Front. Electr. Electron. Eng. China 2008, 3(1): 105–109
DOI 10.1007/s11460-008-0021-7

RESEARCH ARTICLE

Hongjun ZHOU, Jinjin ZHENG, Tonglin HUO, Guobin ZHANG, Zeming QI, Pengfei ZHONG

Quantitative research on higher order harmonics in metrology beamline

Abstract The synchrotron radiation spectra of the spherical grating monochromator (SGM) working in the soft X-ray and VUV region are often contaminated by significant amounts of higher order harmonics. They cannot be suppressed completely by suitable filters. Higher order contributions in the spectral radiation standard and metrology beamline were researched using transmission grating (made in-house) and IRD AXUV100G (USA) photodiode detector. The exit beam was dispersed with the transmission grating behind the exit slit of the monochromator, and the contributions of the different orders were analyzed. The higher order distributions were quantitatively determined for three gratings with line densities of 1800, 600 and 200 l/mm. Experiment results show that in wavelengths between 5 nm and 15 nm the contributions of the higher orders to the detector signal are restricted to less than 7% even without the use of filters. In wavelength regions between 5 nm and 34 nm, the contributions of the higher orders to the detector signal are less than 14% with proper Al, Si_3N_4 and Zr filters, and after being modified by quantum efficiency of the detector, the higher order contributions are restricted to less than 6.5%. The study also shows that higher orders are almost totally suppressed by MgF_2 filter when the wavelength ranges between 115–140 nm.

Keywords spectral radiation standard, higher order harmonics, spectral radiation metrology

Translated from *Optics and Precision Engineering*, 2007, 15(5): 640–645 [译自: 光学精密工程]

Hongjun ZHOU (✉), Tonglin HUO, Guobin ZHANG, Zeming QI
National Synchrotron Radiation Laboratory, University of Science and Technology of China, Hefei 230029, China
E-mail: hjzhou@ustc.edu.cn

Jinjin ZHENG, Pengfei ZHONG
Department of Precision Machinery and Precision Instrumentation, University of Science and Technology of China, Hefei 230026, China

1 Introduction

A synchrotron radiation beam continuously passes through the entrance slit, irradiates on a grating monochromator and is then dispersed. However, the exit monochromated beam λ is always contaminated by higher order harmonics $\lambda_n = \lambda/n$, since the higher order harmonics diffraction angle is in the same direction according to the grating function $d\sin(\alpha+\beta) = m\lambda$. The presence of higher orders in monochromated synchrotron beams is a universal problem.

International research on higher order contribution suppression has been conducted by several researchers and laboratories, such as W. R. Hunter and M. Kühne et al. [1,2]. Kühne conducted research on higher order contributions in the synchrotron radiation spectrum of a toroidal grating monochromator determined by the use of a transmission grating. A thin-film reflecting interference filter was designed and built by W. R. Hunter and J. P. Long for the purpose of suppressing grating harmonics when monochromatizing synchrotron radiation. For the higher energy region, a glass capillary array was used to suppress higher order harmonics by A. Erko [3], while T. Matsushita [4] did research on mismatching the second crystal of the double crystal monochromator to suppress higher order harmonics. At Advanced Light Source (USA), a differentially pumped harmonic filter was developed by A. G. Suits [5] in the chemical dynamics beamline, while a triple-mirror "order suppressor" was incorporated in the reflectometry and scattering beamline (6.3.2) by E. M. Gulliksion [6], etc.

VUV can be absorbed easily and it is difficult to find out proper filters in the region of 35–115 nm to suppress higher orders. Even if suitable filters in the specified wavelength regions are used, the higher orders can be suppressed to some extent, but in general some contributions will still remain. It is imperative to know the higher order distributions quantitatively to raise the metrology accuracy.

This investigation aims to determine the higher order contributions of spherical grating monochromator

(SGM) in spectral radiation standard and metrology beamline (U27) by the use of 840 l/mm free-standing transmission grating (TG). The wavelength region is between 5–140 nm. The detector used here is AXUV100G photon diode [7–10] (PD) made by IRD company. Research is also conducted on the higher order suppression efficiency of Al, Si_3N_4 and Zr filters in the region of 5–34 nm. The results of higher order suppression by MgF_2 in the region of 115–140 nm are presented.

2 Structure of SGM branch in U27

U27 beamline is dedicated to VUV radiometry in the Second Phase Project of the National Synchrotron Radiation Lab (NSRL). SGM is one branch of U27 used to measure the performance of the optical component and calibrate detector. Figure 1 shows the optical structure of the SGM branch. A toroidal focusing mirror (TM1) can image synchrotron radiation (SR) into the entrance slit (S1) of SGM, with S2 an exit slit and TM2 a postposition toroidal focusing mirror. The SGM is equipped with three Laminar spherical gratings of 1800 l/mm, 600 l/mm, and 200 l/mm which cover 5–12 nm, 12–34 nm, 34–140 nm respectively. The detector (detec) can rotate by 180° around the axis of the sample stage.

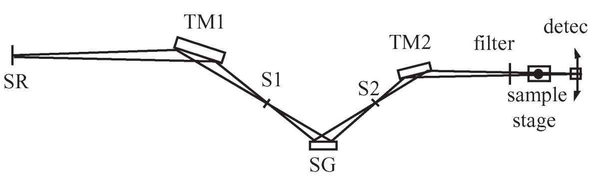

Fig. 1 Sketch map of SGM branch optical system

3 Higher-order distribution research

The purpose of constructing the U27 beamline and endstation is to set up a soft X-ray and EUV national metrology standard. The endstation is equipped with a reflectometry [11] for measuring optical performance and a rare gas ionization chamber as the absolute detector [12–15]. The purity of spectra directly affects the accuracy of calibration.

3.1 Research method

To determine the higher order contributions with an acceptable degree of uncertainty, a transmission grating (TG) (shown in Fig. 2) was chosen to disperse the exit beam behind the SGM. This allows one to determine the fraction of higher orders at every desirable wavelength.

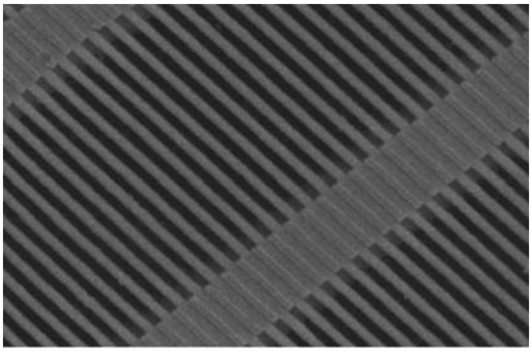

Fig. 2 SEM image of 840 l/mm transmission grating

For the wavelength in the region of 5–34 nm, an 840 l/mm free-standing gold TG is inserted in the filter holder position (Fig. 3) to increase the angular resolution of the detector. SGM is set to a constant wavelength and an angular scan of photon diode (PD) detector is performed in the diffraction plane. The detector rotates around the axis of the sample stage and records the zero-order at 0° diffraction angle and higher orders of TG. The measured diffraction angle β can be calculated by geometric relations and the known detector angle 2φ:

$$\beta = \arctan\left(\frac{2b\tan\varphi}{a+b}\right).$$

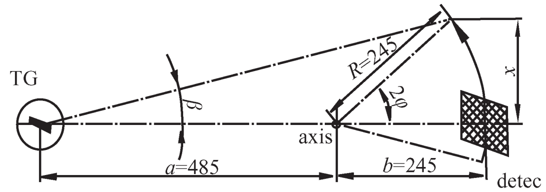

Fig. 3 Geometric structure of TG and detector

In theory, according to the grating formula:

$$d\sin(\alpha_T+\beta_T)=m\left(\frac{\lambda}{n}\right) \qquad (m=0,1,2,\ldots;\ n=1,2,3,\ldots),$$

$$\beta_T = \arcsin\left(\frac{m\lambda}{dn}\right) = \arcsin\left(\frac{0.84m\lambda}{10^3 n}\right).$$

α_T is SR incidence angle ($\alpha_T = 0$), d is grating period. β_T is the theory diffraction angle when the wavelength is λ. The orders of diffraction peaks can be determined by comparing β and β_T.

When $n = 1$, λ/n is called base-wavelength λ. Different m corresponds to different diffraction orders.

When $n \geqslant 2$, λ/n is called higher order harmonics of base-wavelength λ.

The TG is installed in axis position (Fig. 3) when measuring higher order contributions in the region of 34–140 nm. Then the rotation angle 2φ of the detector is the diffraction angle β.

3.2 5–34 nm higher-order distributions

In the region of 5–34 nm of the wavelength, proper filters are available for suppressing the higher orders. The higher orders were measured under two conditions: without any filter, and with proper filters in this region. The filters of Zr (200 nm), Al (200 nm), and Si_3N_4/Mo/Si (100 nm/50 nm/200 nm) made by Tongji University (China) are used at certain wavelengths.

Figure 4 depicts the intensity curves of detector scan without any filter with the wavelength between 6–12 nm, from which it can be seen that the first-order diffraction angles increase with the wavelength. Figure 5 (a) is the intensity curve of 12 nm and (b) is an amplified version of its shadow area. The first-order diffraction angle is at 1.7 degrees, and the small peak at 0.85 degree denotes the higher order peak. The higher order intensity contributions can be obtained by integral. After the detector signal has been corrected by the quantum efficiency (QE), the contributions of the higher orders presented in the spectrum can be determined.

Through repeating the experiment with filters Zr (200 nm), Al (200 nm), and Si_3N_4/Mo/Si (100 nm/50 nm/200 nm) at certain wavelengths, it can be seen that the higher orders can be suppressed efficiently. Figure 6 demonstrates signal curves of 20 nm both with and without Al filter.

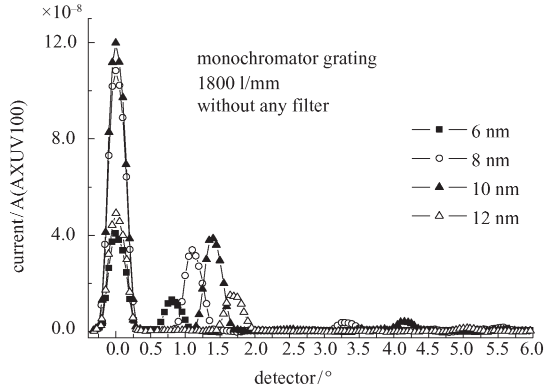

Fig. 4 Intensity curves done by detector scan while wavelength is between 6–12 nm, the step is 2 nm

Figure 7 shows higher order contributions in the region of 5–34 nm in different conditions. Curve 1 shows the higher order intensity contributions of SGM without any filter. After modification by the PD quantum efficiency, curve 2 is obtained. Curve 3 shows the higher order intensity contributions with the proper Zr, Si_3N_4/Mo/Si, Al filters, while curve 4 shows the result with the proper filters after modification by the PD quantum efficiency.

According to the result of experiment, the three filters' working regions are shown in Table 1.

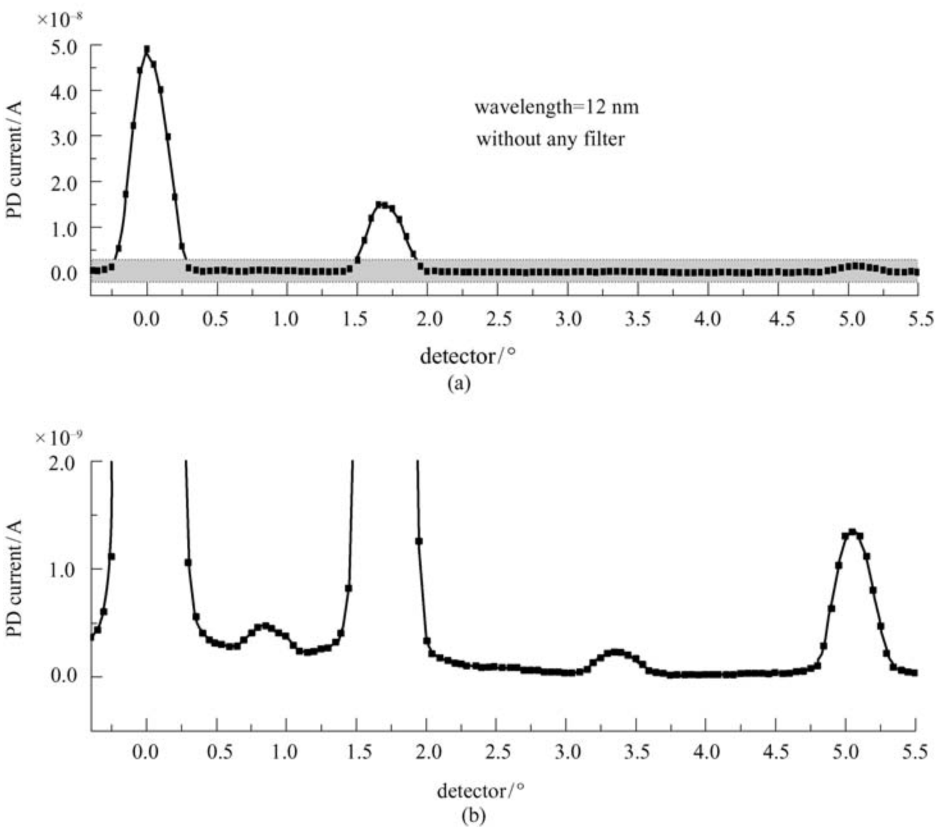

Fig. 5 Intensity curves of 12 nm wavelength

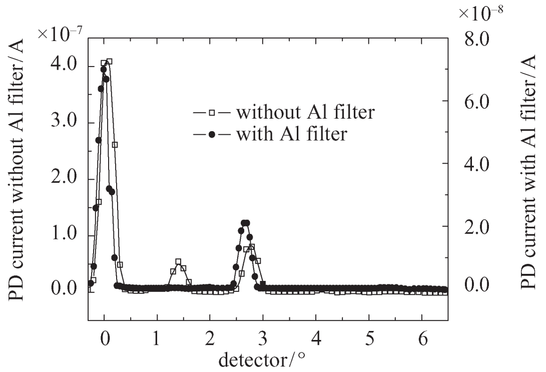

Fig. 6 Intensity curves of 20 nm with and without Al filter

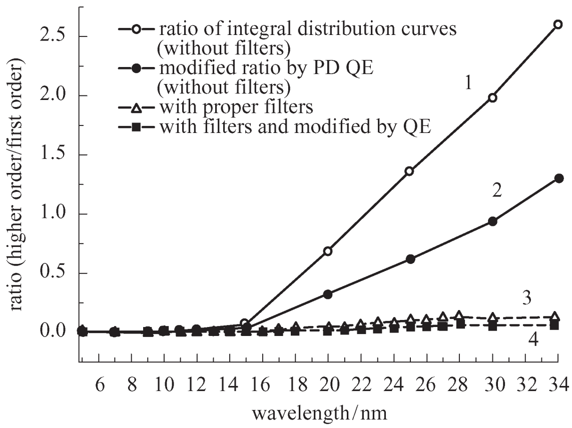

Fig. 7 Ratio of higher order to first order

Table 1 Wavelength region of suppressing higher orders by Zr / Si_3N_4/Al filters

filter	λ region /nm	higher order/ base λ	contributions after modified by QE
–	5–9	0%	0%
Zr	10–12	< 3.4%	< 1.2%
Si_3N_4	13–20	< 12%	< 5.5%
Al	21–34	< 14%	< 6.5%

3.3 34–140 nm higher-order distributions

Region of 34–140 nm is covered by the 200 l/mm grating. Figure 8 shows the intensity curve of the TG diffraction with the wavelength at 40 nm. It is evident here that the higher orders are more complex when the wavelength is longer than 34 nm.

Figure 9 shows the higher order contributions of SGM in the region of 34–140 nm. Curve 1 is measured by NSRL utilizing TG and PD (AXUV100G); Curve 2 is measured by Sergey V. Kuzin (P. N. Lebedev Physical Institute, Russia) utilizing TG and CCD (E2V, UK).

In the region of 115–140 nm, MgF_2 filter is inserted in the beam to suppress higher orders. Figure 10 shows intensity curves at the wavelength 127.5 nm with and without MgF_2 filter. It can be seen that higher orders are almost totally suppressed in this region by using MgF_2.

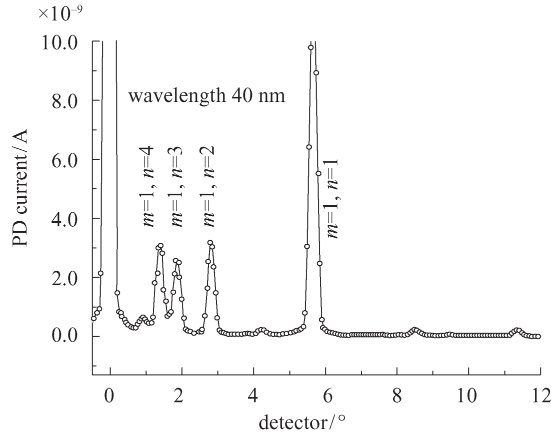

Fig. 8 Intensity curve of 40 nm wavelength

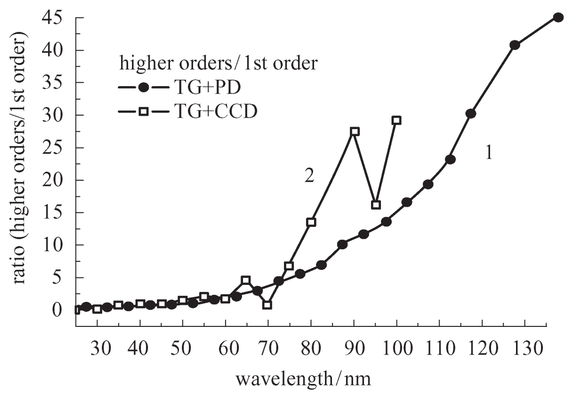

Fig. 9 Ratio of higher order to first order with 34–140 nm wavelength, the step is 5 nm

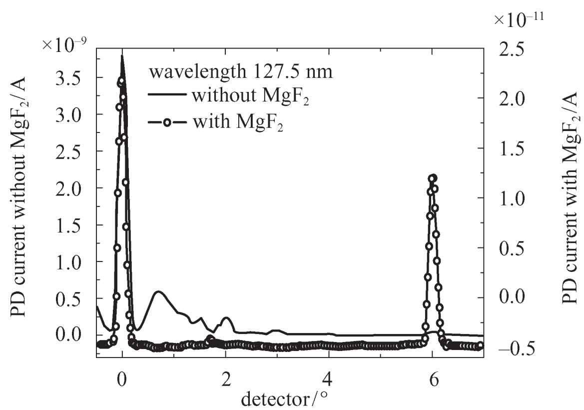

Fig. 10 Intensity curves at wavelength of 127.5 nm with and without MgF_2 window

4 Conclusions

Research results show that when the wavelength region is between 5 and 15 nm, the contributions of the higher orders to the detector signal are restricted to less than 7% even without the use of filters. When the wavelength region is between 5 and 34 nm, the contributions of the higher orders to the detector signal are less than 14% with Al (200 nm), Si_3N_4/Mo/Si (100 nm/50 nm/200 nm) and Zr (200 nm) filters. After being modified by quantum efficiency of the detector, the higher order contributions are restricted to less than 6.5%. The research also shows that higher orders are almost totally suppressed by MgF_2 filter when the wavelength is between 115–140 nm. In the region of 36–115 nm, higher order harmonics are more complex and no suitable filter can be found at present. The research of Sergey V. Kuzin validates the results of NSRL.

Acknowledgements Thanks to professor Shaojun Fu of NSRL, Zhanshan Wang and Yonggang Wu of Tongji University for their beneficial discussions and technical support. This work was supported by the National Natural Science Foundation of China (No. 10575097 and 60473133), the 100 Talents Programme of the Chinese Academy of Sciences; the National Basic Research Program of China (No. 2006CB303102) and Specialized Research Fund for the Doctoral Program of Higher Education (No. 20060358050).

References

1. Hunter W R, Long J P. Thin-film interference mirror to suppress grating harmonics in vacuum-ultraviolet radiation. Applied Optics, 1994, 33(7): 1264–1269
2. Kühne M, Müller P. Higher order contributions in the synchrotron radiation spectrum of a toroidal grating monochromator determined by the use of a transmission grating. Review of Scientific Instruments, 1989, 60(7): 2102–2104
3. Erko A, Langhoff N, Bjeoumikhov A A, et al. High-order harmonic suppression by a glass capillary array. Nuclear Instruments and Methods in Physics Research A, 2001, 467–468: 832–835
4. Matsushita T, Hashizume H. X-ray monochromators. Handbook on Synchrotron Radiation. Amsterdam: North-Holland, 1983
5. Suits A G, Heimann P, Yang X M, et al. A differential pumped harmonic filter on the chemical dynamics beamline at the advanced light source. Review of Scientific Instruments, 1995, 66(10): 4841–4844
6. Gullikson E M, Mrowka S, Kaufmann B B. Recent developments in EUV reflectometry at the advanced light source. SPIE, 2001, 4343: 363–373
7. Kjornrattanawanich B, Korde R, Boyer C N, et al. Temperature dependence of the EUV responsivity of silicon photodiode detectors. IEEE Transactions on Electron Devices, 2006, 53: 218–223
8. Korde R, Prince C, Cunningham D, et al. Present status of radiometric quality silicon photodiades. Metrologia, 2003, 40(1): S145–S149
9. Gullikson E M, Korde R, Canfield L R, et al. Stable silicon photodiodes for absolute intensity measurements in the VUV and soft x-ray regions. Journal of Electron Spectroscopy and Related Phenomena, 1996, 80: 313–316
10. Canfield L R, Vest R E, Korde R, et al. Absolute silicon Photodiodes for 169 nm to 254 nm photons. Metrologia, 1998, 35(4): 329–334
11. Xue S, Shao J H, Lu Q P, et al. Reflectivity measuring device in the national synchrotron radiation laboratory. Optics and Precision Engineering, 2004, 12(5): 480–484 (in Chinese)
12. Cao J H, Ni Q L, Chen B. Calibration of soft X-ray detector. Optics and Precision Engineering, 2004, 12(1): 118–121 (in Chinese)
13. Zhou H J, Wang Q P, Zheng J J, et al. Design and measurement of a portable ionization chamber. Optics and Precision Engineering, 2006, 14(3): 351–355 (in Chinese)
14. Feigl T, Yulin S, Benoit N, et al. Multilayer optics for the EUV and soft X-rays. Optics and Precision Engineering, 2005, 13(4): 421–429
15. Wang Z S, Wang F L, Zhang Z, et al. Research of multilayers in EUV, soft X-ray and X-ray. Optics and Precision Engineering, 2005, 13(4): 512–518