Highly efficient blazed grating with multilayer coating for tender X-ray energies

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Abstract: For photon energies of 1 – 5 keV, blazed gratings with multilayer coating are ideally suited for the suppression of stray and higher orders light in grating monochromators. We developed and characterized a blazed 2000 lines/mm grating coated with a 20 period Cr/C- multilayer. The multilayer d-spacing of 7.3 nm has been adapted to the line distance of 500 nm and the blaze angle of 0.84° in order to provide highest efficiency in the photon energy range between 1.5 keV and 3 keV. Efficiency of the multilayer grating as well as the reflectance of a witness multilayer which were coated simultaneously have been measured. An efficiency of 35% was measured at 2 keV while a maximum efficiency of 55% was achieved at 4 keV. In addition, a strong suppression of higher orders was observed which makes blazed multilayer gratings a favorable dispersing element also for the low X-ray energy range.

OCIS codes: (340.0340) X-ray optics; (050.1950) Diffraction gratings; (230.4170) Multilayers.

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1. Introduction

Crystals and gratings are used in monochromators at synchrotron radiation sources since about five decades to filter out wavelengths for experiments in the hard and soft x-ray photon energy range. Although it was about 30 years ago that the gap between grating and crystal monochromators has been closed at 2 keV, the performance of both types of monochromators is still problematic in this range. Between 1.5 keV and 2.5 keV, sometimes also called the "tender x-ray range", there is still a grey zone left where none of the monochromator types performs satisfactorily.

Above 1 keV, grating monochromators require very shallow grazing angles of incidence in order to keep a high reflectivity. This means the gratings have a small acceptance and they are working close to the total external reflection angle, which pulls off intensity from +/− 1st and other diffraction orders. E. g. a 2000 l/mm blazed grating reaches barely 5% efficiency at 2 keV. In addition, the amount of stray light increases above 1.5 keV when the angular spacing between first and zero order becomes very small.

Approaching this grey zone from the high energy side (below 4 keV) with crystal monochromators one has to switch to a HV- or UHV-compatible design because of significant losses of the radiation due to absorption in air. And below about 2.5 keV the almost normal incident beam on the crystals causes serious heat load problems and hence instabilities.

One of the most promising candidates for dispersing elements for this region is to cover gratings of different kinds with multilayers [1–5]. This changes a normal surface grating into a crystal-like volume grating with much increased efficiency. In addition, the angle of incidence can be made larger without losses in reflectivity, respectively efficiency, and hence increasing the acceptance. The multilayer coating can be applied to lamellar gratings as well as to saw-tooth (blazed) gratings.

The simplest lamellar multilayer grating can be manufactured by lithographically exposing a resist on a multilayer coated substrate with either e-beam writing or holographic exposure followed by reactive ion etching [6].

Recently a more sophisticated manufacturing process led to a lamellar multilayer with a high efficiency (up to 27% at 2.2 keV) over a large energy range [7]. Here a lamellar grating was coated with a B_4C/Mo_2C-multilayer, which had to have precisely the layer thickness as the height of the lamellar profile. This grating then successfully has been installed in a monochromator at SOLEIL [8]. Unfortunately the Pt- and Mo-absorption edges of the mirrors and grating obscured the advantages of such a volume grating in the high energy range.

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The manufacturing of a blazed multilayer grating is even more complex. A saw-tooth profile has to be ruled into a gold coated surface. Reactive ion etching then transfers the profile into the substrate. The period of the multilayer again then has to fulfil certain conditions in order to fit to the blaze angle [4]. This kind of grating is subject of this paper.

Another not less complex possibility for the production of small blazed multilayer gratings is to anisotropically etch asymmetrically cut silicon crystals. This leads to precise and smooth saw-tooth profiles and an efficiency of 52% has been achieved at 13.4 nm [9].

In the following chapter we describe the design considerations for the blazed multilayer grating as well as manufacturing steps and the conditions for the measurements. After that the results of the measurements will be presented and compared with calculations. Finally in the last chapter, we will summarize the results and give an outlook for future applications.

2. Design considerations and technical realization

For combining a grating and a multilayer both the grating-equation and the multilayer-equation have to match. The grating-equation for on-blaze condition can be written as

$$m\lambda = 2d_G \sin \Upsilon \sin \theta, \tag{1}$$

where m is the diffraction order, λ is the wavelength, d_G is the line spacing, ϒ is the blaze angle and 2θ is the deviation angle of the diffracted light.

The multilayer-equation can be written as

$$n\,\lambda = 2\,d_{ML} \sin \theta \left(1 - \left(2\,\delta\right) / \left(\sin^2 \theta\right)\right)^{1/2}, \tag{2}$$

where n is the order, d_{ML} is the period, δ is the average refractive index decrement of both materials and 2θ is the deviation angle of the diffracted light.

For short wavelength the Eq. (2) can be approximated by the simple Bragg-equation

$$n\,\lambda \approx 2\,d_{ML} \sin \theta. \tag{3}$$

At 2000 eV the approximation is good to 7%, i.e. θ needs to be corrected by 7%.

Combining Eqs. (1) and (3) one gets a relation (4) between blaze angle, multilayer period and line spacing which has to be fulfilled if multilayer and grating should act together constructively

$$d_{ML} \approx \left(\mathrm{n}/\mathrm{m}\right) d_G \mathbf{sin}\Upsilon. \tag{4}$$

Since it was intended to optimize the multilayer blazed grating (MLBG) for first order and to use the multilayer in the most efficient 1st order, this relation simplifies to

$$d_{ML} \approx \left(\mathrm{n}/\mathrm{m}\right) d_G \sin\Upsilon. \tag{5}$$

In order to get a most realistic test grating with a reasonable energy resolution in the range of 2 keV, a line number of 2000 l/mm has been chosen. The grating has been ruled into a thick gold layer on top of a 0.7 mm thick Si wafer using the ZEISS ruling engine GTM6 which is in operation at HZB since 2012 [10]. The ruled blaze angle was 6°, the anti-blaze angle 25° (see Fig. 1).

After the ruling process the grating structure was reactive ion etched into the silicon substrate. Due to the different etching rates of gold and silicon the blaze angle reduced to 0.84° in silicon. With Eq. (5) the required period of the multilayer was determined to 7.3 nm.

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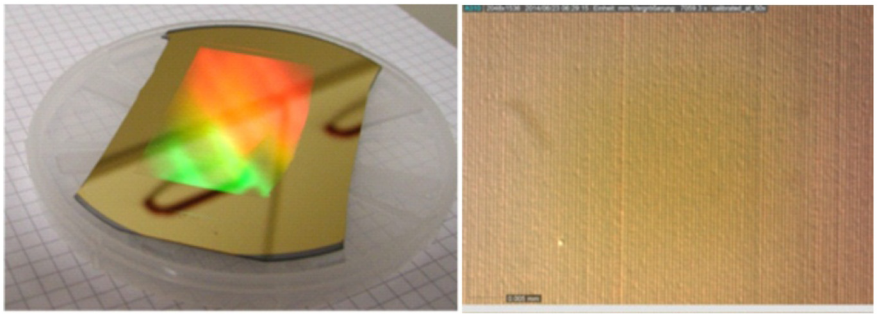

Fig. 1. After a 6 days ruling process a 30 mm by 60 mm large grating with 2000 l/mm and a blaze angle 6° has been achieved (left). The lines with a distance of 500 nm are barely visible with a microscope (right).

Chromium and carbon have been selected as the material combination for the multilayer since neither of them has absorption edges between 0.6 keV to 5.9 keV. A 7.3 nm Cr/C multilayer with the thickness ratio of 0.6 (carbon layer thickness to d-spacing) can provide a high theoretical reflectivity of 26% to 90% from 1keV to 5.9keV, respectively. The experimentally fabricated Cr/C multilayer also shows sharp interfaces [11]. Although a number of bilayers of 40 yields a maximal reflectivity, 20 periods was used in the deposition - hence 5% less reflectivity (relatively) at 2.5 keV in theory- in order to limit the smoothing effect of the grating profile during deposition. No interface roughness or diffusion was taken into account in the reflectivity calculations here.

The deposition of the multilayer has been done simultaneously on the grating and a witness sample at the University of Twente. The witness sample was deposited on a super-polished Si wafer positioned near the grating. Both chromium and carbon were deposited using direct current magnetron sputtering. For the first experiment, the thickness uniformity of the witness sample was not optimized which make the d-spacing about 8% thinner than the multilayer deposited on the grating. After the multilayer deposition AFM measurements showed a rms roughness of only 0.2 nm on the facets of the MLG (Fig. 2, top). The AFM used to measure the groove profiles is a Bruker SIS-Ultraobjective. The tip applied is a silicon SPM-sensor for non-contact mode, resonance frequency: 190 kHz / force constant: 48 N/m. The tip is shaped like a polygon based pyramid with a tip-radius of less than 8nm. Thus the achievable spatial resolution is in the range of 10-15nm as has been shown by recent work [12]. Despite the deposition the blaze angle of 0.84° did not change (Fig. 2, bottom).

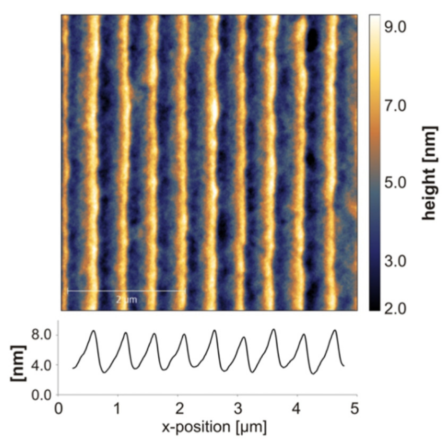

Fig. 2. A 5 x 5 μm^2 view field with a roughness 0.2 nm on the facets (top) and an integrated (1 µm along the lines) line cross section showing the blaze profile (bottom).

Since this multilayer grating was designed for covering the upper part of the soft x-ray range and the lower part of the hard x-ray range i.e. "tender x-ray range", the measurements had to be carried out at two different types of beamlines. For the low energy range we chose the new optics beamline PM-1 [13] from 0.6 keV to 1.8 keV with the stationary new reflectometer [14] at BESSY II and for the high energy range the double crystal monochromator KMC-1 [15] from 2 keV to 4.8 keV with the mobile small reflectometer [16].

3. Results and calculations

To achieve an angle resolution of about 0.05 degrees, an entrance slit of 125 µm was placed in front of the detector (GaAsP-photodiode). Figure 3 shows an angular detector scan in the plane of the incident beam at a fixed photon energy of 2.2 keV and fixed 1.7° angle of incidence on the grating. The measured signal (I) has been normalized to the intensity of the primary beam (I0), which regularly was recorded by the same detector when the sample was moved out of the light axis.

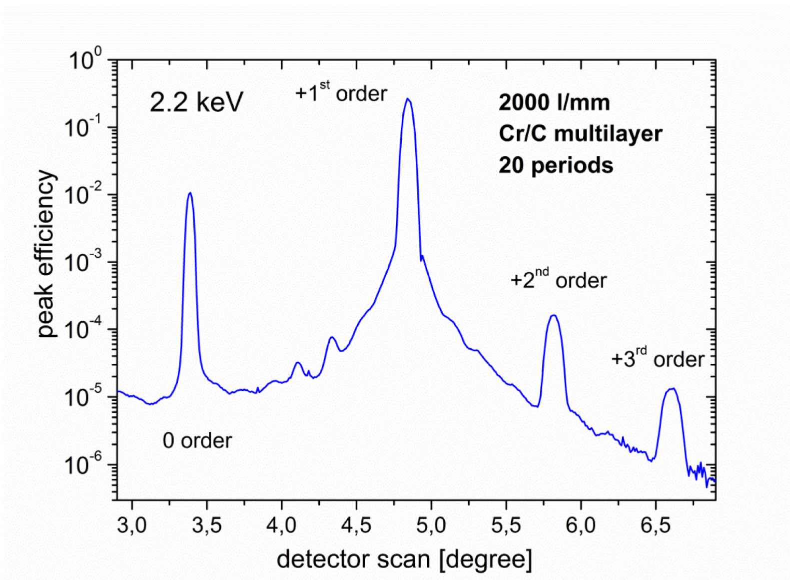

Fig. 3. The normalized angle dependent signal from the multilayer blazed grating for 1.7° fixed incidence angle and at 2.2 keV photon energy is shown. The + 1st order dominates the higher orders by more than three orders of magnitude. Even the 0th order signal is a factor of 20 lower.

Because of the grating dispersion the peak width changes as one goes away from zero order. For that reason the efficiency had to be calculated by dividing the integrated peak area, e.g. of the + 1st order by the integrated area of the I0-peak. The efficiency of the + 1st order at 2.2 keV was determined to be 39.0%. For the simulations the commercial "modal transmission line theory" code DiffractMOD [17] has been used. For a blaze angle of 0.84° and an anti-blaze of 2.3°, but without roughness an efficiency of 44.9% has been calculated at this energy. The reason for the measured efficiency loss of 13% (relatively) is assumed to be an interface roughness/diffusion contribution from the multilayer and or a smoothing of the saw-tooth profile, although a small (measured) anti-blaze angle of 2.3° was used for the calculation which should already account for the latter.

In order to help clarifying these questions, the witness multilayer was measured at the same energies. As usual for multilayer investigations, sample and detector were scanned in a θ-2θ-mode (Fig. 4).

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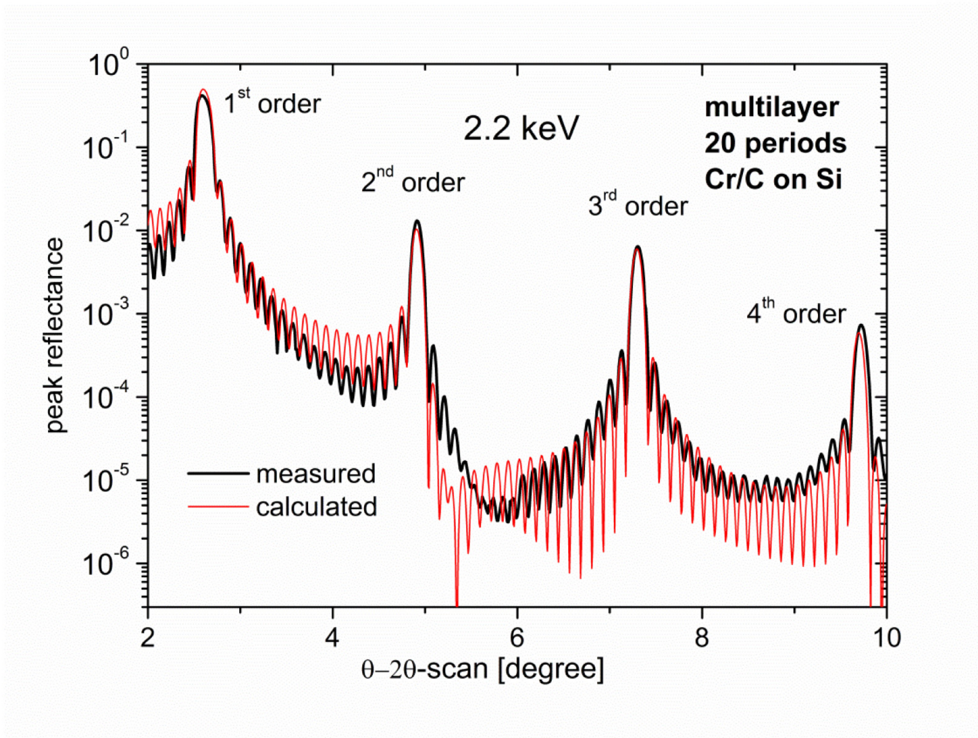

Fig. 4. An angular (θ-2θ)-scan shows the peak reflectance of a 20 periods witness multilayer at 2.2 keV.

For these measurements the detector was used without slit and the reflectance was determined by dividing the peak intensity by the peak I0-signal. The reflectance curve of the witness multilayer measured at 2.2 keV is shown in Fig. 4. A maximum peak reflectance of 45% has been measured in 1st multilayer order. The curve was fitted using the IMD software [18] to analyze the layer structure. A two layer model was used in the fitting procedure while the layer density, thickness and interface width were set as fitting parameters. It was found that the layer density of both Cr and C are close to bulk materials while an interface width of 0.44 nm and 0.32 nm were obtained for Cr-on-C and C-on-Cr interface, respectively [11].

Simulations also accounting for the measured roughness of the multilayer of 0.17 nm rms (AFM measurement) gave instead a peak reflectance of 60% at 2.2 keV. The simulations carried out with DiffractMOD have been compared with the BESSY code REFLEC [19] and both showed equal results.The efficiency and the reflectance measurements were performed over a large photon energy range from 0.6 keV to 4.8 keV.

Figure 5 shows the spectral distribution of the efficiency of the multilayer coated grating. A very high efficiency can be observed for photon energies above 2 keV. At 4.07 keV it reaches 55%, which is to our knowledge the highest value ever measured in the medium to hard x-ray range. Over almost the whole range the simulated results are higher than the measured, which we already assigned to diffusion at the interfaces. Above 4 keV calculated and measured efficiency starts to drop. In this range the angle of incidence becomes smaller than 0.6° and hence approaches the range of total external reflection. This is also confirmed by the increase of the measured and calculated efficiency of the 0th order (Fig. 5, stars and red line). Above 4 keV the difference between measurements and calculations seems to vanish. It is unclear whether or how this is correlated with the onset of the total external reflection.

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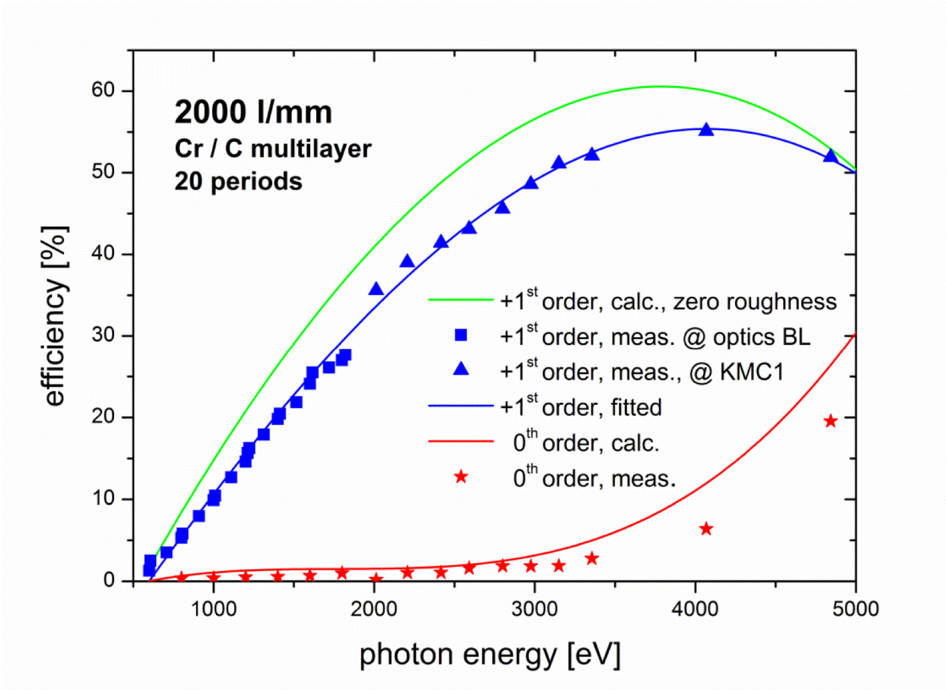

Fig. 5. First diffraction order efficiency measurements from 0.6 keV to 4.8 keV are shown (squares & triangles) as well as a curve fit for the measurements (blue line) and compared to calculated + 1st order efficiencies (green line). Stars and red line indicate results for 0th order measurements and calculations.

The witness multilayer also has been measured over a large energy range and provided useful information. As can be seen in Fig. 6 the calculated and the measured reflectance differ with increasing photon energy. The calculated reflectance for 6.7 nm period thickness without roughness and for the measured 0.17 nm rms roughness are practically identical. For this reason, we can either exclude roughness as originator for the deviation between measured and calculated reflectance and one could assume most likely diffusion at the interfaces as reason for the losses or the AFM results underestimate a higher roughness in the high spatial frequency range below 15 nm which is essential in the high photon energy range. A Debye Waller fit would yield a roughness of 0.5 nm bellow 2 keV and 0.8 nm to 1.1 nm above 2 keV.

The measured reflectance around 2 keV shows clear deviations from the fit for all measured results (see also Fig. 5) indicating problems at both beamlines in this range. For the Optics Beamline it was the strong increase of stray light which falsified the results above 1.8 keV. At the KMC-1 beamline it was difficult to achieve thermally stable conditions at the first Si-crystal between 2 keV and 2.3 keV because of its almost normal incidence angle to the power of the synchrotron radiation. So, not accidentally we encountered a problem what this multilayer grating is intended to solve in the future.

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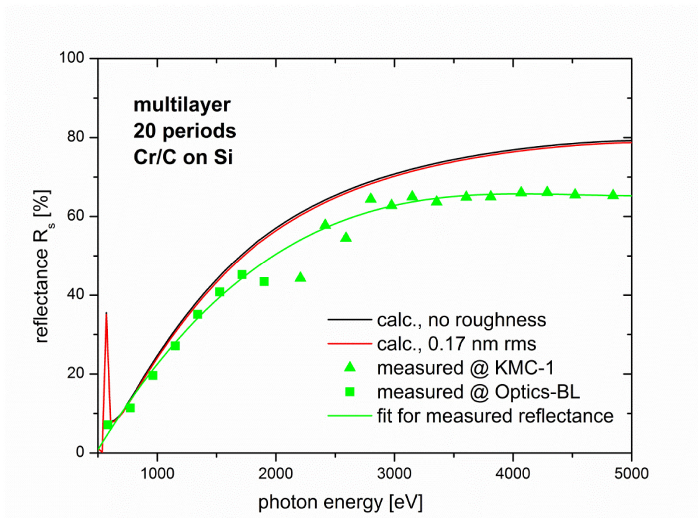

Fig. 6. Calculated reflectance of the witness multilayer without (black line) and with the measured 0.17 nm roughness (red line) as well as the measured reflectance (squares) is shown. The green curve indicates a fit for the measurements.

If the measured efficiency of the multilayer grating is divided by the reflectance of the multilayer one gets the relative efficiency of the grating, which should be a material-independent information about how well the grating equation and the Bragg equation (which is only an approximation) work together. In Fig. 7 the measured relative efficiency is shown with considerably high values over a large range of 4 keV and with a maximum of more than 80% above 3.1 keV. But, the smaller period of the witness multilayer of 6.7 nm instead of 7.3 nm leads to a reduction of the reflectance by up to 5.5%. For this reason the relative efficiency curve should be corrected (reduced) by maximal 5.5% (high energy end).

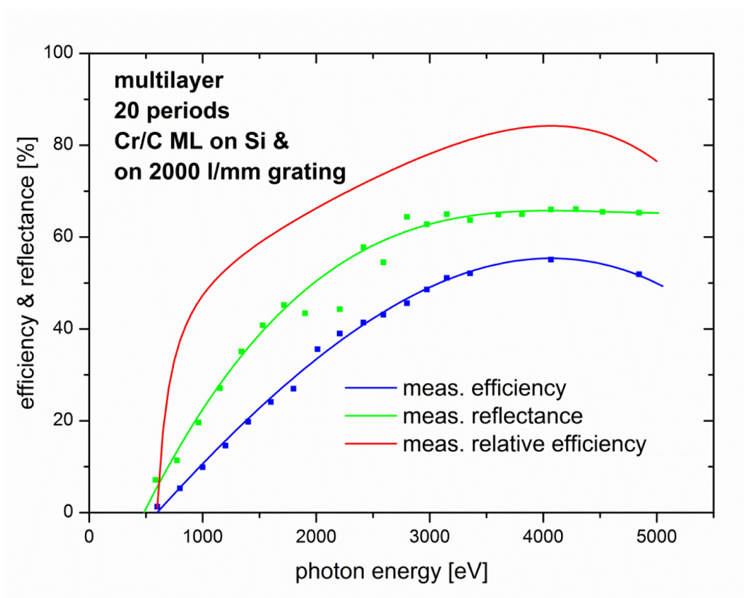

Fig. 7. The measured efficiency of the MLBG (blue) divided by the reflectance of the ML mirror (green) yields the material-independent relative measured efficiency (red).

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Nevertheless, the still high relative efficiency indicates that the approximation made in "Eq." (5) holds sufficiently well from 1 keV to 5 keV. The drop from 4 keV towards 1 keV can probably be assigned to the neglected correction to the Bragg-equation (Eq. (2)). This has an increasing effect for lower photon energies. However, since these corrections are different for different materials, the shape of this measured relative efficiency might still depend on the materials used. The drop above 4 keV is caused by the onset of the total external reflection as mentioned before.

Knowing that blazed gratings exhibit relatively large efficiencies for higher orders we doubled the incident photon energy for all measured points, but kept the incidence angle the same. The second order then should appear at the same detector angle where the first order showed up for the half energy before.

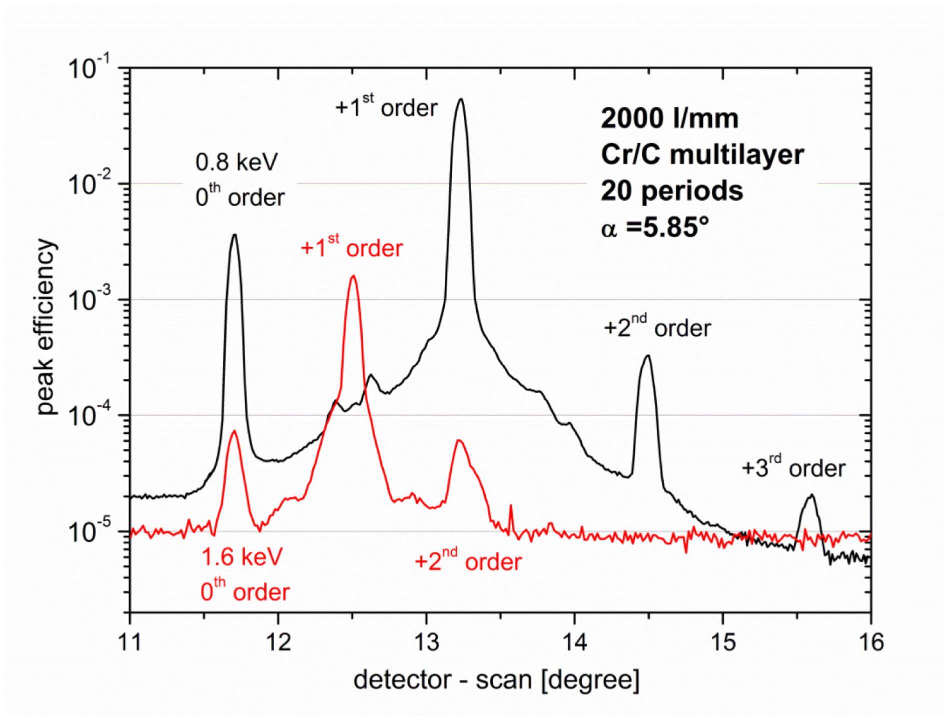

Fig. 8. Detector-scan for the energy 0.8 keV (black) and the double energy 1.6 keV (red). The incidence angle was kept fixed at 5.85°. Each spectrum has been normalized to its I0-flux.

In Fig. 8 the zero orders for 0.8 keV and 1.6 keV are visible at the same angle since the angle of incidence was kept constant at 5.85°. The first order for 0.8 keV appears at 13.2° as expected with high intensity. The first order for 1.6 keV shows up at 12.5° and the second order for this energy appears at 13.2°. But, in contrast to normal blazed gratings the second and also higher orders are several orders of magnitude suppressed. This can be accredited

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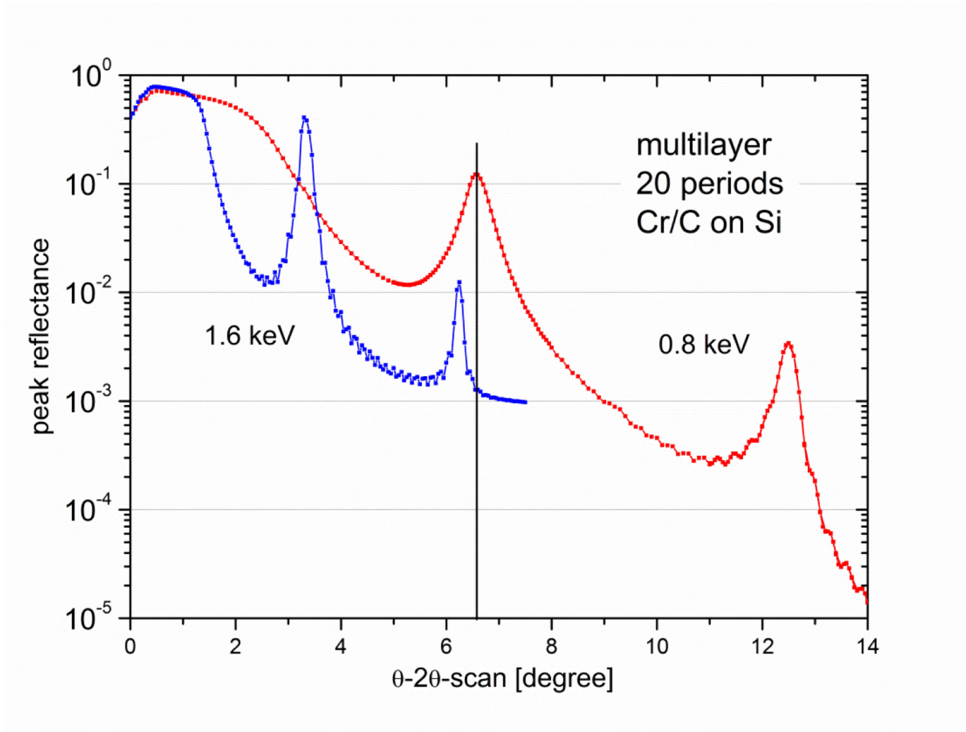

Fig. 9. A normalized (θ-2θ)-scan for 0.8 keV and for 1.6 keV shows that the first multilayer order at 0.8 keV does not coincide with the second order of 1.6 keV. The shift is caused by the difference of the refractive indices at different photon energies.

to the optical properties of the multilayer. The different refractive index of the layers (Eq. (2)) at the higher photon energy prevents that the second order peak of the second order energy appears at the same angle as it is normally the case for gratings and crystals. The shift observed of the 2nd order peak for the multilayer is about 0.4° in Fig. 9 and hence is responsible for the strong higher order suppression of the multilayer grating. The strong suppression of second and higher orders has been observed at all measured photon energies from 0.8 keV to 4.8 keV.

4. Summary and outlook

It has been shown that the effort to change a surface grating into a volume grating by coating it with a multilayer leads to a strong increase of the efficiency of about one order of magnitude. In addition, due to a larger possible angle of incidence the acceptance can be increased. Very interesting is the strong suppression of higher orders which also for grating monochromators working at lower photon energies might be a very attractive feature. Also the material choice Cr/C turned out to be optimal for the tender x-ray range. All these features make multilayer gratings attractive for future monochromator designs.

We have seen that multilayer gratings can be used over a broad energy range in monochromators, where incidence and diffraction angle can be tuned independently as it is the case for 15 collimated Petersen monochromators at BESSY II [20]. In this case the rotating plane mirror has to be covered with a multilayer of the same materials and period as the grating. In order to exploit the energy range above 2 keV one also should reduce the angle of incidence at all beamline mirrors down to slightly below 1°. Pt- and Au-coatings are inappropriate and should be changed or switchable to Rh, Ru or Cr instead. Provisions of this

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kind are already made at BESSY II in order to install a multilayer grating at the UE30-XM beamline in 2017.

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