Development of a flat-field spectrograph with a wide-band multilayer grating and prefocusing mirror covering 2-4 keV

T Imazono1, M Koike1, N Hasegawa1, M Koeda2, T Nagano3, H Sasai3, Y Oue2, Z Yonezawa1, S Kuramoto2, M Terauchi3, H Takahashi4, N Handa4 and T Murano4

1 Quantum Beam Science Directorate, Japan Atomic Energy Agency, 8-1-7 Umemidai, Kizugawa, Kyoto 619-0215 Japan
2 Device Department, Shimadzu Corp., 1 Nishinokyo-Kuwabara-cho, Nakagyo-ku, Kyoto 604-8511 Japan
3 Institute of Multidisciplinary Research for Advanced Materials, Tohoku University, 2-1-1 Katahira, Aoba-ku, Sendai 980-8577 Japan
4 EO Peripheral Components Business Unit, JEOL Ltd., 3-1-2 Musashino, Akishima, Tokyo 196-8558

E-mail: imazono.takashi@jaea.go.jp

Abstract. A flat-field spectrograph equipped with a wide-band multilayer grating and prefocusing mirror covering 2–4 keV without any mechanical movement has been developed. To realize this, a new multilayer structure consisting of W and B4C layers has been invented, which enhances the diffraction efficiency of the grating over the whole energy range at a fixed angle of incidence as well as the reflectivity of the prefocusing mirror. The multilayer has been deposited on a laminar-type varied-line-spacing holographic replica grating and a spherical mirror substrate. The diffraction efficiency of the multilayer grating varies between 1.2% and 3.3% at 88.65° in the 2.1–4.0 keV range. Also the reflectivity of the prefocusing mirror varies between 2.7% and 12% at 88.00° in the same range. The overall throughput of the spectrograph with the multilayer optics is 104 times higher than that of gold-coated optics.

1. Introduction

Transmission electron microscopy (TEM) is useful for structural and elemental analysis on the nanometer scale. It is very attractive to introduce soft x-ray emission spectroscopy (SXES) into TEM to study chemical properties. This is because the soft x-rays emitted by the decay of valence electrons give information about the partial density of states of the valence band which strongly reflects the electronic structure of the material. The technique is called TEM-SXES. A soft x-ray flat-field spectrograph equipped with a mechanically-ruled blazed varied-line-spacing (VLS) diffraction grating was attached to a conventional transmission electron microscope to measure the B-K emission spectrum of boron nitride in a nanometer scale area for the first time [1]. This spectrograph covered a range of 60–250 eV with high resolving power. Furthermore, it was successfully improved to cover the 60–2000 eV range using a laminar holographic VLS grating [2].

We initiated a project to extend TEM-SXES to a wider range between 50 eV and 4000 eV. The energy range was divided into four ranges as follows: 50–200 eV; 155–350 eV; 300–2200 eV; and 2000–4000 eV, and the gratings for the respective ranges were designed so as to be accommodated.
with a single flat-field spectrograph by interchanging the gratings. Laminar and blazed VLS holographic gratings for the 50–200 eV range have been successfully developed [3, 4]. For a soft x-ray grating a gold film is typically deposited on the surface, but the reflectivity of the coating is impractical in the range above the Au-M edges of ~2.2 keV. Thus, a multilayer coating in place of the Au single-layer should be applied to enhance the diffraction efficiency in the 2–4 keV range. A conventional multilayer grating, i.e., a grating coated having a constant period multilayer, has high diffraction efficiency, but a narrow energy bandwidth at a fixed angle of incidence. This indicates that the SXES instrument needs mechanical movement to scan in energy in order to cover the required energy range. To overcome this difficulty, a novel multilayer structure was invented to uniformly enhance the reflectivity in the few keV range at a constant angle of incidence and was also applied to the grating in the 2–4 keV range. We call this a wide-band multilayer grating. The multilayer consists of W and B$_4$C layers with the topmost W layer, and only the last B$_4$C layer just below the topmost layer is two times thicker than the other B$_4$C layers [5]. It is thought to be similar to a supermirror reflecting x-rays having short and long wavelengths, respectively, at the bottom and top regions in the multilayer. In our previous study, the multilayer was deposited on a laminar VLS holographic master grating. A wide-band multilayer grating covering the 2–4 keV was successfully developed [5].

If the new multilayer structure is deposited on a concave substrate, it acts as a wide-band focusing mirror. When the focusing multilayer mirror as well as the multilayer grating are installed in a spectrograph, the throughput would significantly increase compared to that employing a gold-coated mirror and grating. This means that a spectrograph equipped with full wide-band multilayer optics is useful for not only TEM-SXES, but also the observation of multi-keV x-rays produced by a short-pulse laser, etc.

First, in this paper, we briefly describe the flat-field spectrograph interchangeable with four VLS gratings and the result of ray-tracing in a spectrograph equipped with a spherical prefocusing mirror in the 2–4 keV range. Then a wide-band multilayer grating and mirror deposited, respectively, on a laminar VLS holographic replica grating and spherical glass substrate are described. Finally, we show the efficiencies of the multilayer grating and mirror measured in the 2.1–4.0 keV range using synchrotron radiation.

2. Design of a flat-field spectrograph with a VLS holographic grating and prefocusing mirror

From the viewpoint of practical use of the four gratings to cover the 50–4000 eV range, the spectrograph was designed to be compatible with all the gratings. The design concept was to fix the positions of the light source and image plane, and the direction of the grating normal in the spectrograph equipped with each grating [3].

Figure 1 shows a schematic of the flat-field spectrograph equipped with the wide-band multilayer grating and a prefocusing mirror for the 2–4 keV range. The light emerging from the source point $S_0$ is incident on a spherical mirror having a 5000-mm radius of curvature and a size of 40 mm (W) × 30 mm (H) at the angle of 88.00°. The meridional rays are focused on an entrance slit $S$. A thin filter such as aluminium between the entrance slit and grating is used to reduce unwanted light. The grating centre is located at the origin O and its normal is on the x-axis. The distance PO is 236.69 mm, where P is the projection of the position of S onto the y-axis. The light passing through the entrance slit is incident on the grating at 88.65°. The design parameters of the grating are as follows: radius of curvature 11,200 mm; effective grating constant 1/2400 mm; groove depth 2.8 nm; duty ratio (land/grating constant) 0.5; effective ruled area 46 mm (W) × 26 mm (H) [5]. Also the VLS groove parameter is described in ref. 5. The diffracted light in the first-order is focused on an image plane $\Sigma$.
parallel to the x-axis and intersects at Q perpendicular to the y-axis. The image length on $\Sigma$ is 3.33 mm for a range of 2–4 keV (0.31–0.62 nm). The distance OQ is 233.50 mm. Figure 2(a) shows the imaging property of the spectrograph evaluated by our own code for ray tracing. A self-luminous source having dimensions of 50 $\mu$m $\times$ 50 $\mu$m, a slit width and height of 30 $\mu$m $\times$ 10 mm, and a vertical aperture of 8-mm height in front of $\Sigma$ were assumed. The spot diagrams were constructed with 10,000 rays for $\lambda$ and $\lambda \pm \Delta\lambda/300$ ($\lambda = 0.31$, 0.48, and 0.62 nm). The line profiles were constructed by counting the number of spots falling into the each pixel having a width of 0.7 $\mu$m on $\Sigma$. The resolving power $\lambda/\Delta\lambda$ calculated from the spot diagrams is given in figure 2(b). It is sufficiently higher than 300 in the wavelength range of 0.31–0.62 nm, i.e., 2–4 keV. When a CCD pixel size is 20 $\mu$m, it corresponds to $\lambda/\Delta\lambda = 200–300$. Also when the entrance slit width of 30 $\mu$m is assumed, $\lambda/\Delta\lambda = 130–200$. This means that they affect the practical spectral resolution rather than the aberrations of the spectrograph.

A laminar-type master grating was fabricated holographically by an aspheric wavefront recording optics and reactive ion beam etching method [5–7]. A pair of gold-coated replica gratings (AGs) was replicated from the master by Shimadzu Corp. One of the two gratings was used to fabricate the wide-band multilayer grating (MLG) and the other was not modified to compare the diffraction efficiencies. The groove depth, duty ratio, and surface roughness of the AG before depositing the multilayer were evaluated to be ~2.5 nm, 0.50, and 0.34 nm root-mean-square (RMS), respectively, by using an atomic force microscope, and diamond stylus type and optical surface profilometers.

The MLG was fabricated by an ion beam sputtering method as follows. First a W/B$_4$C multilayer with the topmost B$_4$C layer was deposited on the AG, of which the period length, thickness ratio of the W layer to the period, and number of layers are 5.6 nm, 0.50, and 38, respectively. Then an inverted bilayer, B$_4$C/W, with a 5.6 nm period length and 0.50 thickness ratio was deposited on the W/B$_4$C multilayer. The total number of layers is 39, but not 40 because the last B$_4$C layer just below the topmost W layer is a continuous layer. Therefore, the thickness is two times larger than the other B$_4$C layers [5]. The wide-band multilayer differs from an ordinal multilayer in that a single W layer is deposited on the W/B$_4$C multilayer with the topmost B$_4$C layer. This scheme is sort of similar to a supermirror for hard x-ray optics. The top region with a 1.5 times larger period length than the bottom one optimized at 3.5 keV enhances the reflectivity around 2.3 keV, two thirds of the energy of 3.5 keV. In spite of this, the light around 3.5 keV is transparent in the top region and reflected by the bottom. Consequently, the integral reflectivity increases 20%. The wide-band multilayer deposited on the grating was evaluated to have a period length of 5.594 nm and a thickness ratio of 0.50 with an x-ray diffractometer. The same multilayer was also deposited on a spherical glass substrate having a 0.2-nm RMS roughness. This is called MLM.

3. Performance of the multilayer grating and spherical mirror

The absolute diffraction efficiencies of MLG and AG, and the reflectivities of MLM and the gold-coated mirror (AM) were measured using a goniometric apparatus [5] at a soft x-ray double-crystal monochromator beamline (BL-11B) of the Photon Factory, KEK, Tsukuba, Japan [8]. Figure 3(a) shows the experimental diffraction efficiencies of MLG and AG at a constant angle of incidence of 88.65° in the 2.1–4.0 keV range. The maximum and minimum efficiencies of MLG are 3.3% and 1.2%, respectively. Thus, MLG shows a high and uniform efficiency over the measured energy range. Moreover, shown in figure 3(b) is the measured reflectivities of MLM and AM at a
constant incidence angle of 88.00°. The reflectivity of MLM varies from 11.9% to 2.7% in the measured energy range. It is almost constant compared to that of the AM. In the figures the solid lines are the calculated diffraction efficiencies and reflectivities performed with a commercial code (GSOLVER V4.2c, Grating Solver Development Co.) and our own code, respectively. Unfortunately, the surface and interfacial roughness of the grating and multilayer, and the deviation from the nominal groove depth are considered to result in inconsistencies with the calculations such as in the reduction of efficiency. Shown in figure 3(c) is the throughput of the spectrograph with multilayer optics (MO) and gold-coated optics (AO) estimated as the product of the efficiencies of the optics ignoring geometrical vignetting due to aberrations. The throughput of the MO achieves from 1 at 2.1 keV to $10^4$ times at 4.0 keV higher than that of the AO.

4. Conclusion

The new multilayer scheme we show is obviously effective to uniformly enhance the diffraction efficiency of the grating as well as the reflectivity of the mirror at a constant incidence angle in the 2–4 keV range. Therefore, it is concluded that the flat-field spectrograph equipped with the grating and prefocusing mirror deposited with wide-band multilayers is useful for multi-keV x-ray spectroscopy as well as TEM-SXES.

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