AN X-RAY TOMOGRAPHIC MICROSCOPE WITH SUBMICRON RESOLUTION*

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X-ray tomographic microscopy (XTM) is a powerful non-destructive investigation method, that has been applied in many fields of modern research (material science, microelectronics, medicine, biology, archaeology). Sofar the major limitations were imposed by low detection efficiency and low spatial resolution. With the advent of third generation synchrotron facilities excellent high intensity X-ray sources became available that by far counterbalanced low efficiency. On the other hand the resolution of presently used detector systems is restricted by scintillator properties, optical light transfer, and CCD granularity. They impose a practical limit of about one micrometer, while the progressing research demands urgently an advance in the submicron region. A break-through in this respect is being achieved by a novel detector type. It uses the properties of asymmetric Bragg reflection to increase the cross section of the reflected X-ray beam. A suitable combination of correspondingly cut Bragg crystals yields an image magnification that even at higher energies may surpass a factor of 1000. In this way the influence of the detector resolution can be scaled down accordingly. Such a device is being constructed and installed at the SLS which delivers an optimal X-ray beam of about 23 keV. The special properties of this experiment will be presented.

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1. General considerations

A powerful method to study the inner local structure of opaque material is X-ray tomographic microscopy (XTM). An X-ray beam of fixed energy penetrates the sample and the corresponding intensity distribution is registered by a suitable detector. By using a two-dimensional position-sensitive detector larger areas can be monitored simultaneously. Rotating the sample during exposure, allows to reconstruct a three-dimensional image of the internal structure. When the energy is tuned across an absorption edge of involved elements, high contrast, element sensitive studies can be performed.

The detector systems that are presently used consist of three main components: The X-rays hit onto a scintillation screen, which converts them to visible light. The corresponding image is magnified by a suitable optical lens system. The light output is finally observed by a high granularity CCD camera. On the left side of Fig. 1 the principal arrangement is sketched.
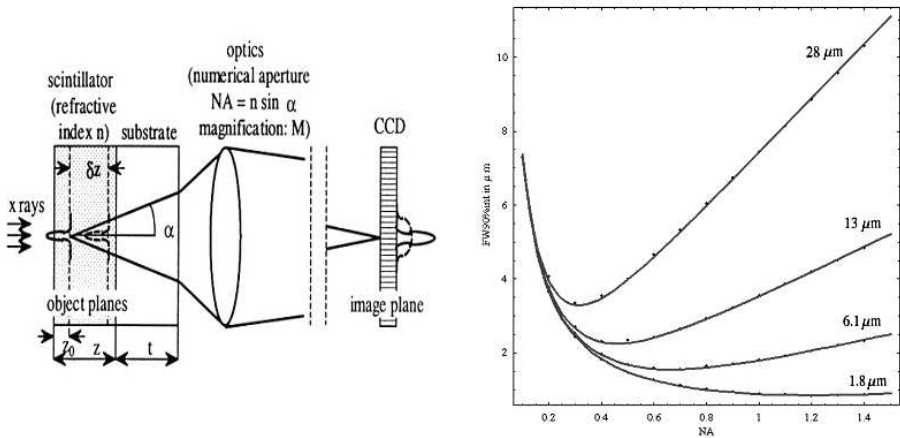

Fig. 1. The standard XTM detector system. On the left side the principal components of the setup are shown: the scintillator, the optical imaging system and the CCD. On the right side the resolution as function of the numerical aperture for several values of the scintillator thickness is displayed.

For all three components there exists a variety of solutions concerning the tradeoff of efficiency *versus* resolution as function of scintillator material, granularity, homogeneity, thickness, transmission, optical magnification, imaging errors, and CCD performance. The dependencies and implications have been studied in great detail [1–4]. As a typical example, on the right side of Fig. 1 is shown the resolution as function of the numerical aperture NA for different values of the scintillator thickness z. As a result, all of these studies indicate, that for the resolution there is a final limit in

the order of 1 μm. For an experimental confirmation the knife-edge procedure is commonly applied: a sharp edge of heavy material is illuminated by a parallel X-ray beam. The intensity distribution is fitted by a superposition of three Fermi functions [5]. The resolution is calculated analytically as derivative. A typical example is given in Fig. 2.

Optimizing a high quality standard detector system for maximum resolution at 23 keV, the following parameters have been found: 5 μm thick YAG scintillator, aberration-corrected optical lenses with NA=0.7, CCD efficiency=22 %. With an overall efficiency of about 5 % a spatial resolution between 1 μm and 2 μm can be expected.
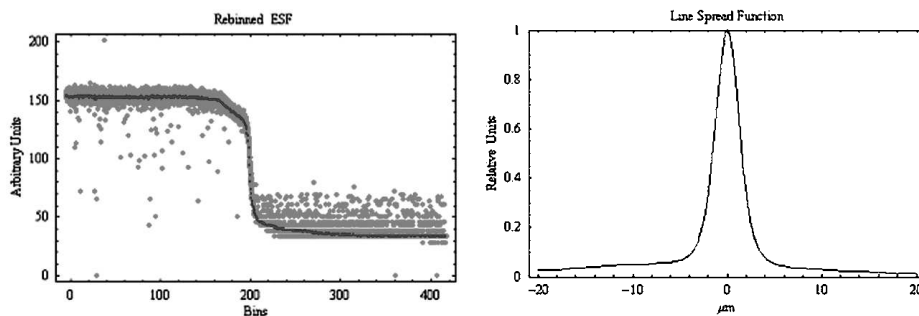

Fig. 2. Experimental determination of the resolution. On the left side is shown the experimental intensity distribution of a sharp edge together with the rebinning (black line). An analytical derivation of the fitted line yields the resolution as depicted on the right side.

TABLE I

Typical research fields using XTM

Research medium	Objects of interest	Required resolution
Metal compounds	Defects, phase interfaces	$< 1\mu$m
Ceramics	Porosity, heterogeneity	$< 1\mu$m
Fibers	Defects, density fluctuations	$< 1\mu$m
Tissue engineering	Porosity, structure	$< 1\mu$m
Microelectronics	Cracks, grain boundaries	$< 1\mu$m
Medical bone research	Osteoporosis	$< 1\mu$m
Biological tissues	Structure, composition	$< 1\mu$m
Geological rocks, ore	Structure, composition	$\approx 1\mu$m
Archaeological bones	Structure, composition	$\approx 10\mu$m
Archaeological artifacts	Structure, composition	$\approx 10\mu$m

With the advent of modern synchrotron facilities high quality X-ray sources became available that more than counterbalances the low efficiency of the standard detection systems. The corresponding beams are monochro-

matic, highly collimated and of high intensity. They opened several new research fields for XTM applications. Some typical examples are summarized in Table I. As can be seen from the last column of Table I the advance of research to smaller and smaller structures leads to a mandatory request for a resolution well below 1 μm. But this cannot be realized with the present standard detector systems.

2. The Bragg magnifier

In order to overcome the above discussed limitations a novel detector type has been developed [6–8] that is going to provide a break-through concerning resolution and will at the same time offer a drastic increase of efficiency. The idea is based on the principle of asymmetric Bragg reflection. The main features are given in Fig. 3. An X-ray beam is hitting at a small angle θ_{in} onto the surface of an ideal crystal the lattice planes of which exhibit an asymmetry angle α with respect to the surface of the crystal. If the energy of the incoming X-rays is such that Bragg's law is fulfilled, *i.e.* $\theta_{in} = \theta_B - \alpha$, the beam is reflected at an angle $\theta_{out} = \theta_B + \alpha$ with respect to the surface. Simple arguments show that the cross section of the reflected beam d_{out} is related to that of the incoming beam d_{in} as $d_{out} = M\, d_{in}$, while the divergences scale in the inverse way: $\Delta\theta_{in} = M\,\Delta\theta_{out}$. By a suitable combination of two asymmetric Bragg crystals C1 and C2 (see right side of Fig. 3) an enlargement of the beam in both dimensions can be achieved. In this way a total magnification $M = M(\mathrm{C1})\,M(\mathrm{C2})$ up to a factor 1000 is feasible. The resulting magnified image can be observed with the help of a standard XTM detector. As in this case its resolution is less stringent, it can be equipped with a much thicker scintillator layer, so that its efficiency is drastically improved.
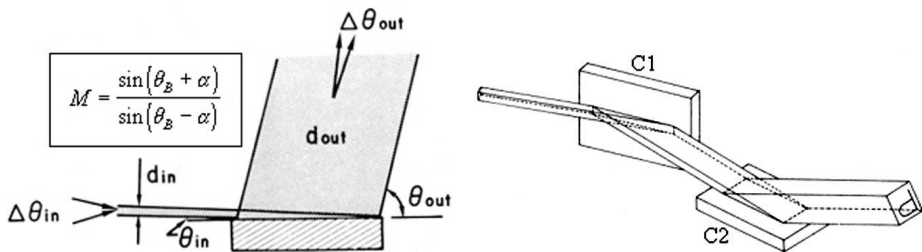

Fig. 3. Left side: definition of asymmetrical Bragg reflection. Right side: the principle of the Bragg magnifier.

To build such a prototype of a Bragg magnifier at the SLS, several Bragg crystals from Si and Ge have been studied. For practical reasons Si (220) crystals were selected for both crystals of the setup. The rocking curves [9] and the essential parameters are shown in Fig. 4.
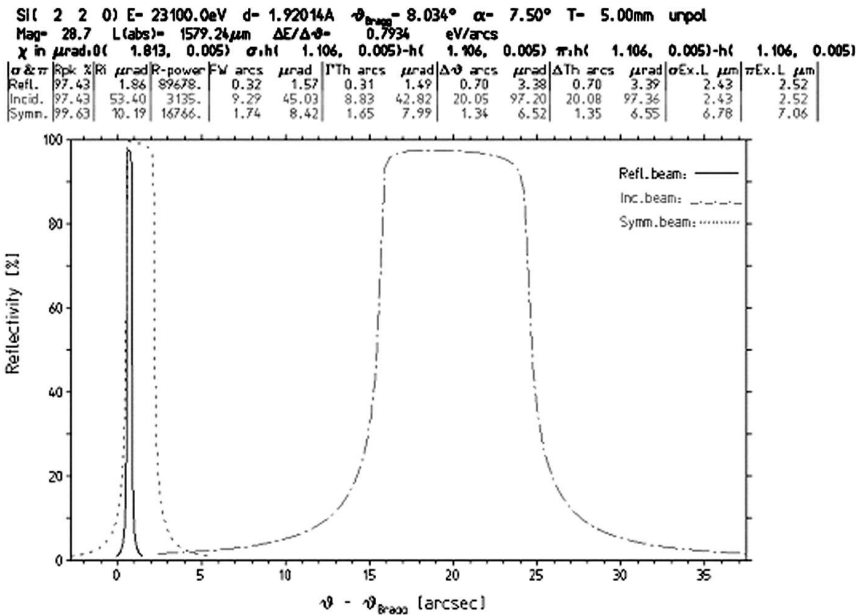

Fig. 4. The rocking curves of Si (220).

To study the optimal parameterization of the setup at the 4S-beamline of the SLS, extensive simulations with the ray tracing program SHADOW [10] have been performed. The X-ray light for the 4S-beamline originates from a wiggler in the SLS ring. The optical elements of the beamline: a Si (111) double crystal monochromator and two Rh mirrors and the entrance and at the exit allow to produce a well collimated beam [11] at 23 keV with a divergence less than 20 μrad (see Fig. 5). A comparison with the relevant rocking curve implies that the first Si (220) Bragg crystal will accept 95 % of the incoming intensity.

With this realistic source distribution the complete Bragg magnifier has been simulated. In the simulation, the beam is collimated by a slit to a 1×1 mm^2 profile, just before impinging on the first crystal of the Bragg Magnifier. The beam is diffracted by the first crystal and magnified along the Z-direction. Then, the second diffraction (perpendicular to the first one) occurs, which magnifies the beam along the X-direction. The enlarged profile emerging from the magnifier after double diffraction is represented in Fig. 6. The results of the simulation suggest that with such a setup a linear amplification of a factor of 30 in both dimensions is feasible.
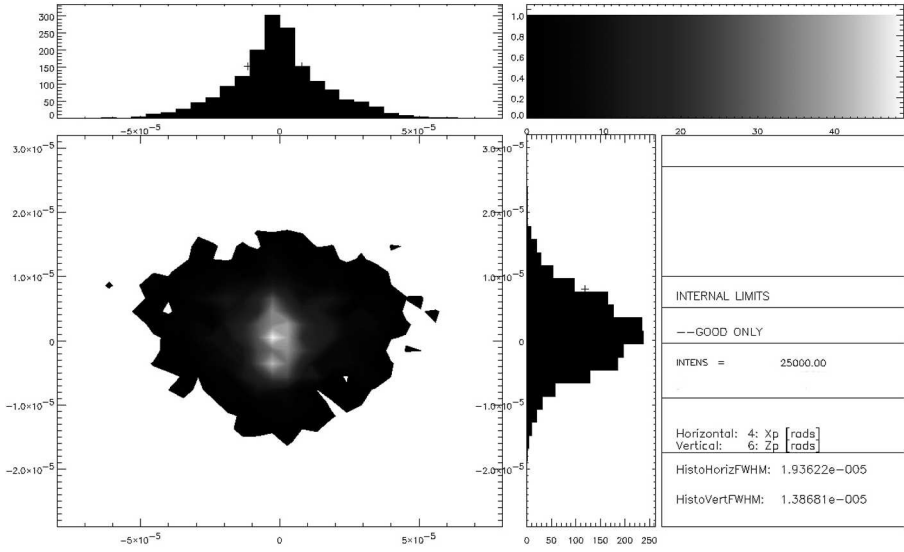
Fig. 5. SHADOW simulation showing the divergence of the 4S-beamline. Intensity is given in arbitrary units.
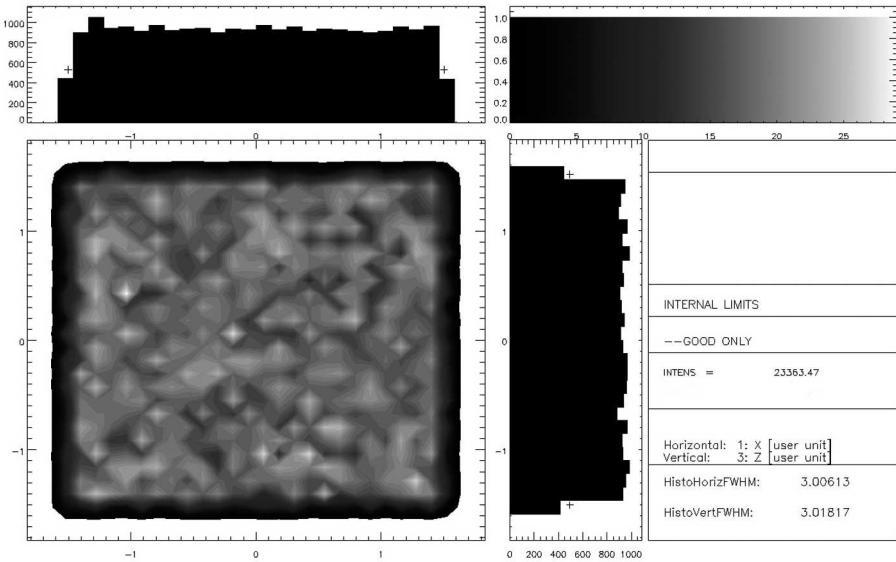

Fig. 6. SHADOW simulation showing the intensity distribution in the final image plane. Histogram FWHMs are given in cm.

Consequently the realization of the experimental setup has been started. The crystals have been prepared in collaboration with the Institut für Kristallzüchtung, Berlin, with an angular accuracy of about one minute of arc and a surface roughness of better than 1 μm. After a high quality polishing of their backside, the crystals have been fixed by optical contacting on a high precision glass support [12]. In this way no mechanical deformation of the crystals themselves can occur.

For each crystal unit the mechanical mounting allows an adjustment around the three rotation axis [13]. The movement that corresponds to the Bragg angle can be performed in steps of .05 arcsec to cope with the very narrow rocking curve at 23 keV (see Fig. 4). In addition, the second crystal can be positioned by means of a XY-table.

3. Outlook

The whole setup including the standard XTM detector has been assembled during the last weeks. After an offline adjustment and testing period using Cd fluorescence X-rays photoexcited by means of an X-ray tube, it is expected to bring the instrument online and to perform first measurements during the next month.

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[12] Polishing and optical contacting have been performed by the optical company Carl Zeiss, Germany.

[13] The components have been produced by the company Kohzu Precision Co. Ltd., Japan.