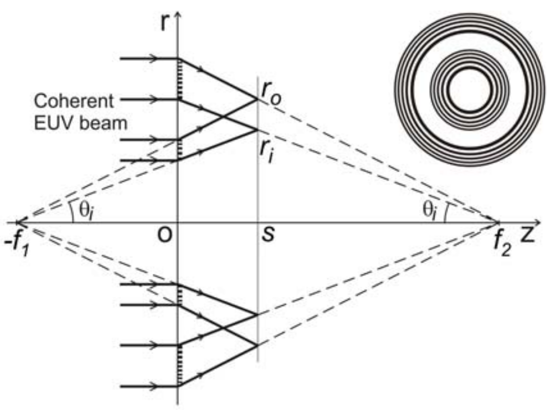

Fig. 1. Achromatic holography scheme for recording a FZP. A schematic frontal view of the mask is shown in the upper right.

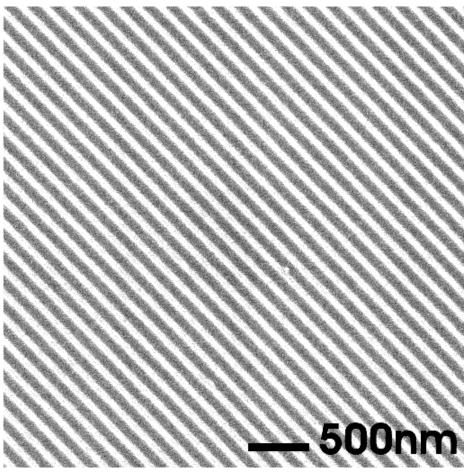

Fig 2. SEM image of 66 nm wide zones printed with EUV holography and etched into a Cr film.

The development of hard x-ray optics for MIRRORCLE-6X microscope beam line

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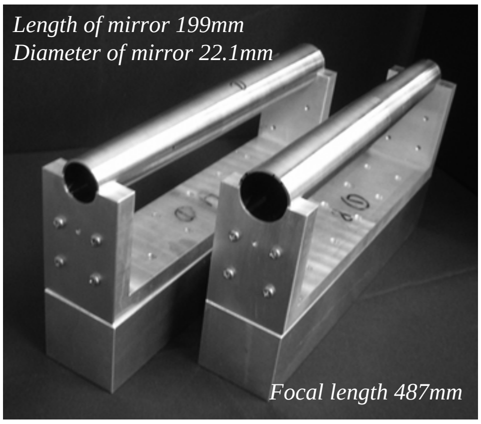
A laboratory-scale hard x-ray microscope based on the portable synchrotron named MIRRORCLE-6X is developed at SLLS. MIRRORCLE-6X is a novel x-ray source suitable for hard x-ray microscopy because of its x-ray source size having the order of micron, and its high brilliance. We have developed Wolter type-I mirrors using grazing incident optics. For focusing X-rays into sub-mm size we use a set of two identical Wolter mirrors. Each is shaped in an axially symmetric hyperboloid and paraboloid surface. One mirror reflects incident x-rays at large angles and transforms them into parallel beam. The second Wolter type mirror placed along the optical axis focuses these parallel x-rays to the point. Diverging X-rays are focused after 4 times-reflections with two mirrors.

We manufactured Wolter type mirror by replication method. Nickel electroforming was applied to the master mandrel made from oxygen-free copper. The smooth surfaces were manufactured by super precision machining. The surface roughness of master mandrel is made within 1nm r.m.s. We could perform the successful replication between Nickel and oxygen-free copper without losing surface roughness. Figure.1 shows the fabricated set of Wolter type grazing incidence mirrors. We have observed 90% reflectivity for 2-8keV x-rays with one aspheric surface of this mirror. We have observed the focused spot of X-rays from MIRRORCLE-6X. We will demonstrate microscopy images at the conference, although our final goal is a deposition of multiplayer to these Walter type mirror.



Investigation of Soft X-ray Multilayer Mirror Fabrication by an *In-Situ* Ellipsometric Deposition Monitor

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Soft X-ray multilayer mirrors are key components for high resolution imaging in soft X-ray microscope and other applications. For high reflectance at wavelengths of a few to several tens nm, the multilayer has to be fabricated at optimized condition for the smoothest interface at every boundary.

To investigate the layer structure during ion beam sputtering fabrication of Mo/Si multilayers, we have applied our *in-situ* automatic null ellipsometer [1] with rapid layer-by-layer analysis [2]. Figure 1 shows two sets of *in-situ* data observed at Ar ion acceleration voltages of 1400 V and 900 V. The ellipsometric data of the complex relative amplitude attenuation were recorded at every 150 msec and plotted on a complex plane as growth curves. A growth curve of each period is composed of a Mo segment and a Si segment up to the final layer. At the latter stage of fabrication, the growth curve should form a closed-loop as depicted in Fig. 1. This is because the total thickness of the multilayer exceeds the penetration depth of the incident He-Ne laser light.

Direction and length of each segment on the complex plane represent the optical constants and layer thickness, respectively. Therefore, the growth curves visualize differences of layer properties.

Judging after the growth curve properties [3], Mo forms an island structure on Si at 1400 V growth in the early stage just after a target switching. In contrast, a silicide layer is likely to form on Si with no Mo island growth at 900 V. These information could be essential to study for ideal homogeneous and isotopic layer growth.

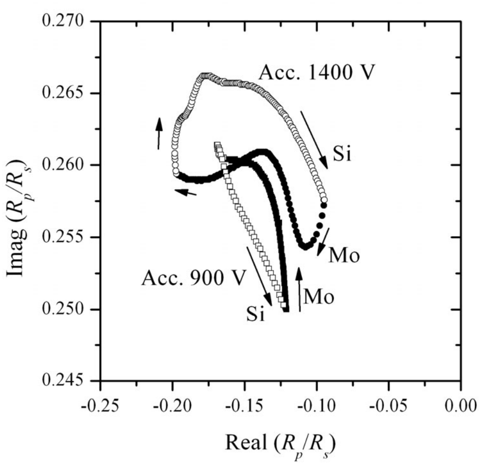

Fig. 1 Ellipsometric growth curves of Mo/Si multilayer at the 30th period measured at acceleration voltages of: 1400 V (circles) and 900 V (squares). Sputtering durations of Mo and Si were for 240 sec and 280 sec, respectively.

References

[1] M. Yamamoto, Y. Hotta, and M. Sato, Thin Solid Films **433** (2003) 224
[2] T. Tsuru, T. Tsutou and M. Yamamoto, Thin Solid Films **455-456** (2004) 705
[3] M. Yamamoto, T. Namioka, Appl. Opt. **31** (1992) 1612

The Hard x-ray Nanoprobe Beamline at the Advanced Photon Source

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The hard x-ray nanoprobe beamline at sector 26 of the Advanced Photon Source is designed to characterize nanoscale systems and devices at a spatial resolution of 30 nm, using x-ray fluorescence spectroscopy, x-ray diffraction, and transmission imaging.[1] X-ray fluorescence will provide element-specific imaging with sensitivity to individual nanoparticles embedded in thick specimens. X-ray diffraction and scattering will probe strain state and ordering of nanoscale systems and their environment. Transmission imaging will allow visualization of thick specimens and devices in 3D. To allow x-ray fluorescence spectroscopy of most elements in the periodic system, the beamline will deliver x-rays with photon energies between 3 keV and 30 keV. Two collinear insertion devices with a period of 3.3 cm and a combined length of 4.8 m are used as source of x-rays. This maximizes the coherent x-ray flux available in the nanoprobe instrument. The beamline optics are designed to allow two modes of operation: a scanning probe mode, where the spatially coherent fraction of the undulator beam is focused by a high-resolution x-ray optic on a small specimen area, and a full-field transmission mode, where the full, partially coherent undulator beam is used to allow transmission imaging at high resolution.

The nanoprobe instrument will use Fresnel zone plates as high-resolution x-ray optics. The system is being designed to ultimately accept zone plates providing a spatial resolution of 10 nm. To understand how to achieve the required mechanical stability of 5 nm, we are performing experiments with an early version of the nanoprobe instrument.[2] The zone plate is driven by a custom flexure stage, and will be scanned for data acquisition at high spatial resolution. The specimen stage is used for positioning and coarse scans only; it will provide x/y/z positioning as well as a single degree of rotation for tomography and microdiffraction. High precision positioning is achieved by measuring the position of both zone plate and specimen stages in x and y with individual laser Doppler interferometers with respect to a reference frame. Deviations of the position from the desired values are corrected using the flexure stage. A feedback loop operating at a frequency of 100 Hz provides active vibration control. First measurements of the system performance at have yielded a source-size limited resolution of 70 nm at a photon energy of 7.4 keV, and a noise level of below 30 nm.

We will present the overall beamline concept, as well as first data from the nanoprobe instrument.

References

[1] J. Maser, G.B. Stephenson, D. Shu, B. Lai, S. Vogt, A. Khounsary, Y. Li, C. Benson, G. Schneider, *AIP Conf. Proc.* **705**, AIP (2004) 470 – 473.

[2] D. Shu, J. Maser, B. Lai, and S. Vogt, *AIP Conf. Proc.* **705**, 1287-1290 (2004).

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Development of focusing optical system for 6 nm X-ray

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The development of novel technologies for lithography, spectroscopy and microscopy is currently being envisaged by various researchers. These schemes require highly efficient focusing X-ray optics and narrow bandwidth, high brightness soft X-ray source. We have constructed a micro-XPS system with line-focused laser plasma X-ray source and have demonstrated its performance of X-ray photo electron microscope using 13 nm X-ray[1]. It is desirable to introduce shorter wavelength X-ray source for own system to extend its applicability. In this system, it is necessary an efficient focusing optics for short wavelength X-ray.

The schematic of the experimental setup is shown in Fig.1. A Nd:YAG laser system operates with a repetition rate of up to 10 Hz and a maximum energy of 3 J. The X-ray source consists of segmented lens system, tape target and debris shield. Emitted X-rays go through a 0.1 micron thickness Al film and a pinhole (100 micron diameter), and impinge into the focusing X-ray optics system. An elliptic mirror coated with Au layer was set on the beamline at a distance of about 1m from the pinhole. The size of the microbeam was measured using the knife-edge method. The knife edge was set on a micro-motor stage. X-rays after the knife-edge were detected by a microchannel plate (MCP) with phosphor screen. The visible image on the screen was recorded by a CCD camera.

We measured the variation of the microbeam size with respect to the position of pinhole vertical and along the optical axis and the distance of the knife from the mirror exit. Comparisons between experimental results and calculations were made, and it was found a good agreement. The minimum spot size measured in this experiment was less than 5 micron.

On the other hand, we are constructing a focusing optics by using Schwarzschild mirror for 6 nm X-ray.

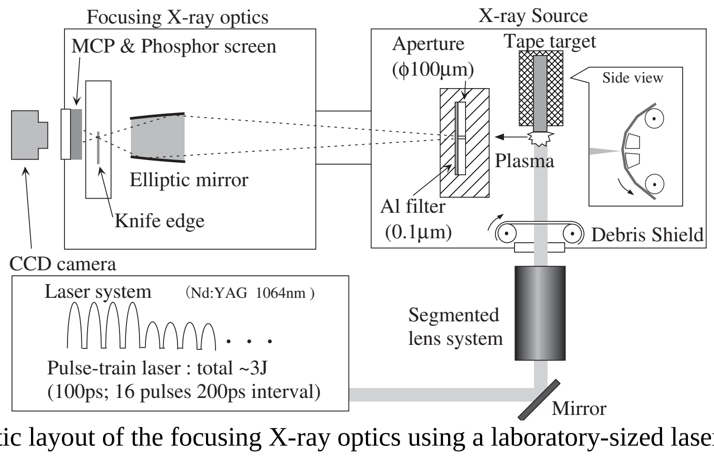

Figure 1. Schematic layout of the focusing X-ray optics using a laboratory-sized laser-plasma source.

[1]Y. Nishimura, Z. Takahashi, A. Sakata, H. Azuma, N. Yamaguchi and T. Hara, Rev. Laser Engineer. 32 (2004) 799.

Development of a Soft X-ray Telescope with an Adaptive Optics System

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We are developing a soft x-ray telescope with an adaptive optics system for future astronomical observation with very fine angular resolution of an order of milli-arc-second. From a technical point of view, we are trying to develop a normal incident telescope with multi layers. Thus the wave length is limited to be around 13.5 nm with a band pass of roughly 10nm. Since the x-ray telescope must be installed on a satellite, a stable conditions of temperature, gravity etc, can not be expected. Therefore, we investigate to use an adaptive optics system using an optical light source attached in the telescope. In this paper, we report our present status of the development.

The primary mirror is an off-axis paraboloid with 80 mm effective diameter and 2 m focal length. This mirror has been coated with Mo/Si multi-layers. The reflectivity of the 13.5 nm x rays is ranging from 35% to 55%. We use a deformable mirror for the secondary mirror, which has been coated with Mo/Si multi-layers. This mirror consists of 31 element-bimorph-piezo electrodes. The surface roughness of the mirror is ~6 nm rms. The reflectivity of the 13.5 nm x rays are roughly 65%[1].

The adaptive optics system using an optical laser and a wave front sensor has been performed. We are using a shack-hartmann sensor (HASO 32) with a micro-lens array and a CCD. A pin hole with one micron diameter is used for the optical light source. The precision of the measurement of the wave front shape is a few nm.[2][3]

X-ray exposure test has been achieved, although the optical adaptive optics system has not yet be installed. The x-ray detector is a back illumination CCD. The quantum efficiency for 13.5 nm x ray is ~50%. The pixel size is 24 micron square. X-ray source is an electron impact source. We confirmed the x-ray intensity around 13.5 nm region is bright enough for our experiment. The imaging performance is now trying to improve and the adaptive optics system will be installed in this year.

References

[1] S. Kitamoto, N. Yamamoto, T.Kohmura, K. Suga, H. Sekiguchi,, Y. Ohkawa, J, Kanai, S. Chiba, H. Sato, K. Sudo, and T. Watanabe, Proceedings of SPIE, 5488, (2004), 460-467.
[2] S. Kitamoto, H. Takano, H. Saitoh, N. Yamamoto, T. Kohmura, K. Suga, H. Sekiguchi, Y. Ohkawa, J. Kanai, S. Chiba, Proceedings of SPIE, 5169, "Astronomical Adaptive Optics Systems and Applications", (2003), 268-275,
[3] S. Kitamoto, H. Takano, H. Saitoh, N. Yamamoto, T. Kohmura, K. Suga, H. Sekiguchi, Proceedings of SPIE, 5037, (2003), 294-301,

Hard X-ray mapping and microscopy with lithographic CRL developed at ANKA Synchrotron radiation facility

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At ANKA Karlsruhe, deep X-ray lithography in SU-8 resist is being successfully applied for the production of efficient compound refractive polymer lenses for hard X-rays. The SU-8 polymer is characterized by low X-ray absorption and high irradiation resistance. The lithographic technique allows arbitrarily shaped structures (parabolas, segments, kinoform) together with extreme small radii, so lenses with very short focal lengths of a few cm and comparatively large effective apertures can be produced [1,2]. Enabling very large demagnifications the lenses focus synchrotron radiation with energies between 5keV to more than 30keV into sub-µm spots with very high intensity gain. Test and characterisation results of the lenses at ANKA and at the ESRF will be presented together with examples from applications in micro probe X-ray spectroscopy and magnified X-ray imaging [3].

1 V. Nazmov *et al*. Microsystem Technologies, 10 (2004) 716
2 V. Nazmov *et al*. AIP conference proceedings, 705(2004) 752
3 V. Nazmov *et al.* Proceedings of SPIEE, 5539 (2004) 235

Fabrication and characterization of Depth-graded X-ray multilayers

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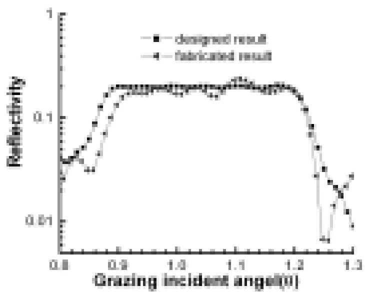
Depth-graded X-ray multilayer structures (also called supermirror), including nanometer scale layers, have currently been developed to provide broadband reflectivity for a variety of applications including synchrotron radiation and medical optics, and in particular for space-borne astronomical hard X-ray telescopes above 20keV. In the depth-graded X-ray multilayer, the layer thicknesses vary with depth into the film (in contrast to a periodic X-ray multilayer), so that the incident wavelength will penetrate deeply into the stack with minimal loss and reflect efficiently from as many interfaces as possible. The aim that we design depth-graded W/B_4C multilayers is to have a high and flat reflectivity in the grazing incident angle range of 0.9-1.2° at the wavelength of 0.154nm by using the Kozhevnikov's design method. The depth-graded W/B_4C multilayers were fabricated by using a high vacuum DC magnetron sputtering coater model JGP560C6 made in China. The base pressure is less than 8×10^{-5}Pa in the process of deposition. The working gas is Ar gas, the purity of which is larger than 99.99%, and the working pressure is 0.27Pa. The distance between targets and substrates is 80mm. The power of W and B_4C target are 20W and 130W respectively. The deposition rate of W is 0.47nm/s，and the one of B_4C is 0.32nm/s at above condition. According to the deposition rate, the computer controls the time needed to stay over each target for making depth-graded multilayers. The depth-graded X-ray multilayers fabricated by us were characterized by X-ray reflectivity measurements on a laboratory X-ray diffractometer . The angular resolution of the diffractometer is ~0.004°. Figure 1 shows the specular reflectivity around the first Bragg maximum of the samples as a function of the incident angle at the wavelength of 0.154nm. The Figure shows how the experimental result is close to the design.



Keywords: depth-graded multilayers, magnetron sputtering, X-ray diffractometer

Fabrication of the beam splitters for 13.9nm x-ray laser applications

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The x-ray laser Mach-Zehnder interferometer is an important tool to measure the electron densities of a laser-produced plasma near the critical surface. The design of a multilayer beam splitter at 13.9nm for soft x-ray laser Mach-Zehnder interferometer is completed based on the standard of designs of maximizing product of reflectivity and transmission of the beam splitter. The performance of semitransparent beam splitter is simulated using interface roughness and interdiffusion. The 100nm silicon nitride membranes of clear area of 10mm×10mm is used as the substrate of a beam splitter in the fabrication. First of all, the deposition thickness of Mo and Si per second is required when Mo/Si multilayer is deposited on the 100nm silicon nitride membrane. The thickness of the Mo layer in the beam splitter is 3.65nm, and the one of Si layer is 3.65nm. Mo/Si multilayer is deposited by using DC magnetron sputtering, and the layer thickness is controlled by time. The base pressure is less than 8×10^{-5}Pa in the process of deposition. The working gas is Ar gas which purity is larger than 99.99% and the working pressure is 0.25Pa. The distance between targets and substrates is 90mm. The power of Mo and Si target are 20W and 20W respectively. The deposition rate of Mo is 0.43nm/s，and the one of Si is 0.31nm/s at above condition. According to the deposition rate, the time needed to stay in different target is controlled by a computer. The figure error of the beam splitter is measured by the ZYGO profiler, which show the beam splitter surface shape precision reached the nanometer magnitude in the center region of a beam splitter, which meeting the request of experiment. Synchrotron radiation measurements in BSRF at 13.9nm provide a reflectivity of 18% and a transmission of 22% shown in Figure 1. From these measurements the reflectivity and transmission product of the beam splitter is close to 4%. The beam splitters were successfully used in a X-ray laser Mach-Zehnder interferometer and interference fringes were obtained.

Key words: x-ray beam splitter multilayer plasma diagnostics

Design of the broad angular multilayer analyzer for soft x-ray and extreme ultraviolet

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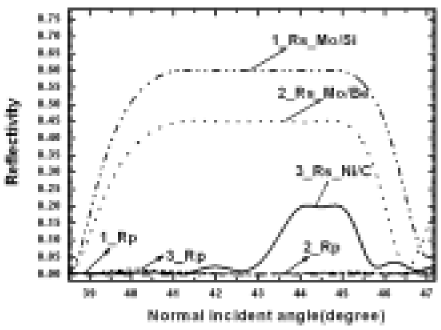
A new design method for the soft x-ray (SXR) and extreme ultraviolet (EUV) broad angular multilayer analyzer has been presented. The traditional multilayer analyzer should be placed at the Quasi-Brewster's angle, which is very difficult and complicated in practice. To overcome the shortcoming, the non-periodic broad angular analyzer using the numerical method is developed. The broad angular multilayer analyzer can deviate the Quasi-Brewster's angle several degree and show very high polarization. The main feature of our approach is the use of an analytical solution as a starting point for direct computer search, and the desired results can be given in a reasonable time. The method can be applied in different spectral range for suitable material combination. Figure shows s-reflectivity and p-reflectivity of Mo/Si, Mo/Be and Ni/C broad angular analyzers optimized with the use of direct computer algorithm to provide the plateau s-reflectivity for different material combination. (1)Mo/Si multilayer, R_0=0.60, N=40, λ=13nm,[41-45degree]. (2)Mo/Be multilayer, R_0=0.45, N=40, λ=11nm, [41-45degree]. (3)Ni/C multilayer, R_0=0.16, N=60, λ=5.2nm, [44-45degree]



Key words: Soft x-ray, Extreme ultraviolet, Multilayer, Broad angular, Analyzer, Quasi-Brewster's angle

Wide-range reflection multilayer for normal-incident optics in the 200-25nm region

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In EUV region, the working wavelength range of normal incidence optical systems has been limited above 40nm, because normal incidence reflectances of usual mirror-materials are small below 40nm. Therefore, the development of multilayers working range in shorter wavelength region has been made [1, 2]. In this study, new high reflectance mirrors with the short wavelength limit of 25nm are designed and fabricated improving the previous one [2].

A conceptual structure of the wide-range multilayer is composed of a top-single-layer (TSL) reflecting light in the 200–65nm region, a middle-aperiodic-layers (MAL) reflecting light in the 65-25 nm region, and a bottom piled-double-layers (PDL) reflecting light around 25 nm. Measured reflectance of the fabricated wide-range multilayer for an angle of incidence of 5° is compared with those of the usual coating materials in Fig. 1 (a). Obtained reflectance of the multilayer is designated as the solid curve, and simulated reflectances of the materials, as the broken curves. The measured reflectance is comparable with that of Pt in the 200–35nm region and is higher than any materials in the 35–25nm region. Measured reflectance is also compared with the simulation results of the designed multilayer in Fig. 1 (b). The reflectance is lower than that of simulation in the 200–62nm region, which suggests that the fabricated thickness of the TSL layer (SiC) is thinner than the designed one. The reflectance is comparable with the simulation in the 62–41nm and 31–23nm regions. These results suggest that the layer structure of the fabricated PDL fulfills the designed one. The reflectance in the 41–31nm region is lower than the simulation, and this may be caused by the difference of the optical constants between the fabricated Y_2O_3 and/or Mg layers in MAL and the data used in the simulations [3].

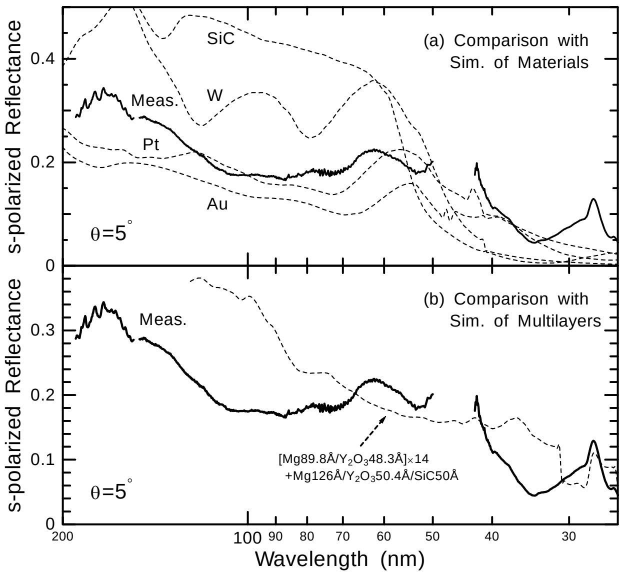

Fig. 1: Measured reflectance of the wide-range reflection multilayer compared with reflectances of SiC, W, Pt, and Au (a), and with simulated one (b).

References

***** Present affiliations: Shanghai Dianji Univ., Shanghai Jiaotong Univ., and Saga Univ.

[1] Y. Kondo, T. Ejima, K. Saito, T. Hatano, and M. Watanabe, Nucl. Inst. Meth. Phys. Res. A **467-468** (2001) 333.

[2] T. Ejima, K. Sudo, T. Hatano, and M. Watanabe, J. Phys. IV France, **104** (2003) 259.

[3] B. L. Henke, E. M. Gullikson, and J. C. Davis, At. Data Nucl. Data Tables **54**, 181 (1993).

Phase change observation of EUV reflection multilayer by total electron yield X-ray standing wave method

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In extreme ultraviolet (EUV) wavelength region, normal incidence mirrors are made available with use of reflection multilayers. One of reflection multilayers at around 13nm wavelength is composed of a material pair, Mo and Si, and its reflectance is more than 60%. This reflectance is high enough; therefore many imaging optics using Mo/Si multilayers are planned including projection lithography in next generation. In reflection optics of high imaging quality like in lithography camera, figure errors should be 1/16 or less. To obtain such accurate optics, surface milling at the top of reflection multilayer was proposed as the figure-error-correction method [1, 2]. To correct the figure error, phase information of the optics should be known in advance of the correction.

Total electron yield (TEY) intensity is optically represented by three terms: absorption-, reflection-, and interference- terms [3]. TEY intensity of reflection multilayers is approximately represented by a simple phase term, which is included in the interference term. When the attenuation length L of emitted photoelectrons from the top layer is smaller enough than the thickness d of the top layer, the interference term of TEY intensity is represented by

$$2\ R\ \mathbf{cos}\ (\delta - 2\xi d - 2\xi L), \tag{1}$$

where R is the real part of the complex reflectance of the top layer, δ is the phase at (m-1)-th layer (m-th layer is the top layer in the formula [3]), and ξ is the real part of the top-layer's propagation-vector represented by $\mathrm{Re}[(2\pi/\lambda)(\varepsilon - \varepsilon_v \sin^2\theta)^{1/2}]$ using angle of incidence θ and complex dielectric functions, ε and ε_v, of the top layer and the vacuum, respectively.

In this study, [Mo 2.6nm/Si 4.1nm]×20 multilayers with different thicknesses of top Mo layer were fabricated on a same Si substrate. Thicknesses of the top Mo layer were accurately controlled by the shutter that was placed in front of the sample, and were deposited from 0.4nm to 3.2nm at 0.4nm intervals. TEY spectra of these aperiodic multilayers were measured with reflection spectra. Obtained TEY spectra showed that peak-positions changed as the increase of the film thickness of the top Mo layer. These TEY spectral changes give the phase change on reflection according to the equation (1).

References

[1] M. Yamamoto, Nucl. Instrum. Meth. Phys. Res. A, **467-468** (2001) 1282.
[2] M. Singh, M. F. Bal, J. M. Braat, D. Joyeux, and U. Dinger, Appl. Opt., **42** (2003) 1847.
[3] T. Ejima, Jpn J. Appl. Phys. **42** (2003) 6459.

Zone Plates with Optimized Zone Profiles

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It has been previously demonstrated that zone plates with slanted zones that meet the Bragg condition can have improved efficiency over zone plates with vertical zones [1-2]. This is of great interest for soft x-ray microscopy since it both improves the efficiency of the zone plates and makes higher order imaging feasible. There has previously been a proposed technique to fabricate a zone plate that approaches the goal of tilted zones by a multi-step electron beam lithography process, but the extremely tight specifications for placement accuracy for each step have made this challenging. We propose a one step method to fabricate zone plates with the optimally tilted zones intended for both improved efficiency and higher order imaging, and will present preliminary data on their fabrication.

[1] G. Schneider, Appl. Phys. Lett. 71, 2242 (1997)
[2] D. Hambach and G. Schneider, J. Vac. Sci. Technol. B, 17(6), 3212, Nov/Dec 1999.

Accuracy Evaluation of the X-ray interferometer for EUVL Optics

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Extreme ultraviolet lithography (EUVL) is expected as the next generation technology. EUVL projection optics has NA0.2-0.3 and the wavefront aberration must be less than 0.5 nm rms. In order to fabricate such accurate optical system, higher accurate, e.g., 0.1 nm rms, wavefront metrology tool is required. In EUVA, at-wavelength interferometer for wavefront metrology has been studied. Recently we evaluated absolute measurement accuracy and obtained good results.

Figure 1 shows the point diffraction interferometer (PDI) built at NewSUBARU. Wavelength is 13.5 nm and the test optics is Schwarzshild optics with NA0.20. Wavefront from the 1st pinhole is separated by the grating to two. One through the window is the wavefront to be tested. The other through the 2nd pinhole with 50 nmϕ becomes spherically reference wavefront. These two interfere on CCD. Wave front aberration can be obtained by analyzing the interferogram.

Absolute accuracy of the interferometer has been evaluated by the following. Measured data is composed of the real wavefront and the systematic error. By rotating the test optics, real wavefront to be tested rotates with the test optics and the residual component is the systematic error. Figure 2 shows the wavefront before and after 90 degree rotation. Both show similar wavefront shapes. Systematic error can be estimated from the difference wavefront by some calculations and as about 0.10 nm rms.

Acknowledgement

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Reference

T. Hasegawa et. al., Proc SPIE , vol. 5374 (2004) 797-807

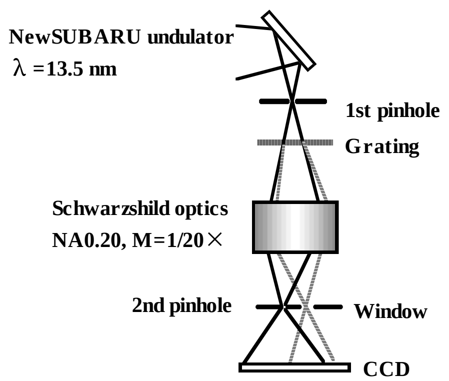

Figure 1. Concept of the PDI.

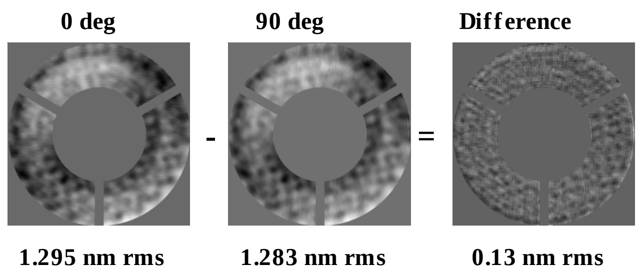

Figure 2. Measured wavefronts and the difference.

Xradia's Path To 25-nm X-ray Zone Plates For Hard X-ray Imaging

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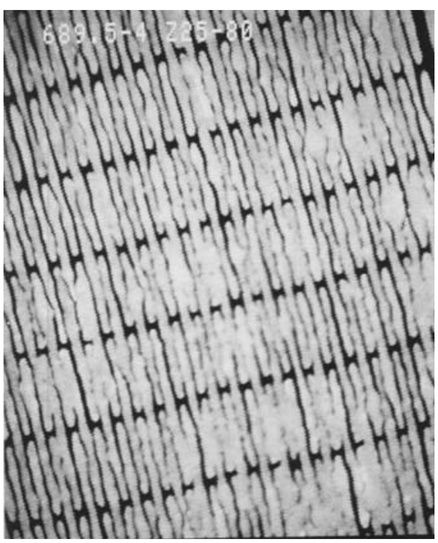
We present the latest results on Xradia's effort towards high-resolution, high aspect ratio gold zone plates for x-ray focusing and imaging applications. Recent successes include the fabrication of zone plates of 40nm zone width and with 700nm Gold zone height and zone plates of 25nm zone width and with 200nm Gold zone height.

Fabrication of zone plates for hard x-ray applications is very challenging because it requires fabricating nanometer scale fine structures with a thickness many times of the feature size. For example, producing 25-nm resolution x-ray zone plate with optimal focusing efficiency for 8 keV x-rays would require a thickness about 1,600 nm, which is about 80 times the smallest zone width of 21 nm required for 25-nm resolution. Currently, generating zone plate patterns with the required zone width and registration can be achieved by many commercial e-beam lithography systems. The limiting factor in making high-resolution zone plates for hard x-ray imaging using this technology is the transfer of these structures into high aspect ratio structures made out of a high-Z material such as gold. In the last few years Xradia has refined a tri-level process scheme to fabricate zone plates with outstanding efficiency and resolution for hard x-rays. We will report the latest results of our zone plate fabrication capabilities.



Outermost zones of a Gold zone plate with 25nm outermost zone width and 200nm Gold height fabricated for Xradia's nano-XFI x-ray fluorescence imager.

Diffraction efficiency of multi-level zone plate fabricated by sputtered-sliced method for hard X-ray focusing

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We have been developed a kinoform type zone plate (KZP) fabricated by sputtered sliced (SS) method for hard X-ray microspectroscopy. The KZP has much higher diffraction efficiency than conventional zone plates [1]. The ideal diffraction efficiency of the KZP is 100% without considering the X-ray absorption. Especially, the SS-KZP can be applied for the higher energy X-ray focusing due to the high aspect ratio.

As a first step of the KZP development, we fabricated a multi-level-type (Cu/Al, 4-level) zone plates at AIST and tested at BL-20XU of SPring-8 [2, 3]. The Cu/Al 4-level zone plate has two half layers between a transparent layer and an opaque layer. In this study we discuss the calculated focusing efficiency of the multilevel type zone plate fabricated by the SS method. Moreover, the multi-level zone plates composed of other material combinations such as W/C, Cr/C, Ag/C, Cu/C are also discussed.

[1] M. Yasumoto et al., Proc. of XRM2002, pp189-192
[2] N. Kamijo et al., in this conference
[3] S. Tamura et al. in this conference

Helical-undulator beamline for soft X-ray microspectroscopy at Saga LS

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An X-ray microscopy project has been proposed at the new synchrotron light source (Saga-LS) that is a third generation 1.4-GeV storage ring [1]. In the project, a planar undulator is used as the insertion device and the following specifications are expected. The linearly polarized X-ray is ~70-nm diameter spot; ~2×10^3-energy resolution (E/dE) and over 10^9-photon flux (photons/sec) at sample in the X-ray region of water-window.

On the other hand, there is an increasing interest in using a circularly polarized (CP) radiation generated from a helical undulator. The CP radiation has been recognized as a powerful tool in the investigation of polarized properties such as X-ray magnetic circular dichroism measurement. Thus, we studied the feasibility of the helical undulator beamline for soft X-ray microspectroscopy at the Saga LS. Details of the feasibility will be presented at the conference.

Reference

[1] M. Yasumoto et al., Proc. of XRM2002, pp63-66, 2003.

Egorov's Type X-ray Waveguide

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As it was stated by Egorovs [1], planar x-ray waveguides with air slit can be used for the production of high intensity guided beams, those size in one dimension is of several microns. To verify it, planar x-ray waveguide has been fabricated and a series of measurements was done. The waveguide structure is shown in insert to Fig1. Incident x-ray beam (Mo target, 30 kV, 5 mA), passed through the vertical slitt (0.15 mm), was directed into the waveguide inlet. A horizontal slit of 0.15 mm was mounted in front of detector, measuring the guided beam intensity. A distance between waveguide exit and detector was 15 mm. A guided beam of high intensity ($1.6x10^6$ total counts per 100s) was recorded at the exit of the particular waveguide (Fig.1).

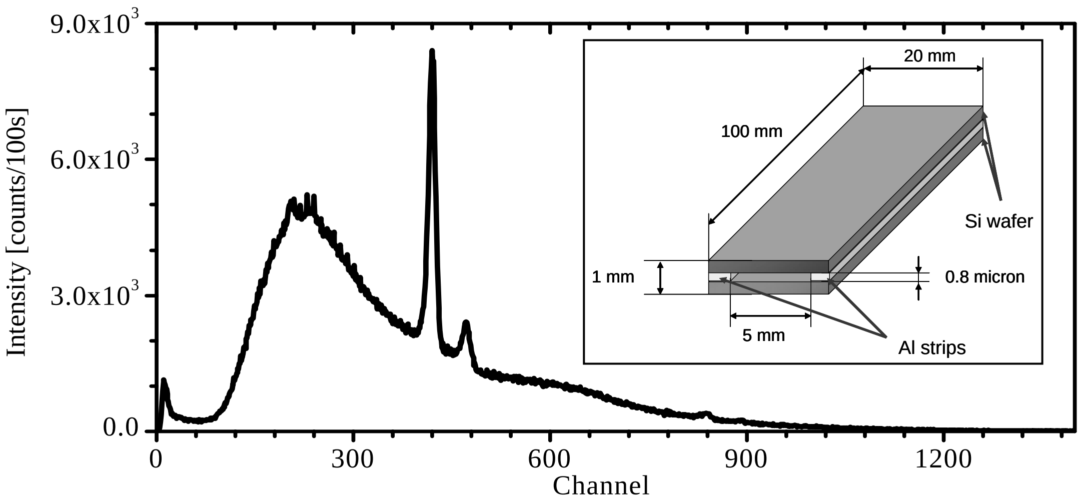

Figure 1. Spectrum of the x-ray beam, guided by Egorov's type planar x-ray waveguide. Guiding element structure is shown in insert.

References

1. VK Egorov and EV Egorov, Spectrochim Acta B, **59**, 1049-1069, 2004.

Shutter Speed Function of Shortest Elapsed Time in Thickness Distribution Controlled Multilayer Deposition

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For wavelength matching in soft X-ray multilayer fabrication for imaging optics, the Research Center for Soft X-ray Microscopy developed an ion beam sputtering deposition system with a shutter moving at a programmed speed in front of the spinning substrate[1]. Figure 1 shows a view from the sputtering target toward the substrate. The shape of the shutter is triangular for a longer exposure near the edge of the substrate than at the center. The sector angle ψ was tentatively chosen as 60°.

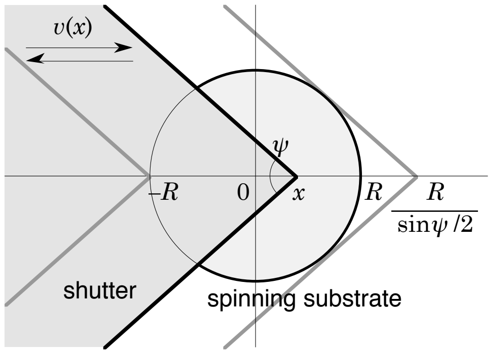

Fig. 1. Front view of a spinning substrate and a programmable shutter for thickness distribution control in multilayer deposition.

The theoretical description on the derivation of the shutter speed function was given in ref. 1. It was found that switching the function at the center enables an accurate thickness control on whole area of the substrate. If the inverse of the speed function is expressed by a third polynomial, switching increases the degree of freedom by 3, which can be used for a shorter elapsed time within limitations of the shutter driving mechanics. In this study the procedure to obtain the shutter speed function of the shortest elapsed time was found and its sector angle dependence was estimated with deposition parameters of our system assumed. Open squares and closed circles in fig. 2 show the shortest elapsed times for one layer deposition of alternative materials. A Mo/Si multilayer for a wavelength of 13.5 nm on a concave substrate of a 300 mm radius of curvature and a 100 mm size in diameter was assumed. The solid line shows the stroke of the shutter in a unit of the radius of the substrate. In the range of ψ < 90° the elapsed time is almost the same while the stroke rapidly increases toward ψ = 0°.

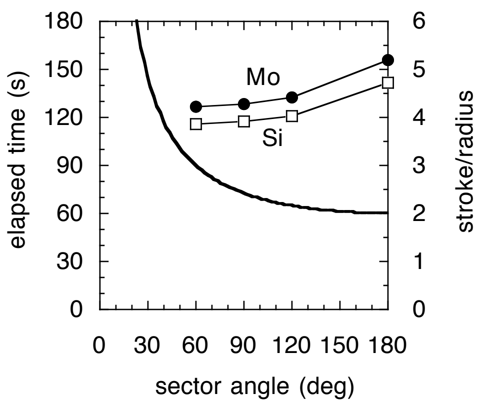

Fig. 2. Sector angle dependent shortest elapsed times for one layer deposition in a Mo/Si multilayer and stroke of the shutter.

[1] T. Hatano, H. Umetsu and M. Yamamoto, *Precision Science and Technology for Perfect Surfaces, JSPE Publication Series No. 3* (JSPE, Tokyo, 1999) pp. 292−297.

A fixed-exit high-efficiency Kirkpatrick-Baez micro-focusing optics for XAS and XRF microspectroscopy in the 6-13 keV X-ray energy range.

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Producing a microbeam is one way of getting spatially resolved X-ray images of large specimens (with respect to the size of the probe beam) via raster scanning methods. To perform spectro-microscopy imaging, additional constraints on the optics, such as achromaticity and ability to reach many absorption edges while preserving a high flux, can greatly simplify the experimental setup.

For this purpose, we have developed a special Kirkpatrick-Baez multilayer-coated optics. Prior to the experiment the shape of each individual mirror is optimized on-line according to the Bragg angle corresponding to the mean X-ray energy of the XAS spectrum or to the excitation energy (XRF). The multilayer coatings have been designed to produce an energy bandpass $\Delta E/E$ of 15% for any X-ray energy set within the 6-13 keV range, so that both focus size and throughput can be preserved while scanning the energy.

The setup is presently installed on the ESRF bending-magnet beamline BM5. It is combined with either a double-reflection Si(111) monochromator (XAS mode) or a low-pass double-reflection multilayer monochromator (XRF mode). Both are mounted upstream the KB with additional slits to further define the incident beam direction and position. In the focal plane, the smallest beam size achieved is presently of the order of 1 micrometer (40 m from the source) with a flux of 2 10^8 ph/s per mA of ring current when using the multilayer monochromator. In light of few examples we will present the performance and the limits of the system and emphasize the conditions for beam stability, in position and in size, over time.

Easily replicable large aperture Fresnel lenses of highly regular structure for the micro-focusing of hard x-rays

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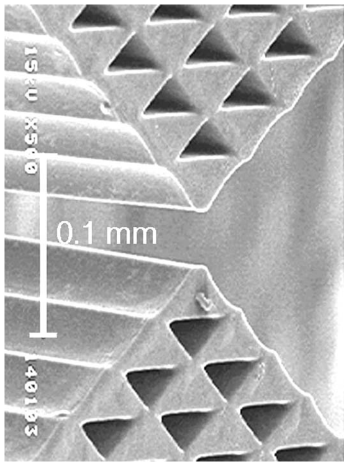
The object shown in the SEM picture shows the central part of a new refractive transmission lens for x-rays. This lens is composed of segments of almost identical shape, i.e. two large prisms, which are each composed of many smaller prisms or prism like objects of the same shape.

The parameters for the first prototypes of these lenses were adjusted for their production and replication by deep x-ray lithography (DXRL) in photo resists [1]. This results in focal length, which are very similar to those used in other concave refractive lenses for the focusing of x-rays. The lens belongs to the category of Fresnel lenses or kinoform lenses, as in it optically passive material, which will change the phase of the passing wave field by integer multiples of 2pi, has been removed. The average amount of the remaining absorbing material grows linearly with distance from the optical axis of the lens.



This contribution will discuss the limitation in the lens aperture due to the absorption and due to the state-of-the-art for the structure depth. The latter will have to match the lens aperture in a crossed configuration for bi-dimensional focusing. In addition the effect of the spherical abberations on the image size and the photon energy tunability will be discuss. The results of the latter discussion are applicable for all forms of Fresnel lenses.

The performance of the first lenses of this concept for 8 keV photon energy was:

- an image size of 2.8 microns could be measured for an expected image size of 1.75 microns with prism heights of 18.34 microns and rather careless lens alignment.
- a lens with a geometrical aperture of 1.5 mm showed an average refraction efficiency of 40%. This results in an effective aperture of 0.6 mm, which is identical to what can at best be achieved with linear concave lenses of the same focal length even if made in the less absorbing beryllium.
- structure depth of 0.4-0.6 mm, i.e. about 25 times the prism size, and 50% of the theoretically expected refraction efficiency can be achieved routinely.

[1] W. Jark et al, J. Synchrotron Rad. **11,** 386-392 (2004)

Fabrication of a Condenser Mirror for a Soft X-Ray Microscope

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There have been many attempts to investigate nanometer-scale fine structure. A soft X-ray microscope can be used to investigate hydrated specimens, particularly living cells, with a resolution several times better than that possible with visible light microscopes. The reflecting optics used in a soft X-ray microscope require supersmooth surfaces and a highly accurate figure. We consider a Wolter type I microscope mirror as a condenser optic. This consists of two axially symmetric confocal surfaces of revolution: an ellipsoid and a hyperboloid.

We have optimized the design of the Wolter type I mirror for observing living cells at a wavelength of 2.3 nm in a laboratory prototype. The condenser mirror of 1/4 X magnification was chosen. The effective solid angle, which is defined as the product of the geometrical solid angle of the mirror and the reflectivity after two reflections, showed a maximum at the desired 2.3 nm wavelength.

Since the grazing angle of incidence is about 2 degrees, the mirror has a small diameter and a long length. It is not easy to fabricate such a mirror by direct machining and polishing because of the internal reflecting surfaces. As an alternative, a replication method was chosen. Machining of the master mandrel is very important because the quality of the replicated mirrors critically depends on the surface roughness and figure of the master mandrel. We first prepared the master mandrel by single-point diamond turning electroless nickel that was plated onto an aluminum alloy. The surface of the master mandrel was then polished. Finally, we fabricated Wolter type I mirrors using an epoxy replication technique. A thin gold layer, 300 nm in thickness, coated onto the master mandrel plays an important role as a parting agent to separate the master mandrel and the mirror substrate with a gold reflecting surface. The gold-coated mirrors replicated the master mandrel very well, although there were minor variations in the surface roughness and figure. After separation there was no surface degradation and figure change on the master mandrel, and several mirrors could be successfully made from one master mandrel. The surface roughness and figure error of the replicated gold mirror were 1.2 nm rms and 160 nm PV, respectively.

Laser-plasma x-ray source using ceramic target

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We are investigating table-top x-ray lasers based on the recombination plasma scheme. In this scheme, the well-known lasing line is Al XI 3d-4f transition line at 15.47 nm. In x-ray laser experiments, pure aluminum slab or tape target is often used. After laser irradiation, some craters are created on the target surface or a part of aluminum tape is ablated, resulting much debris particle production. It causes one of the technical issues for development of practical x-ray lasers and also laser-plasma x-ray sources. On the other hand, ceramic materials containing Al would produce few debris because of its high sublimating decomposition temperature (2450 °C), whereas the melting point of Al metal is 660 °C. Though, ceramic materials could be useful as target materials, there are no attempts for laser-plasma x-ray sources.

We have carried out x-ray laser experiments using aluminum nitride as target material. A sintered AlN plate of 2 mm thickness was irradiated by a train of 16 pulses (100 ps width, 200 ps interval) with power density of about 10^{11}-10^{12} W/cm^2. Incident laser was focused in a line of 11 mm length onto the target. Soft x-ray spectrum originating from Al XI (Li-like Al) ions was measured with the same intensity as in the case of Al slab target, though the concentration of Al is lower than in pure aluminum. Craters on the AlN plate after single laser shot were not clear under optical microscope observation. It was found that when the laser was focused on the same position of the target, the 15.47 nm x-ray intensity for each laser shot was almost constant for 65 shots and more. Whereas in the case of an Al slab target, the x-ray intensity decreased for more than 10 laser shots. It would be possible to construct a long-lived and compact laser-plasma x-ray source with less debris using a peace of AlN plate.

Quantitative results on x-ray radiation, debris and target surface deformation will be presented.

Ionization and explosion dynamics of atoms in clusters irradiated by ultrashort, intense laser pulses

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Atomic clusters offer a unique area for studying high-intensity laser-matter interactions. Recently, the development of the laser-based x-ray sources using cluster targets has attracted lots of attention. This is because, compared to the solid targets, cluster targets produce less debris and are easy to handle. Although extensive studies on intense x-ray emissions from laser-cluster interactions have so far been carried out [1], the detailed physical mechanism is still ambiguous because of the highly nonlinear nature of the laser-cluster interaction. Thus, it is essential to develop the model which includes various atomic and relaxation processes as well as non-Maxwellian effects in a self-consistent manner.

In this study, short pulse laser-cluster interaction and resultant ion explosion dynamics with various charge states are investigated in detail by employing a particle based integrated code including ionization and relaxation processes. We have revealed that a formation of an enhanced electric field near a cluster surface is the key to understand complex ionization and explosion dynamics of atoms in a cluster. We have also found that a formation of an exploding ion front and a resultant sheath field affect an electron energy distribution. A combination of the non-Maxwellian electron energy distributions thus obtained and atomic kinetics rate equations will enable a more realistic modeling of the x-ray emission spectra obtained under the action of superintense laser radiation.

[1] Y. Fukuda et al., Appl. Phys. Lett. **85**, 5099 (2004).

Laser-based debris-free x-ray sources for picosecond x-ray diffraction

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Ultrafast time-resolved x-ray diffraction studies using laser induced x-ray pulses from solid targets have been extensively performed and revealed new phenomena such as ultrafast melting. Recently, the development of the laser-based x-ray sources using cluster targets has attracted a lot of attention. This is because, compared to the solid targets, cluster targets produce less debris and are easy to handle. Thus cluster targets have been suggested as clean sources of x-rays for various purposes.

We have investigated x-ray radiation properties of relativistic cluster plasmas created by the action of super-intense laser irradiation [1]. In order to demonstrate the practical capability of x-rays thus produced, the pulse x-ray diffraction is examined from Si(111) crystal with this source [2]. Figure 1 shows the typical CCD image of the diffracted x-rays. The diffraction pattern consists of well resolved *K*-shell emissions from highly ionized Ar ions. The number of photons in a 4π sr solid angle for $He_{\alpha 1}$ resonant line of Ar (λ=3.9491 Å, 3.14 keV) was calculated as 4×10^8 photons/s/pixel. Thus we have demonstrated that x-rays produced from the laser-irradiated clusters are strong enough to utilize as a debris free light source for time-resolved x-ray diffraction studies.

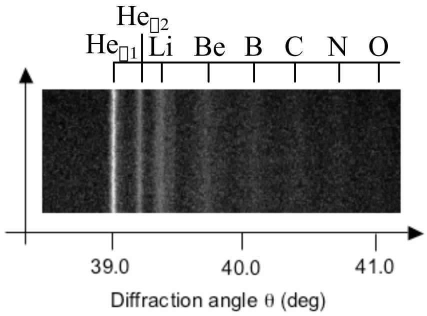

Fig. 1 The typical CCD image of diffracted x rays measured at the peak intensity of 6×10^{18} W/cm^2 with 30-fs pulse duration.

[1] Y. Fukuda et al., Laser Part. Beams **22**, 215 (2004).
[2] Y. Fukuda et al., Appl. Phys. Lett. **85**, 5099 (2004).

Projection X-ray microscopy with MIRRORCLE-6X

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The MIRRORCLE-6X is a brilliant x-ray source based on a portable synchrotron. Its orbit radius is only 15cm, and its magnet is made of one set of normal conducting coils. MIRRORCLE-6X is suitable for hard x-ray imaging, particularly for a projection x-ray microscopy without using optical elements. Since the x-ray target placed in the central orbit determines the effective x-ray source size, the X-ray emitter size is extremely small as an order of micron. The X-ray brilliance is the same order as large synchrotron light sources, because the electron beam is re-circulating. X-ray radiation angle is determined by the kinematics $1/\gamma$ (=85mrad for 6MeV), thus the irradiation field is rather large compared with conventional SR sources. Placing a 2-dimensional imaging device far from the specimen realizes the magnified projection microscopy with high space resolution. We can perform the best kind non-destructive inspections and medical diagnosis. X-rays are dominated by hard components of more than 30keV, but due to the phase contrast effect, hard constructions as well as soft tissues can be observed. We will soon have a sub-micron target for 100 times magnification. Figure.1 shows x-ray images of green pepper with different magnification observed by the imaging plate, which has 150-micron pixel size. (FCR XG-1 produced by Fujifilm Ltd.). We will report many high quality aspects of MIRRORCLE.

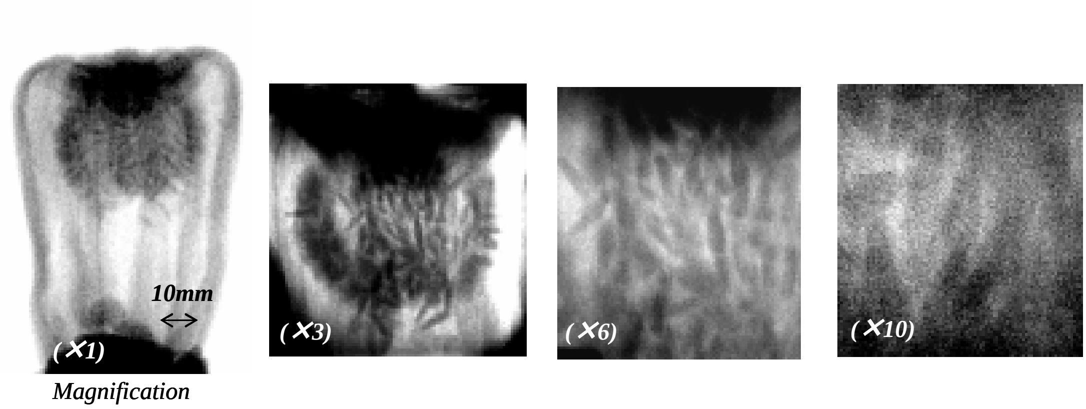

Fig.1 Ten times magnified x-ray images of green pepper taken by MIRRORCLE.

New Type of Targets for Projection X-Ray Microscopy of Samples constiting of light Elements

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In the case of projection X-ray microscopy, it is necessary to use suitable target to give long wavelength X-rays for good image contrast to the minute biological samples which consist of light elements. We reported that Ti k-line : 0.27 nm and Ge L-line : 1.04 nm are good choice in such a case. It is rather difficult, however, to select suitable metal elements as the target to give much longer wavelength soft X-ray for sufficient image contrast utilizing absorption edge effect because desired elements from viewpoint of wavelength are not always stable ones having high melting point and electric conductivity like F, S, Cl. If we consider their chemical compounds, freedom of the choice will be increased. From this point of view, we consider targets of chemical compounds for imaging of biological samples such as hair, chromosome and so on. As possible combinations of the compound targets, MoS_2 for DNA, AgS for Al, Si, P, MgF_2, CrF_3 for O (water droplet) are taken into consideration. These compound targets are formed by vacuum evaporation or sputtering on thin Be substrate which works as the vacuum seal window and finally electric conductivity is given to them by evaporation of carbon or Be thin layer to prevent charging up. X-ray images of human chromosome were obtained with the compound target. Some other experimental results will be reported.

Liquid jet targets as a water-window radiation source

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One problem of laser-plasma X-ray sources from a solid target is the debris which can coat and scratch delicate x-ray optics, degrading their performance. To avoid this problem, tape targets, gas targets, liquid targets, gas-shielding, and so on have been suggested, tested and developed to a degree in the past years. We have endeavored to develop a monochromatic radiation source using ethanol and liquid nitrogen which can be used to an X-ray microscope equipped with zone plates for the best imaging.

In this presentation, the liquid jet system for ethanol and liquid nitrogen will be described. Nanosecond, picosecond, femtosecond lasers have been used to produce plasmas from the liquid jets. Radiation characteristics from these plasmas will be also discussed. Optimized conditions from these plasmas will also be discussed in terms of conversion efficiency.

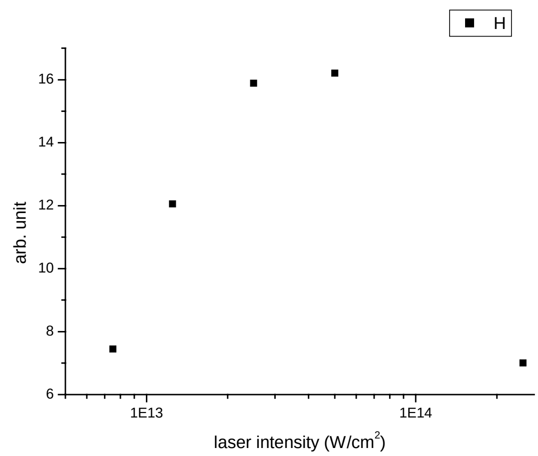

Fig 1 Conversion efficiency for 2.87 nm from liquid nitrogen plasma using a 120ps pulsed laser

Development of 50 Hz Laser-Produced Plasma Soft X-ray Source Using Tape-Target

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We are researching basic technologies of photoelectron microscopy equipments. As a part of the research, we have been constructing laser-produced plasma x-ray sources which can be operated at 50Hz repetition rate. The system consists of a Q-switched YAG laser (4-8 ns, 0.6 J), a focusing optics with combination of concave and cylindrical lenses and a target chamber containing a debris prevention system and an open reel tape-target driver. We developed the debris stopper consisting of rotating thin glass-plate and metal-disk with a small hole. A reel of tape can be irradiated three times within its width and be operated as long as 2 hrs without exchanging the reel.

When aluminum tape-target was used, the dominant spectral line appeared around 13 nm with the incident laser power density of 2×10^{11} W/cm^2. The spatial distribution of 13 nm radiation was investigated by using space-resolving soft x-ray spectrograph. It was found that it peaked around 1 mm distant from the target surface. By settling a small aperture near the source at the peak position, we can extract nearly monochromatized soft x-ray from laser-plasma source without using any dispersion optics.

Furthermore, we have newly developed a simple EUV calorie monitor which consists of a multilayer mirror, a thin filter and an absolutely calibrated silicon photodiode. By using this calorie meter, we measured the absolute radiation energy of 13 nm x-ray at the source position. The result was about 2 mJ/2π str./nm BW/pulse. This calorie meter could be useful for the other x-ray sources.

We have also been developing the tape-target coated with boron for 6 nm x-ray source.

Acknowledgement

A part of this work was performed in the grant-in-aid for scientific research program supported by MEXT and in industrial technical utilization development support program supported by NEDO.

Extreme ultraviolet emission characteristics of a laser-produced Li plasma

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A laser-produced plasma (LPP) has been utilized in various applications such as laser fusion, charged particle acceleration, biological imaging, and microlithography. Present requirement for the next generation microlithography is to develop high average power EUV light sources at 13.5 nm. Due to the high-power requirement of the EUV emission, conversion efficiency (CE) from the plasma-initiating laser energy to the EUV emission energy becomes one of the most important parameters. The EUV CE could be improved by controlling various plasma parameters such as its density and temperature. The use of dual laser pulses could realize such a control of parameters. We have chosen a lithium plasma source, which produced unambiguously defined line emission at 13.5 nm with almost no off-band components[1)].

A Li-mixed aqueous jet target with sub-hundred micrometer diameter was provided in a vacuum chamber. A plasma-initiating laser consisted of dual laser pulses with adjustable delays with pulse widths of 8 and 10 ns (FWHM) at 532 and 1064 nm, respectively. The laser intensity of a main laser pulse was optimized at 3×10^{11} Wcm^{-2}. The EUV CE at 13.5 nm was evaluated within the 2% bandwidth (in-band value) and 2π sr solid angle.

The EUV CE increased as the delay between the two laser pulses increased, and reached its peak value of 0.48% around 100 ns. The value increased by a factor of three from its single-pulse value of 0.15%. The optimized delay time of 100 ns corresponded to the hydrodynamic plasma expansion time, when the plasma density decreased its critical value of the main laser pulse. By optimizing the main laser intensity, the plasma temperature was also controlled, so that the Lyman-α emission intensity at 13.5 nm from Li^{2+} ions increased further compared to that of O^{5+} ions at 13.0 nm. The deployment of dual nanosecond pulses thus produced a plasma with adequate conditions for Li^{2+} ions but not for O^{5+} ions, leading to the improvement of the emission CE at 13.5 nm.

1) C. Rajyaguru *et al*., Appl. Phys. B **79**, 669 (2004); Appl. Phys. B **80**, (to appear in 2005).

High spatial resolution X-ray image detectors at SPring-8

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High spatial resolution X-ray image detectors have been used at SPring-8. They are visible light conversion type and CCD-based detectors and consist of thin phosphor screen, optics (visible light conversion unit) and CCD camera. They are used for many kinds of X-ray microscope as an important device.

The advantage of this kind of detector is to be changed to another kind of detector with changing the CCD camera. That is, the characteristic of the detector almost depends on the characteristic of the CCD camera.

The highest spatial resolution of a detector is better than 1μm (shown in the figure) with the field of view of about 1mm × 1mm while the largest field of view is 24mm × 16mm with spatial resolution of about 12μm using high definition CCD camera (4000 × 2624 pixels).

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

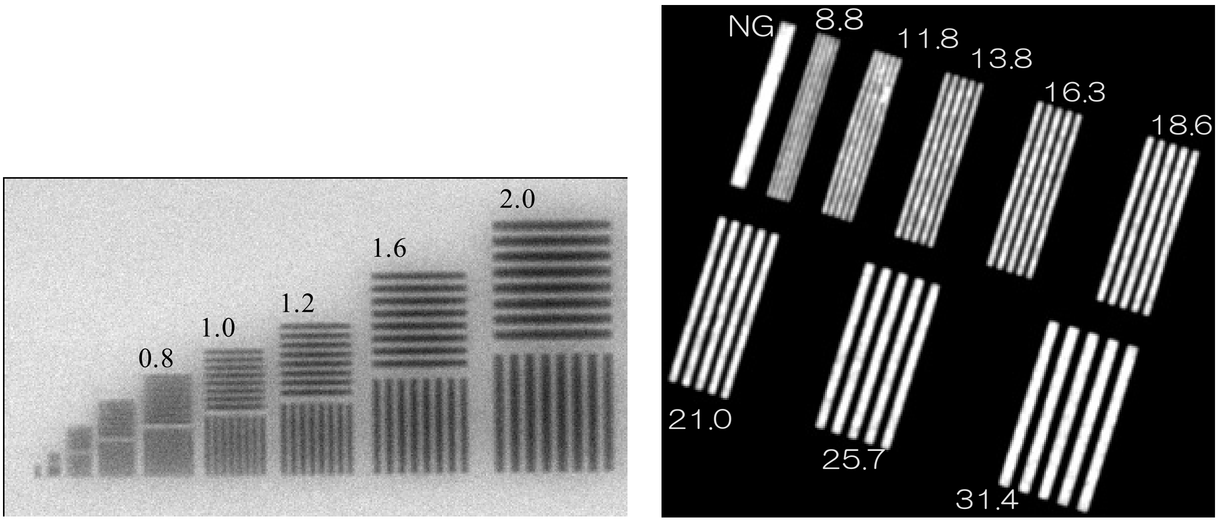

Figure. Left: 0.5μm-thick Ta on Si_3N_4 film. Numbers show period of line and space. The image was obtained by BM3 (x50) and cooled-CCD camera (C4880-10-14A) with the effective pixel size of 0.2μm. X-ray energy was 12keV and exposure time was 8sec. Right: 23μm-thick Au on Kapton film. Numbers show line widths. The image was obtained by BM2 (f=24mm) and cooled-CCD camera (C4880-10-14A) with the effective pixel size of 5.83μm. X-ray energy was 15keV and exposure time was 10sec.

A New Area Detector for High-speed X-ray Diffraction Analysis and X-ray Imaging

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A state-of-art semiconductor technology based area X-ray detector, namely D/teX-25, has recently been developed. The detector enables ultra high-speed X-ray diffraction analysis as fast as 160 degree 2 theta in one minutes when installed to X-ray diffractometer. This is more than 30 times faster than a conventional speed of 5 degree 2 theta per minutes with a scintillation or a proportional counter. Thus it is particularly useful for dynamic and/or *in-situ* studies. In addition to high-speed, the detector makes space-resolved X-ray diffraction study possible. X-ray diffraction with areal resolution is useful for the study of sample uniformity and the possible presence of large or aggregated particles in a specimen, which cannot be aware with a conventional point detector. When the detector is combined a Y-Z stage, a two dimensional X-ray image of large area can be obtained. A few images taken at a preliminary experiment will be shown.

Development of High Resolution Wide-band X-ray Detector: Scintillator-deposited Charge-coupled Device

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We report here a newly developed wide-band photon-counting detector for 0.1−100 keV X-rays possessing high spatial resolution, to be employed as the focal plane detector of the supermirror: the scintillator-deposited CCD (SD-CCD). We employ CCDs as a soft X-ray detector. The scintillator is directly coupled to the back surface of the CCD. The majority of X-rays having energy of above 10 keV cannot be absorbed by the CCD and pass through it. However, they can be absorbed by the scintillator and emit hundreds or thousands of visible light photons. The visible light photons can be absorbed by the same CCD. In order to maximize the number of visible light photons detected by CCDs, the surface of the scintillator is coated by a reflector, such as aluminum, which leads to a better energy resolution.

We measured the X-ray spectral response of the scintillator-deposited CCD (SD-CCD) with SPring-8 BL20B2. We irradiated monochromatic X-ray beam having energy of 20−80 keV and measured the energy dependences of the mean pulse height as well as the energy resolution of the SD-CCD. A good linear relationship between the X-ray energy and the mean pulse height channel can be obtained with our device, suggesting the SD-CCD can surely function as a hard X-ray spectrometer. In order to investigate the imaging capability of the SD-CCD, we measure the contrast transfer function with a test chart having a spatial frequency up to 10 LP/mm whereas the spatial resolution of the SD-CCD far exceeds the limitation by the test charge employed. We thus performed the demonstrative experiment with a sharp-edge structure and obtained (10 ± 3) micron at 17.4 keV with an X-ray photon-counting mode.

Development of X-ray CCDs for the NeXT satellite

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The focal plane detectors of the multilayer supermirror(HXT) are planned to be employed the Wide-band X-ray Imager (WXI) consisting of X-ray CCDs (SXI : Soft X-ray Imager) and CdTe pixelized detector (HXI : Hard X-ray Imager). HXT has a large band effective area in 0.5–80keV. The detector is required of spectroscopic capability in those band. SXI has requirements, one is the high quantum efficiency from 0.1keV to 20keV, the other is to make hard X-ray penetrated the CCDs wafer. In order to fulfill these, We are developing the Back supportless CCDs(BS-CCDs) and N-type CCDs. BS-CCDs are taken the back package and field-free region. N-type CCDs is made of high resistivity N-type silicon, which make the thickness fo the depletion layer. For this CCDs,the QE is improved both in X-ray band and optical band.

We started to develop model of the BS-CCDs as "CCD-NeXT1", whose image area is 24 $\times$ 24 mm and format is 2048 $\times$ 2048 (12 micron) pixels, and N-type CCDs, which are planned thickness of depletion layer to be about 300 micron. The estimated performance of them was obtained used by drive system at Osaka Univertsity. For the first model of "CCD-NeXT1", readout noise was given 11 electrons (r.m.s) and spectral resolution : the full-width at harf-maximun (FWHM) was given 156 eV at 5.9keV (Mn Kα). They are sufficient results at the present time. Next step is to make imaging area large up to 42 $\times$ 42 mm. For N-type CCDs, readout noise and FWHM were given 15 electrons and 255 eV at 5.9keV (Mn Kα). FWHM is lower than BS-CCDs's because of the noise of charge transfer. As previously noted, N-type CCDs shoud has thick depletion layer (about 300 micron) , which is able to measure from countrate of ^{241}Am. the value is under examination. Additionally, charge transfer efficiency (CTE) of N-type CCDs is almost improved at present, about 0.9999995. The development is proceeding smooth.

High resolution Cs-concentration mapping by X-ray CT

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We report the first success of nondestructive, three-dimensional, and high resolution (~20 micron for space and ±2.5 wt% for value) mapping of cesium (Cs) concentration by X-ray CT [1]. This work was performed at BL20B2 of SPring-8 and based on a “subtraction method”, using two energies just below and above an absorption edge of the target element. Although the subtraction method is often used as a qualitative imaging technique of element distribution, we tried to derive quantitative information of element concentration with some corrections. First, nonlinear relation between observed linear attenuation coefficients (LACs) and theoretical LACs was corrected [2]. Secondly, difference of mass attenuation coefficients (MACs) at each slice due to vertical energy shift of an X-ray beam (Fig. 1) was corrected using a homogeneous standard material (Cs-bearing solution). With these corrections and equation in Fig. 1, we could obtain the Cs-concentration maps close to a two-dimensional map acquired by an electron probe X-ray microanalyzer (EPMA).

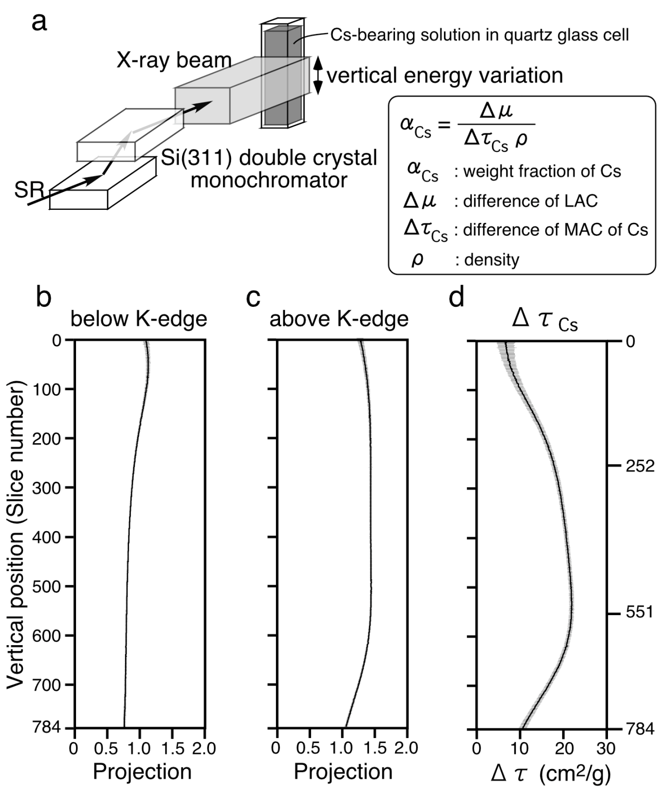

Fig.1 Effect of the variation of X-ray energy. The vertical energy shift within an X-ray beam (a) affects the projections of Cs-bearing solution (b, c) and the difference of MAC of Cs between below and above the absorption edge (d). Horizontal variation (gray error bars) in each slice is much smaller than the vertical one.

References: [1] S. Ikeda et al., Am. Mineral. **89**, 1304-1313 (2004). [2] A. Tsuchiyama et al., Am. Mineral. **90**, 132-142 (2005).