

Diffraction Efficiency Measurements of Laminar-Type Diffraction Gratings Overcoated with Lanthanum Series Layers for Boron-K Emission Spectroscopy

Tadashi HATANO ^{1*}, Masato KOIKE ², Masami TERAUCHI ¹, Alexander S. PIROZHKOV ², Nobukazu HAYASHI ³, Hiroyuki SASAI ³, and Tetsuya NAGANO ³

¹ Institute of Multidisciplinary Research for Advanced Materials, Tohoku University
2-1-1 Katahira, Aoba-ku, Sendai, Miyagi 980-8577, Japan

² Kansai Photon Science Institute, Quantum Beam Sciences Directorate,
National Institutes for Quantum and Radiological Science and Technology
8-1-7 Umemidai, Kizugawa, Kyoto 619-0215, Japan

³ Device Department, Shimadzu Corporation

1 Nishinokyo-Kuwabaracho, Nakagyo-ku, Kyoto, Kyoto 604-8511, Japan

Experimental studies have been performed to evaluate the enhancement of diffraction efficiency of diffraction gratings by overcoating with lanthanum series layers on conventional metal coated laminar-type gratings around B-K emission (6.76 nm, 184 eV) spectrum. In the previous study we observed this effect with a laminar-type Ni-coated diffraction grating overcoated with diamond-like carbon (DLC), TiO₂, and CeO₂ layers which have high reflectivity and small extinction coefficient. In this study, LaF₃ and La/C layers were overcoated on Ni-coated gratings, and diffraction efficiency was measured by use of the reflectometer at the PF-BL11D beamline. As the results, the enhancements of spectral flux at 6.76 nm are 3.62 and 4.41 times at the incident angles of 85.005° and 85.050° for LaF₃ and La/C overcoated gratings, respectively, compared with that of a conventional Ni coated grating used at an incidence angle of 87.070°.

1 Introduction

There has been common practice to use precious metals, Au and Pt typically, as the coating materials of the soft X-ray diffraction gratings for general use. Furthermore, the gratings are usually illuminated at a grazing incidence condition beyond the critical angle to attain high diffraction efficiency taking an advantage of total reflection condition [1]. However, usually the larger incidence angle invites the higher diffraction efficiency and less incoming flux. For the sake of tradeoff relation of between diffraction efficiency and incoming flux it is essentially difficult to increase the spectral flux which is the product of incoming flux and diffraction efficiency, and the parameter directly affecting to sensitivity of the applied grating spectrometer. Also, multilayer coating is an effective method to enhance the diffraction efficiency at an interested spectral range even sacrificing the spectral bandwidth [2]. Therefore, we have initiated feasibility studies to explore the methods to enhance spectral flux essentially not merely increase diffraction efficiency at a traditionally predefined incidence angle as before. In the previous report we showed simulation and experimental results which demonstrate that enhancement of spectral flux around B-K emission spectrum (6.76 nm, 186 eV) by overcoating popular materials of TiO₂ and CeO₂ layers on conventional Ni coated laminar-type gratings. As the results, the enhancements of spectral flux at 6.76 nm were 2.2 and 3.4 times for the cases of TiO₂ and CeO₂ overcoatings, respectively [3-5].

The purpose of this report is to show simulation and experimental results which demonstrate that similar

enhancement of diffraction efficiency can be obtained by use of lanthanum series layers which is known as a promising material for multilayer mirrors with enhanced spectral selectivity for the next generation extreme ultraviolet lithography in the 6 nm range [6,7].

2 Experiment

Table 1 shows the list of common, past and newly investigated coating materials, and their theoretical density, ρ , complex refractive index defined by $n = (1 - \delta) + i\beta$ (β : extinction coefficient), and θ/θ_c , *i.e.*, a grazing incidence angle (θ) of 5.5°, divided by critical angle measured from the surface, θ_c , at $\lambda = 6.76$ nm.

Table 1: Density, optical constant, and grazing incidence angle normalized with critical angle. Here ρ is density, δ and β are components of complex refractive index, and θ_c is critical angle measured from the reflecting surface at $\lambda = 6.76$ nm. $\theta = 5.5^\circ$ is grazing angle of incidence.

	ρ (g/cm ³)	δ	β	θ/θ_c
Ni	8.9	0.025200	0.018933	0.428
Au	19.32	0.014819	0.008417	0.558
Pt	21.45	0.014598	0.014647	0.562
C	2.2	0.008900	0.000757	0.720
DLC	3.1	0.012550	0.001066	0.606
TiO ₂	4.26	0.015919	0.004000	0.537
CeO ₂	7.3	0.022130	0.003725	0.456
La	5.95	0.016448	0.001420	0.529
LaF ₃	5.9	0.019676	0.003093	0.484
La ₂ O ₃	6.51	0.019656	0.002218	0.484

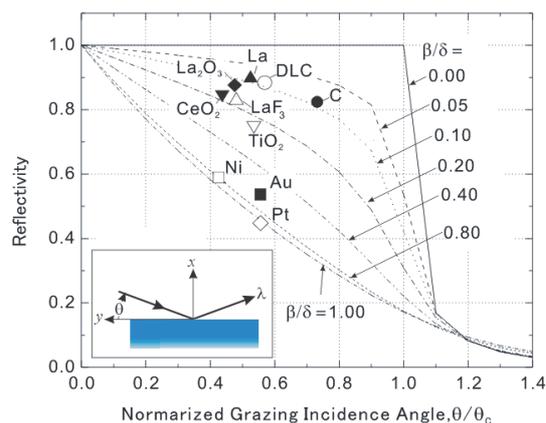


Fig. 1: Calculated reflectivity of Ni, \square , Au, \blacksquare , Pt \diamond , C, \bullet , DLC, \circ , TiO₂, ∇ , CeO₂, \blacktriangledown , La, \blacktriangle , La₂O₃, \blacklozenge , LaF₃, \triangle , at $\lambda = 6.76$ nm and glancing angle of incidence $\theta = 5.5^\circ$. The abscissa indicates the value of θ normalized to θ_c . Reflectance curves for $\beta/\delta = 0.0, 0.05, 0.1, 0.2, 0.4, 0.8,$ and 1.0 are also shown.

Figure 1 shows the graph of the calculated reflectivity of Ni, \square , Au, \blacksquare , Pt \diamond , C, \bullet , DLC, \circ , TiO₂, ∇ , CeO₂, \blacktriangledown , La, \blacktriangle , La₂O₃, \blacklozenge , LaF₃, \triangle , at $\lambda = 6.76$ nm and glancing angle of incidence $\theta = 5.5^\circ$ plotted against respective values of θ/θ_c . The reflectivity curves of $\beta/\delta = 0, 0.05, 0.10, 0.20, 0.40, 0.80,$ and 1.00 are also indicated. This figure indicates that lanthanum series materials, i.e., La, La₂O₃, and LaF₃ would exhibit high diffraction efficiency competitive with the materials investigated previously [3].

Lanthanum exhibits the highest reflectivity, but in air the deposited surface is easily oxidized to smash. Therefore, a protective cap layer of a stable material such as carbon is needed. La₂O₃ is one of the materials widely used for optical coating having a high melting temperature (2,315 °C). Therefore, the deposition phenomenon occurs for the substrate heated up to a high temperature range over 300°C, and it is difficult to apply to the devices which are undurable to heat load like replica gratings. LaF₃ is also the most commonly used material for UV optical coatings, but defers from La₂O₃, and can be deposited by various methods including thermal evaporation method for the sake of its moderate melting temperature (1,493 °C).

Finally, we chose three types of coatings for the experiment, i. e., Ni single layer for a reference (Type A), Ni/LaF₃ coating (Type B), and Ni/La/C coating (Type C). Figure 2 shows the schematic diagrams of the cross section of the three types of gratings. Table 2 summarizes the parameters of the fabricated gratings. In this table, the subscript i of the parameter means a, b, or c.

Fabrications of the laminar-type gratings and Ni coating were performed at Shimadzu Corp., Kyoto, Japan, and residual coatings were conducted at Institute of Multidisciplinary Research for Advanced Materials, Tohoku University. The measurements of absolute diffraction efficiencies of the gratings were carried out at the BL11D beamline at the Photon Factory, KEK, Tsukuba, Japan.

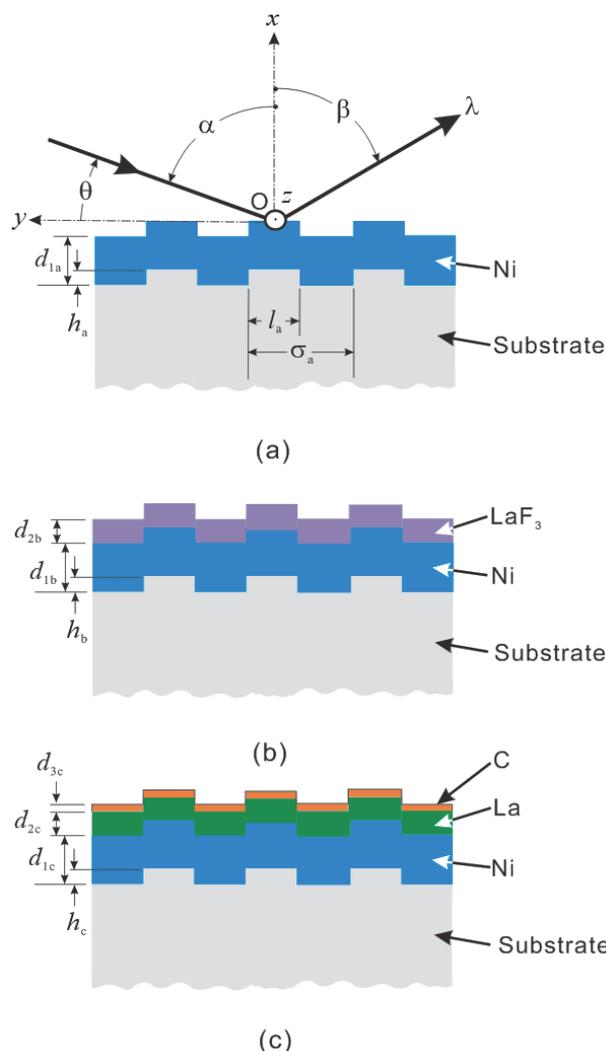


Fig. 2: Schematic diagram of laminar-type gratings coated with single, double, and triple layers, denoted as Types, A, B, and C, respectively.

Table 2: Parameter list of the fabricated gratings. The subscript i of parameters means a, b, or c in Fig. 2.

	Type A	Type B	Type C
coating	Ni	Ni/LaF ₃	Ni/La/C
$1/\sigma_i$ (lines/mm)	1200.00	1200.00	1200.00
l_i (nm)	341.67	316.67	357.14
l_i/σ_i	0.41	0.38	0.40
h_i (nm)	14.00	16.50	15.00
d_{1i} (nm)	30.00	30.00	60.00
d_{2i} (nm)	—	31.20	25.40
d_{3i} (nm)	—	—	4.00

3 Results and Discussion

Figure 3 shows the relation of the incidence angle with calculated and measured diffraction efficiencies of the first order light ($m = +1$) at $\lambda = 6.76$ nm for the gratings coated with Ni (30.0), Type A, Ni/LaF₃, Type B, and Ni/La/C, Type C. The dashed and full curves show calculated and

measured ones. The diffraction efficiencies of the grating of Types A, B, and C were 0.139, 0.223, and 0.254 at the default incidence angle of 87.070° , respectively. The maximum diffraction efficiencies were 0.173, 0.296, and 0.363 at the incidence angles of 84.420° , 85.005° , and 85.050° for the gratings of Types A, B, and C, respectively.

For the case of the width of the diffraction grating in view of the incident light direction is proportional to the amount of incident flux ($\propto \cos\alpha$), the gains of the incidence flux are 1.90, 1.70, and 1.69 times, and finally the enhancements of diffracted spectral flux are 2.33, 3.62 and 4.41 times compared to those of the Ni coated grating used at the incidence angle of 87.070° , respectively.

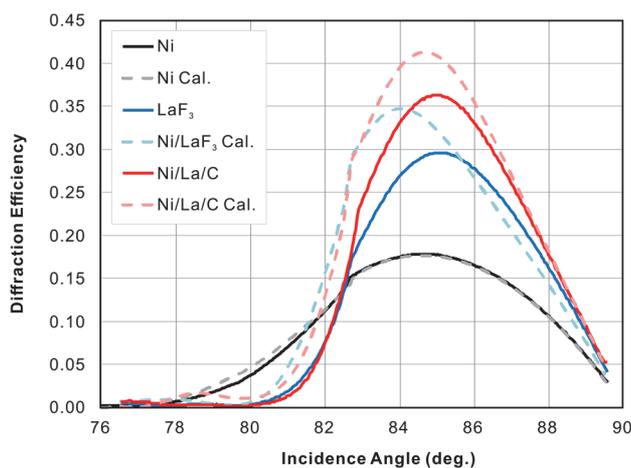


Fig. 3: Calculated and measured diffraction efficiencies for the gratings of Types A, B, and C, coated with Ni, Ni/LaO₃, and Ni/La/C layers, respectively, vs. incidence angle at $\lambda = 6.76$ nm. For details see text.

Figure 4 shows wavelength dependence of the diffraction efficiencies of the gratings of Types A, B, and C for the incidence angles of 87.070° , 84.200° and 84.200° , respectively. The dashed and full curves indicate calculated and measured ones. The deviations of the real peak positions and values of diffraction efficiencies appeared for Types B and C gratings from the calculated results are considered to be due to the discrepancies of real optical constants and thickness of the coating layers from those assumed values for the calculations in addition to the effect of surface roughness, and which imply the possibilities of further improvements. Regarding to oxidization of lanthanum it is necessary to observe whether the protective layer of carbon works effectively as well as well as the aging over time. We will discuss this subject elsewhere.

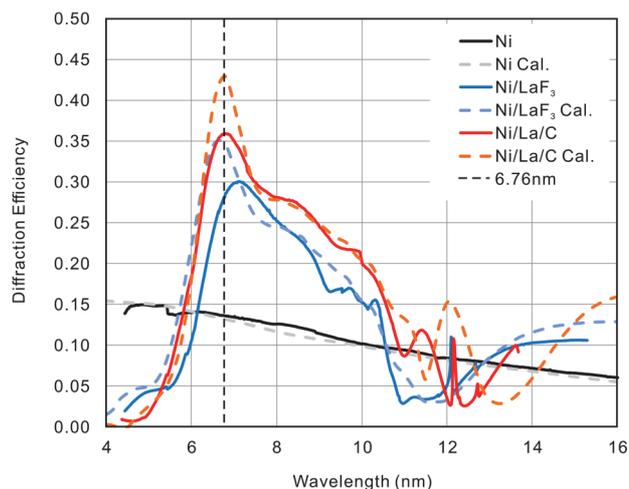


Fig. 4: Calculated and measured diffraction efficiencies for the gratings of Types A, B, and C, respectively, vs. wavelength at incidence angles of 87.070° for the grating of Type A and 84.200° for those of Types B and C. For details see text.

Acknowledgements

The measurements of absolute diffraction efficiencies were performed as a part of the PF synchrotron radiation joint experiment of “Development of low incidence angle high diffraction efficiency diffraction grating in soft x-ray region,” (2018G012, April 2018-March 2020). A.S.P acknowledges the support of Strategic Grant (Creative Research) by President, QST (No.20, April 2018-March 2020).

References

- [1] E. G. Loewen and E. Popov, *Diffraction gratings and application* (Marcel Dekker, Inc., New York, 1997).
- [2] M. Koike et al., *Spectrochimica Acta Part B*, **64**, 756-760 (2009).
- [3] M. Koike et al., *AIP Conf. Proc.* **1741**, 040045 (2016), (4pages); doi: 10.1063/1.4952917.
- [4] T. Imazono et al., *AIP Conf. Proc.* **1741**, 040043 (2016), (4 pages); doi: 10.1063/1.4952915.
- [5] M. Koike et al., *Photon Factory Activity Report 2017*, #35 (2018) (3 pages).
- [6] Y. Platonov et al., *Pros. SPIE* **8076** (2011), doi: 10.1117/12.889519.
- [7] N. Chkhalo et al., *Appl. Phys. Lett.*, **102** (2013), doi: 10.1063/1.4774298.

*hatanotadashi@tohoku.ac.jp