

# Design, prototyping, and evaluation of soft X-ray high diffraction efficiency and wide acceptance angle laminar-type W/C multilayer diffraction grating for 200–900 eV range

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For the soft X-ray grating spectrometers to use for the detection of trace elements, it is extremely important to efficiently disperses the incident light and secure the amount of the spectral flux reaching to the detector. For this purpose, we investigated a method to coat a soft X-ray multilayer optimized for a wide wavelength range centered at the Fe-*L* emission (705 eV, 1.76 nm) on a laminar-type diffraction grating. In this study, W/C multilayer coated laminar-type diffraction grating having Au surface was fabricated based on the results of a numerical optimization for the multilayer structure and incidence angle to maximize the amount of spectral flux reaching the detector. As a result of the measurement of the diffraction efficiency at the KEK PF BL-11D, the maximum diffraction efficiency was obtained at a wavelength of 1.76 nm and incidence angle of 86.2° whose conditions were well agreed with the those predicted by the numerical calculation. Also, the value of the measured maximum diffraction efficiency is 7.09% which corresponds to 66% of the estimation.

## 1 Introduction

There has been a great interest for the characterization and improvement of physical properties based on the spectroscopic analysis of soft X-ray emission from compound samples [1, 2]. Excitation of the sample with focused soft X-ray radiation and an electron beam is an effective method for local composition analysis [3]. Recently, state analysis of steel in the vicinity of Fe-*L* emission (705 eV) is a promising method to specify the effect of trace elements for strengthening and reducing the weight of steel sheets for automobiles. However, the effect of trace elements on the profile of the luminescent spectrum in state analysis is very small. Therefore, for the spectrometer which use a diffraction grating as the dispersion element, it is extremely important to efficiently disperse the incident soft X-ray at the grating and increase the amount of the spectral flux reaching to the detector.

In this study, therefore, we investigated a method to coat a soft X-ray multilayer on a diffraction grating to improve the diffraction efficiency and spectral flux in a wide wavelength range as well as Fe-*L* emission [4–6]. We assumed a W/C multilayer coated laminar-type diffraction grating having Au surface and optimized numerically the multilayer structure and incidence angle to maximize the amount of spectral flux reaching the detector. Furthermore, based on the results, a multilayer grating was fabricated, and the diffraction efficiency was evaluated.

The purpose of this report is to show the specification of the designed grating and experimental results which

demonstrate that the enhancement of diffraction efficiency and spectral flux can be obtained by use of a W/C multilayer and optimization of the incidence angle.

## 2 Laminar-type grating

Figure 1 shows a schematic diagram of the cross section of the laminar-type diffraction grating having a rectangular groove shape which was used for the addition of W/C multilayer. The material of grating blank is SiO<sub>2</sub> and a metal layer of Au layer having a thickness,  $d_1$ , of 30 nm is coated on the grating grooves. Geometrical parameters of

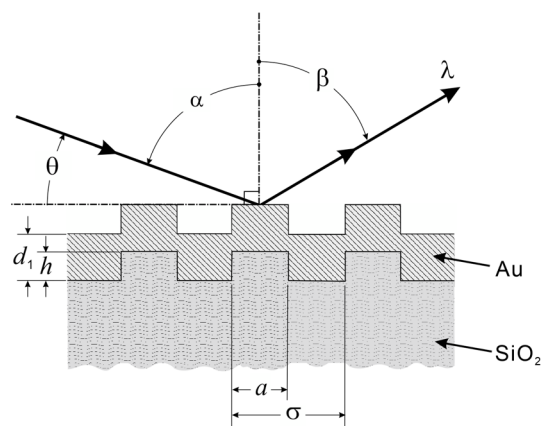


Fig.1: Schematic diagram of laminar-type diffraction grating with Au coating.

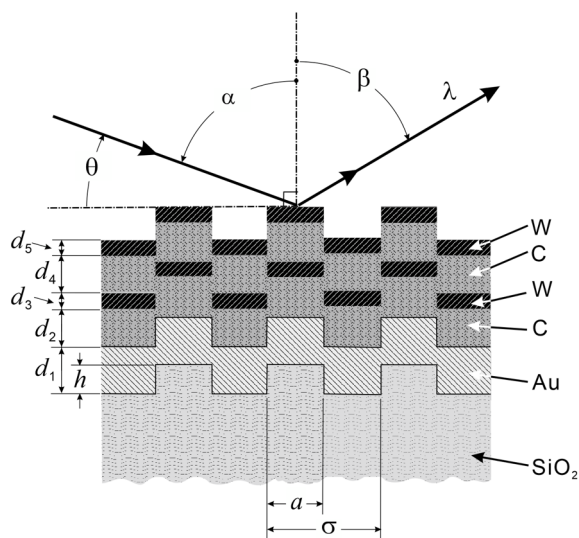


Fig.2: Schematic diagram of lamellar-type W/C multilayer diffraction grating.

the grating are as follows: grating constant,  $\sigma$ , of 1/2400 mm; duty ratio,  $D = a/\sigma$ , of 0.40; groove depth,  $h$ , of 6.2 nm; diffraction order,  $m$ , of +1. Also assumed was a metal layer of Au layer having a thickness,  $d_1$ , of 30 nm.

### 3 Results and Discussion

First, the diffraction efficiency of the lamellar-type grating coated with Au (refer to Fig. 1) was measured with BL-11D of the Photon Factory. Next, the multilayer coating consisting of two pairs of C and W layers was deposited in order by the ion beam sputtering method at IMRAM, Tohoku Univ. (refer to Fig. 2). The thickness of C,  $d_2$  and  $d_4$ , and W,  $d_3$  and  $d_5$ , are 7.4 nm and 6.0 nm, respectively. After then the diffraction efficiency was measured again. Fig. 3 shows the diffraction efficiencies of the incident angle dependence at an energy of 705 eV (a) and the energy dependence at an incident angle of  $86.2^\circ$

(b) obtained by numerical calculation and experiment. As the results of the numerical calculation, the diffraction efficiency up to 10.7% can be obtained at the incident angle of  $86.2^\circ$  and the diffraction angle of  $-83.5^\circ$  for 705 eV, but in the experiment, the measured value of 7.1%, which corresponds to 66% of that obtained by the calculation.

From these results, it can be safely confirmed the validity of the multilayer design scheme because that a clear improvement of the measured diffraction efficiency is obtained at the expected incident angle and energy by the numerical calculation. Since a meaningful discrepancy can be seen in diffraction efficiency between the calculation and experiment for the Au coated grating before the addition of the multilayer, it means that the surface roughness is negligible in the Au coated grating. On the other hand, for the multilayer grating, a significant difference in diffraction efficiencies obtained by the calculation and experiment, and it is considered to be attributed to the properties of the multilayer coating. To overcome this issue, we will proceed to analyze the properties of the coating materials and process of the multilayer coating more carefully to improve the diffraction efficiency much closer to that obtained by the numerical calculation in near future.

### Acknowledgements

The measurements of absolute diffraction efficiencies were performed as a part of the PF synchrotron radiation joint experiments of “Development of low incidence angle high diffraction efficiency diffraction grating in soft x-ray region,” (2018G012, April 2018–March 2020) and “Development of tailor-made keV region soft X-ray gratings using extreme techniques,” (2020G013, April 2020–March 2022). A.S.P acknowledges the support of Strategic Grant (Creative Research) by President, QST (No.20, April 2018–March 2020).

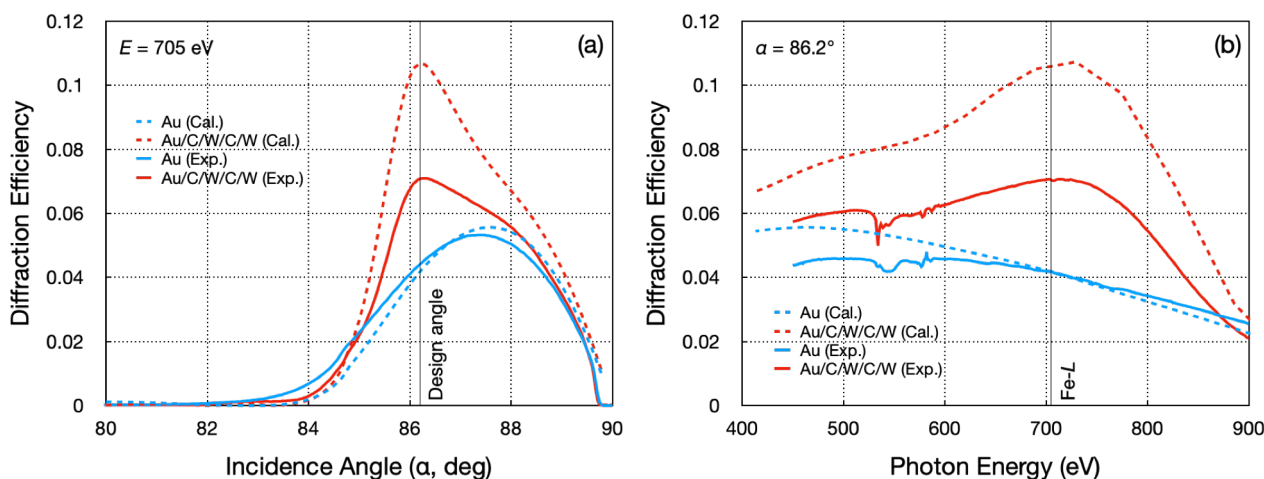


Fig. 3: Comparisons of calculated and measured diffraction efficiencies of the Au coated and multilayer gratings. Figures shown in (a) and (b) are the incidence angle dependence at an energy of 705 eV and photon energy dependence at an incidence angle of  $86.2^\circ$  of Au coated and multilayer gratings, respectively. The dashed and full curves show calculated and measured diffraction efficiencies, respectively.

References

- [1] Y. Leng, *Materials Characterization: Introduction to Microscopic and Spectroscopic Methods 2nd Edition* (Willy-VCH, Weinheim, 2013).
- [2] M. Terauchi, M. Koike, K. Fukushima, and A. Kimura, *J. Electron Microscop.*, **59**, 251–261 (2010).
- [3] R. Kasada, Y. Ha, T. Higuchi, and K. Sakamoto, *Sci. Rep.*, **6**, 25700 (2016), (6 pages).
- [4] E. G. Loewen and E. Popov, *Diffraction gratings and application* (Marcel Dekker, Inc., New York, 1997).
- [5] M. Koike et al., *Spectrochimica Acta Part B*, **64**, 756–760 (2009).
- [6] M. Koike et al., *Photon Factory Activity Report 2017*, #35 (2018) (3 pages).

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