CRYSTAL X-RAY OPTICS FOR METROLOGY AND IMAGING

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Abstract

The contribution deals with crystal X-ray optics based on V-shaped channel-cut monochromators that possess additional functionality of the beam footprint control with direct implications for the X-ray metrology and imaging. The main limitation stems from the refraction effect that reduces severely the output intensity for high compression/expansion ratios due to a poor rocking curve matching of the two diffractors. In the contribution, two types of monochromators with different approaches to solving this issue were tested both in the compression and expansion modes with a laboratory microfocus X-ray source. The X-ray scattering and imaging experiments show that these X-ray optics elements provide a promising alternative with several advantages as compared to traditional measurement schemes.

Introduction

The two-bounce channel-cut monochromators have become an indispensable part of the X-ray optics. While traditional channels with parallel walls provide beam collimation and spectral beam shaping, V-shaped channels offer additional functionality of the beam footprint control, namely compression or expansion depending on the beam propagation direction (Fig. 1). The compression or expansion ratio *m* of one diffractor depends on the asymmetry angle of the diffraction used and is limited by the respective Bragg angle $_{\mathrm{B}}$ (Fig. 2). The ratio $m = m_1\ m_2$ of the V-channel monochromator is in practice limited by severely reduced output intensity for m>10 due to refraction that reduces the rocking curves matching of the two diffractors (channel walls). There are several possibilities to compensate refraction shift of the rocking curves based *e.g.* on temperature or concentration gradients or the beam deflection by a prism [1] that are, however, rather complicated for practical use. A better solution may be an advanced monochromator design with different asymmetries of the two diffractors [1].

The X-ray beam compression and expansion have obvious implications for X-ray metrology and imaging, respectively. For example, there are several commercial table-top setups for small-angle X-ray scattering (SAXS) and grazing-incidence SAXS (GISAXS) measurements with a microfocus X-ray source and traditional beam collimation based on the beam cutting by slits or pinholes which causes a considerable intensity loss. A modified Kratky camera has a similar problem. To achieve effective collection of the beam intensity produced by the laboratory microfocus X-ray sources, the beam compression instead of cutting is the first choice option. In the case of high-flux microfocus sources (rotating anode, liquid metal jet anode), the compressed beam intensity may even provide a laboratory alternative to some synchrotron measurements. The same holds true for laboratory X-ray imaging where the beam expansion helps to overcome resolution limitations imposed by the pixel size of the detector. Here, we present laboratory studies of the advanced V-channel monochromators with high compression /expansion ratios in both the compression and expansiom modes in order to test their performance for applications in the X-ray metrology and imaging.

Determination of the monochromator parameters

Two monolithic V-channel monochromators with the nominal compression/expansion ratios 15 and 21 for the CuK radiation were tested. V15 is a Ge (220) monochromator with different asymmetry angles of 19° and 4.28°. V21 is a compositionally graded GeSi (220) monochromator (asymmetry angle 15°). The monochromators were measured at a home-made GISAXS modular setup [2] with a microfocus I S source (Incoatec) equipped with collimating Montel optics of 500 rad full divergence and $7.5\ 10^{-2}$ bandwidth. The X-ray beam size was probed with a CCD camera (Photonic Science) of 6.4 6.4 m pixel size to determine the divergence and size of the beam diffracted in the compression mode. The beam intensity (photon flux) was measured in the single photon counting regime by 2D Pilatus 100K detector (Dectris, 172 172 m pixel size). No evacuation of the beam path was applied. The compression factors of 18 and 23 for V15 and V21, respectively, were

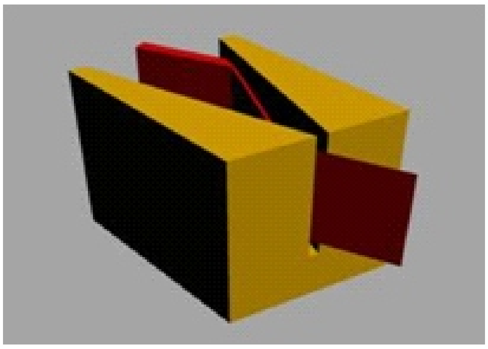

Figure 1. V-channel monochromator.

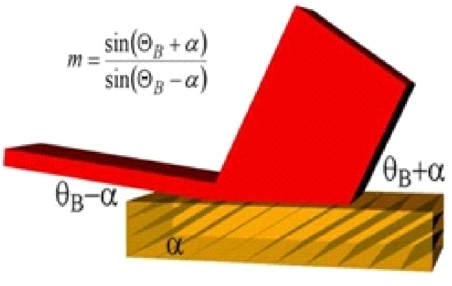

Figure 2. Asymmetric X-ray diffraction.

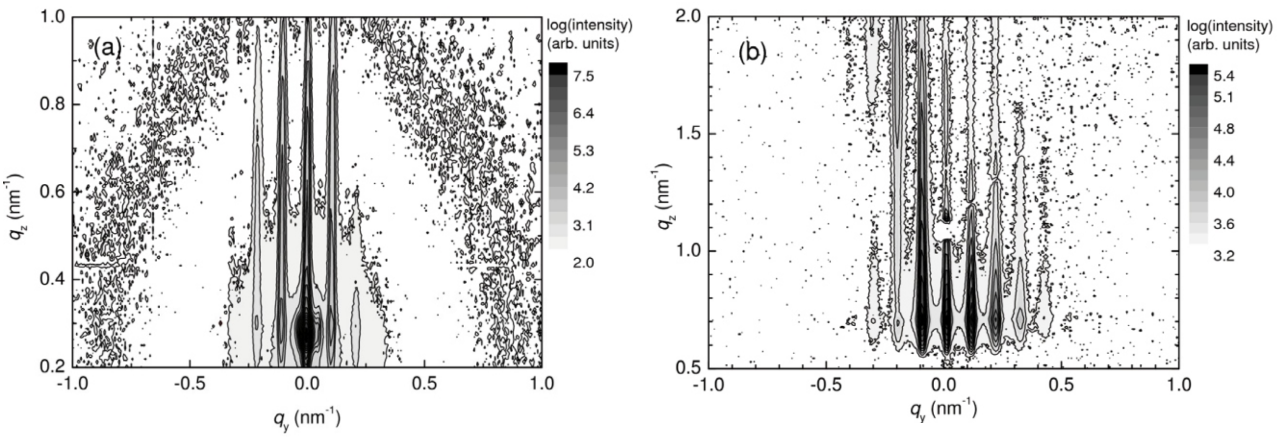

Figure 3. GISAXS patterns obtained on the silicon grating in laboratory (a) and at synchrotron beamline (b).

found. The larger values comparing with nominal ones are presumably due to the limited monochromator acceptance which is smaller than the Montel optics divergence. The photon flux of 10^8 cps was reduced by 2 orders of magnitude behind the monochromator.

Monochromator testing in the compression mode

The instrumental resolution, which can be achieved with V21 monochromator in the compression mode, was tested by GISAXS measurement of a silicon grating with ion-beam induced ripples at the modular setup described above. The angle of incidence of the X-ray beam was set to 0.2° and the ripples were oriented along the incident beam. The GISAXS signal collected by 2D Pilatus 100K detector for 1 hour (Fig. 3a) exhibits several truncation rods oriented along the q_z axis (surface normal). The spacing of adjacent truncation rods along q_y (parallel to surface) obtained by integration of the GISAXS pattern between $q_z = 0.5$ nm^{-1} and $q_z = 1$ nm^{-1} (Fig. 4) provides the lateral period $d = 2 / q_y = 58.573$ nm that compares well with 61.6 nm determined by the atomic force microscopy (AFM) previously [2]. The instrumental resolution of the GISAXS setup with V21 monochromator was evaluated from the zero order truncation rod at $q_y = 0$ nm^{-1} that was fitted with Gaussian function with FWHM of 0.013 nm^{-1}. This suggests the instrumental resolution = 2 / FWHM = 483 nm. The same silicon grating was measured also at the HASYLAB BW4 beamline (DESY, Hamburg) in the lower resolution mode with a beryllium compound refractive lens as focusing element. The GISAXS signal was collected by MarCCD camera for 1000 s at the angle of

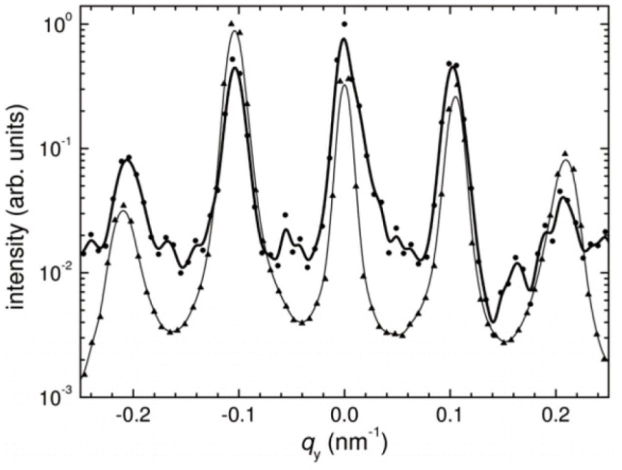

Figure 4. Lateral cuts of GISAXS patterns measured in laboratory (thick line) and at synchrotron (thin line).

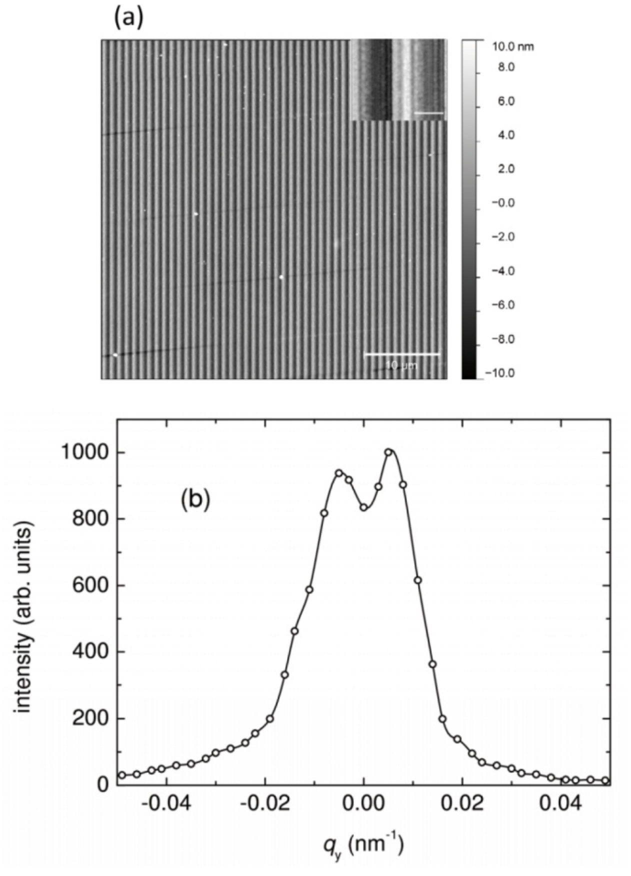

Figure 5. SPDT processed Si(100) surface probed by AFM with an inset showing in detail asymmetric shape of the grooves (a) that is presumably responsible for the shape of the lateral cut of the GISAXS pattern (b).

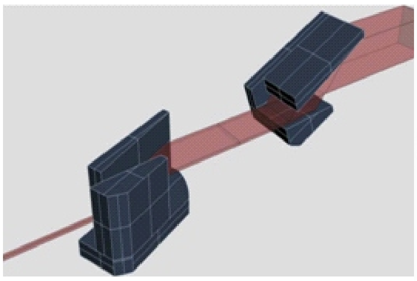

Figure 6. 2D Bragg magnifier.

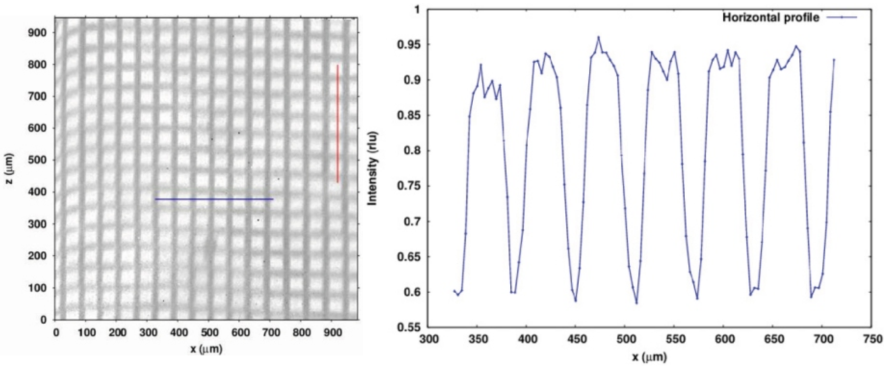

Figure 7. Image of a copper grid (left) and its horizontal profile (right).

incidence of 0.7° (Fig. 3b). The integration of GISAXS pattern between q_z values 1.5 nm^{-1} and 2 nm^{-1} is compared to the laboratory profile on the normalization to maximum intensities in Fig. 4. The higher background of the laboratory measurement is due to the air scattering in the absence of the beam path evacuation. The FWHM value of 0.012 nm^{-1} of Gaussian fit of the synchrotron zero order truncation rod suggests instrumental resolution of 523 nm which is only by 8 % larger than the laboratory value.

An issue of optimization is matching the beam size to the detector pixel size. For GISAXS, the acceptance is inherently limited by the finite sample size in the normal z direction, hence 1D compression in the transversal y direction is sufficient. This one is matched to the Pilatus 100K detector pixel size at the compressor-detector distance of 0.5 m and 0.6 m for V21 and V15 compressors, respectively, relying on their experimentally determined output parameters. At these optimized distances, an oversampling may be achieved by the detector rotation around the normal z axis resulting in a decrease of the apparent pixel size in the transversal y direction. A 50% reduction is achieved for the 60° rotation. The effect was tested with V21 monochromator on a float-zone grown Si(100) crystal processed by a single point diamond turning (SPDT) method [3] which produces highly regular grooves with   1   m periodicity as shown by AFM (Fig. 5a). The lateral cut of the GISAXS pattern taken at 0.2° incidence angle for 1 hour is depleted in the central region (Fig. 5b) that allows to resolve asymmetric truncation rods corresponding to the lateral period of   1   m, practically twice of the instrumental resolution 483 nm as determined above.

Monochromator testing in the expansion mode

Two V15 monochromators in the cross-coupled configuration (Fig. 6) were used as in-line 2D Bragg magnifier with a directly converting single-photon counting Medipix detector (55   55   m pixel size). The same modular setup with the microfocus X-ray source as in the compression mode was employed. The effective pixel size provided by V15 monochromators is 3.85   m. A copper grid used as the sample support in transmission electron microscopy was used for imaging (Fig. 7). The image shows sharp lines that are slightly bent due to imperfections of the diffractor surfaces that distort the X-ray wave front. Its horizontal profile shows a good signal-to-noise ratio despite a reduction of the photon flux behind the magnifier by 4 orders of magnitude. This result demonstrates applicability of the V-channel monochromators for laboratory X-ray imaging, especially when combined with directly converting X-ray detectors.

Conclusions

The V-channel monochromators with the compensated refraction shift allow an extreme X-ray beam compression/expansion with the factor exceeding 10. In the compression mode, a space-saving (GI)SAXS setup with short-distance collimation may replace inherently long slit or pinhole collimators with the resolution approaching a synchrotron beamline. The medium-resolution X-ray diffraction with a V-channel monochromator instead a parallel channel-cut monochromator combined with a slit is another application example. In the expansion mode, a reduction of the effective pixel size of a directly converting detector down to micrometer scale allows laboratory imaging with microfocus table-top X-ray sources where especially the high-power ones with rotating or liquid metal jet anode will open new possibilities.

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