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High-Diffraction-Efficiency Wide-Acceptance-Angle Laminar-Type Diffraction Gratings Overcoated with Oxide Films for Boron-K Emission Spectroscopic Measurements

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Theoretical and experimental studies have been performed to explore the enhancement of diffraction efficiency of diffraction gratings around B-K emission (6.76 nm, 184 eV) spectrum based on overcoating with materials having high reflectivity and small extinction coefficient (T layer) on conventional metal layer (M layer) coated gratings. In the previous study we confirmed this effect with a laminar-type Ni-coated diffraction grating overcoated with diamond-like carbon (DLC). In this report the authors examine this effect using TiO_2 or CeO_2 layer as the T layer and Ni as the M layer by numerical calculations and measurements by use of the reflectometer at the PF-BL11D beamline. As the results, the enhancements of diffraction efficiency at 6.76 nm are 2.2 and 3.4 times with the T layers having optimum thickness of 22.0 and 30.2 nm at the incident angles of 85.262° and 84.492° for TiO_2 and CeO_2 layers, respectively, compared with a conventional Ni coated grating used at an incidence angle of 87.070°.

1 Introduction

There has been a great demand from both academic and industrial communities in highly sensitive spectroscopic measurement in the soft X-ray region containing emission and absorption associated with the inner shell transitions of light elements. Especially boron is the critical trace element due to the advantage of improving quenching characteristics in steel. To detect the B-K emission band at around 6.76 nm more efficiently we performed a feasibility study to enhance the diffraction efficiency of laminar-type diffraction grating based on a newly discovered physical phenomenon in the region of total reflection. The phenomenon is embodied by overcoating a transparent high-density material on a metal layer on grating grooves. We have found that the overcoating material should have middle refractive index between those of vacuum and the metal layer, and small extinction coefficient, i. e. high transparency. We have already found that tetrahedral amorphous carbon, namely diamond-like carbon (DLC), in which sp^3 hybrid orbital (diamond structure) is dominant, is a suitable material for this purpose by numerical calculations [1]. The DLC layer can be produced by use of a filtered arc method [2]. The enhancement of diffraction efficiency by means of this technique was confirmed by the measurements at a synchrotron radiation facility [3].

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 well-known TiO_2 and CeO_2 layers. Our efforts are also extended to the optimizations of the thickness of the oxide layer as well as the incidence angle which increases spectral flux due to widening of the acceptance angle of the grating.

2 Experiment

Figure 1 shows a schematic diagram of the cross section of the new laminar-type diffraction grating having a rectangular groove shape. Geometrical parameters of the base Ni-coated grating used throughout this study are as follows: grating constant, σ, of 1 /1200 mm; duty ratio, D = a/σ, of 0.41; groove depth, h, of 14.0 nm; diffraction order, m, of +1. Also assumed was a metal layer of Ni layer (M layer) having a thickness, d_1, of 30 nm. The

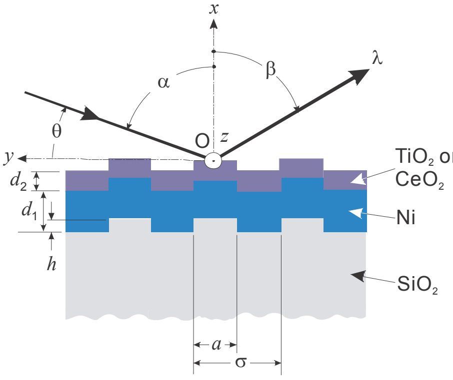

Fig. 1: Schematic diagram of a new laminar-type grating coated with Ni and TiO_2 or CeO_2.

initially designed incident angle, α, of the Ni-coated grating is 87.070°.

It is a common practice to coat the grating with a highly reflective material to enhance the diffraction efficiency of a grating for any spectral region. Table 1 shows the list of common and newly investigated materials, and their theoretical density, ρ, complex refractive index parameters defined by $n = (1-\delta) + i\beta$ (β: extinction coefficient), and θ/θ_C, *i.e.*, glancing angle of incidence, θ (= 90° – α), divided by critical angle measured from the surface, θ_C [4]. Here, a-C and ta-C stand for amorphous graphite and DLC.

Figure 2 shows the reflectance curves for δ/β=0, 0.05, 0.10, 0.20, 0.40, 0.80, and 1.00. The abscissa indicates the value of θ normalized to θ_C. In the figure the calculated reflectance of Ni, □, Au, ■, Si, ◇, SiC, △, SiO_2, ▲, a-C, ●, ta-C, ○, TiO_2, ☆, and CeO_2, ◎, at λ = 6.76 nm and θ = 5.5° are plotted against respective values of θ/θ_C. Carbon based a-C and ta-C show high reflectance as seen in this figure. From this figure it is found that TiO_2 and CeO_2 would exhibit high diffraction efficiency as well as ta-C and a-C comparing with conventional metals, Ni and Au, and silicides, if the grating grooves could be ruled in TiO_2 and CeO_2 substrates. However, it is difficult to realize with both mechanically ruled and holographic gratings from various practical reasons. Therefore it is the most realistic solution to coat the oxide layer having high reflectance on the conventional metal coated grating.

Assuming Ni as the M layer we have searched the optimum layer thickness of the oxide layer (T layer) and incident angle simultaneously which attains maximum diffraction efficiency at the B-K emission by use of a simulation code based on rigorous coupling method [5]. Finally we found that the optimum thicknesses were 25.8 and 31.5 nm at the incident angles of 84.360° and 82.840° for TiO_2 and CeO_2 layers, respectively.

Fabrications of laminar-type grooves and coatings of both M and T layers were performed at Shimadzu Corp., Kyoto, Japan. The measurements of absolute diffraction efficiencies of the gratings were carried out at the BL-11D beamline at the Photon Factory, KEK, Tsukuba, Japan.

Table 1: Density and optical constants of common and investigated coating materials. Here σ is density, δ and β are defined by complex refractive index, $n=(1-\delta) + i\beta$, θ_C is critical angle measured from the reflecting surface at λ = 6.76 nm, and θ = 5.5° is glancing 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
Si	2.33	0.008062	0.009647	0.756
SiC	3.22	0.011704	0.009662	0.627
SiO_2	2.2	0.009525	0.005422	0.695
a-C	2.2	0.008900	0.000757	0.720
ta-C	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

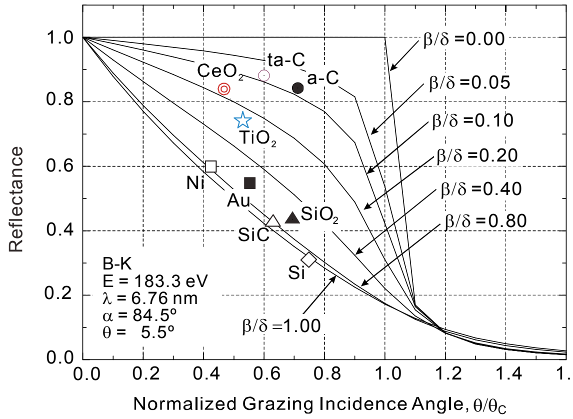

Fig. 2: Calculated reflectance of Ni, □, Au, ■, Si, ◇, SiC, △, SiO_2, ▲, a-C, ●, ta-C, ○, TiO_2, ☆, and CeO_2, ◎, at λ= 6.76 nm and glancing angle of incidence θ = 5.5°. Reflectance curves for δ/β = 0.0, 0.05, 0.10, 0.20, 0.40, 0.80, and 1.00 are also shown.

3 Results and Discussion,

Figure 3 shows the relation of the incidence angle with diffraction efficiency of the first order light (m = + 1) at λ = 6.76 nm for the gratings coated with Ni(30.0), Ni(30.0)/TiO_2(22.0), and Ni(30.0)/CeO_2(30.2). The numbers in the parentheses show the thickness of the layer in nm. The diffraction efficiencies of the grating coated with Ni, Ni/TiO_2, and Ni/CeO_2 layers were 0.139, 0.173, and 0.191 at the incidence angle of 87.070°, respectively. The corresponding maximum diffraction efficiencies were 0.173, 0.206, and 0.282 at the incidence angles of 84.420°, 85.262°, and 84.492° for the gratings coated with Ni, Ni/TiO_2, and Ni/CeO_2 layers, 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 (∝ cos α), the gains of the

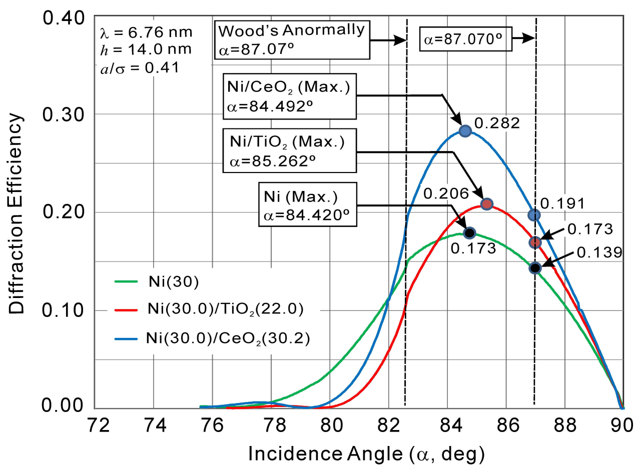

Fig. 3: The measured diffraction efficiencies for the gratings coated with Ni, Ni/TiO_2, and Ni/CeO_2 layers vs. incidence angle at λ = 6.76 nm.

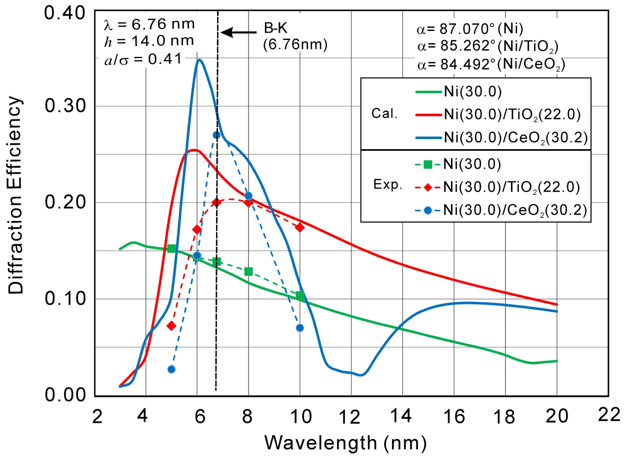

Fig. 4: Diffraction efficiency vs. wavelength for Ni, Ni/TiO_2 and Ni/CeO_2 coated gratings obtained by numerical calculations and experiments. For details see text.

incidence flux are 1.51, 1.61, and 1.88 times, and finally the enhancements of diffracted spectral flux are 1.87, 2.39 and 3.81 times compared to those of the Ni coated grating used at the incidence angle of 87.070°, respectively.

Figure 4 shows wavelength dependence of the diffraction efficiencies of the gratings coated with the Ni, Ni/TiO_2, and Ni/CeO_2 for the incidence angles of 87.070°, 85.262° and 84.492°, respectively. The grating overcoated with CeO_2 shows high diffraction efficiency in the vicinity of B-K. However, it is found that the diffraction efficiency significantly decreases toward 11 nm, which is considered to be attributed to the absorption of $4d^{10}4f \rightarrow 4d^9 4f^2$ transition [6]. The deviations between the calculated and experimental peak positions and values of diffraction efficiencies appeared for Ni/TiO_2 and Ni/CeO_2 coated gratings are considered to be due to the optical constants and thickness of the coatings assumed for the calculations as well as the surface roughness, and required to perform further investigations.

It is well known that TiO_2 is a unique material having photocatalyst effect [7], therefore, it could be expected that TiO_2 layer circumvents the phenomenon of seizure attributed to residual contaminants on the surface of the optical elements including diffraction gratings in the ultra-high vacuum apparatus such as a synchrotron radiation beamline.

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