Monochromator operation in the carbon window region at the reflectometry beamline BL-11D of the Photon Factory

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Abstract. The vacuum ultraviolet (VUV) and soft X-ray beamline BL-11D of the Photon Factory has been recently equipped with a reflectometer to be dedicated to reflectometry for optical elements. The monochromator is of a varied deviation angle type. Focus conditions had been achieved using photoelectron spectroscopy data in the separated regions of 60–200 eV and 300–700 eV with respective gratings. Relying on the monochromator parameters, we programmed a monochromator operation optimized to 250–300 eV region for characterization of carbon window multilayers. Spectral reflectance of a Cr/C multilayer was measured at various angles of incidence, and the peak energy changed, showing that the monochromator has a good spectral accuracy as well as a high resolving power. We also programmed a monochromator operation above 700 eV, and the operable energy range has been extended to 200–1000 eV with one grating. The spectral transmittance and reflectances of Co/C multilayers were measured, and the spectral impurities were discussed.

1. Introduction
The vacuum ultraviolet (VUV) and soft X-ray beamline BL-11D of the Photon Factory (PF) was reconstructed for photoelectron spectroscopy studies in 1996 [1], and the beamline was fitted with a variable deviation angle monochromator. During the photoelectron spectroscopy activities, the monochromator operation system has not been elaborated enough for energy scanning measurements. In 2010, BL-11D was redesigned for reflectometry with the installation of a reflectometer. The monochromator operation system needs to be improved to satisfy the requirements for characterizing mirror samples through spectral reflectance measurements. We have been developing multilayer elements for use in the VUV to hard X-ray regions [2], and one of our recent targets is the carbon window region lying below the C K-absorption edge where organic samples are relatively transparent [3]. Multilayer characterization tools are crucial to the development of high throughput carbon window microscopes based on the multilayers deposited on the mirror substrates. Thus the monochromator must be tuned up especially to the carbon window region for this purpose.

Design and actual operating system of the monochromator are described in the next section. Our approach to making the best use of the monochromator in the carbon window region is given along with verification using a Cr/C multilayer as a test sample. A similar approach was also applied to the higher energy region. Measured spectral reflectances of Co/C multilayers are also presented.

2. Design and actual operation of the monochromator
A schematic side view of the monochromator and post-focusing mirrors is illustrated in figure 1, in which the distance from the source to each element is shown. A cylindrical mirror Mf, set at a grazing
angle of $2^\circ$, will form an image of the entrance slit S1 near S2 if mirror Mp is not in place. Mp is a plane mirror that rotates about point C, which is behind the grating G. The grazing angle of Mp is denoted by $\theta_M$, which is identical to the rotation angle. The convergent beam is reflected by Mp toward the spherical grating G, which configures the negative-incidence-length, varied-deviation-angle-type monochromator. The deviation angle is approximately $\pi - 2\theta_M$. The diffraction angle measured from the grating surface is denoted by $\theta_G$, which is identical to the rotation angle of G, and the generally defined diffraction angle is equal to $\theta_G - \pi/2$. Gratings G1 and G3 are mounted for studies in the soft X-ray and VUV regions, respectively, and have a groove density of 2400 lines/mm and radii of curvature of 55.21 m and 22.945 m, respectively. Scanning of the photon energy is achieved by changing $\theta_M$ and $\theta_G$, so that the requested energy beam is focused on the exit slit S2. Elliptic mirrors, VPFM and HPFM, supply vertically and horizontally focused light to the reflectometer located 30.3 m downstream.

![Figure 1. Monochromator and post-focusing mirrors at BL-11D, PF.](image)

On the basis of the monochromator design, the zero defocus-term condition and grating equation determine a unique $\theta_M$-$\theta_G$ combination for a given photon energy. The calculated $\theta_M$ and $\theta_G$ values are plotted in figure 2 by the thick curves. In contrast, the monochromator output energy was measured by photoelectron spectroscopy at several $\theta_M$-$\theta_G$ combinations in the 300–700 eV region and the data were fitted to fourth order polynomials as shown by the dotted curves. The present software accepts only polynomial functions and the monochromator has actually operated along these curves. Abnormal behavior was found outside the 300–700 eV region. The C 1s-$\pi^*$ absorption, for example, appeared at an energy 13 eV higher than expected. The situation in the VUV region covered by G3 is similar. The operable range is 60–200 eV, and the intermediate region 200–300 eV remains unexploited.

![Figure 2. Mp and G1 rotation angles of zero defocus-term condition (thick), actual operation (dotted), 250–300 eV fitting (thin) and 700–1000 eV fitting (dashed).](image)

3. Monochromator tune up in the 250–300 eV and 700–1000 eV regions
For reflectance measurements of multilayer mirrors used in the carbon window region, the monochromator has to be tuned up with G1 to an energy range of 250–300 eV. The designed $\theta_M$ and $\theta_G$ were fitted to fourth order polynomials in this region, as shown by the thin curves in figure 2. This $\theta_M$-$\theta_G$ combination appears to work well through 200–350 eV.
The performance of the monochromator was tested through spectral reflectance measurements of a Cr/C multilayer at various angles of incidence. The period thickness, Cr layer thickness to period thickness ratio, and period number of the multilayer are $D = 3.06 \text{ nm}$, $\gamma = 0.32$, and $N = 200$, respectively. Reflectances calculated using optical constant data from CXRO with an interface roughness of 0.3 nm are plotted in figure 3(a). The ordinate is magnified by 10 above the C K-edge. Angles of incidence were chosen so that the scattering vector interval was constant. The measured reflectances are plotted in figure 3(b). An AXUV100Ti/C2 (IRD Inc.) photodiode was used as the detector. No unexpected broadening was observed in the spectra. Compared to the calculation, the peak energy difference was within 0.1% in the 230–280 eV region. The spectral accuracy and resolving power were confirmed to be high enough for characterization of the carbon window multilayers. The residual of the polynomial fitting will cause an energy shift of $-0.3\%$ around 200 eV, but a larger energy shift of $-0.8\%$ was found, which might be attributed to an error in the monochromator parameters such as the focal length of $M_f$.

Figure 3. (a) Calculated and (b) measured spectral reflectances of a Cr/C multilayer.

While the detector was coated with 200 nm of Ti, no structure resembling the Ti $L_{2,3}$-absorption was observed around 230 eV. This indicates that the monochromator is free from second order diffraction. We also measured the Co $L_{2,3}$-absorption to determine whether any third order diffraction was present. A similar procedure as 250–300 eV was carried out in the 700–1000 eV region. The fits to the polynomials are plotted as dashed curves in figure 2. For this $\theta_M-\theta_G$ combination, a normal incidence transmittance was measured in the 750–810 eV region. A Co/C multilayer ($D = 2.29 \text{ nm}$, $N = 130$) deposited on a Si$_3$N$_4$ membrane was used as the sample. A similar measurement was performed in the 250–310 eV region along the 250–300 eV optimized $\theta_M-\theta_G$ curves. The results are plotted in figures 4(a) and 4(b). A small dip was found at 275 eV, which could be attributed to a reflection loss of the multilayer. Comparing the structure in the 250–270 eV region with that in 750–810 eV region, it can be concluded that third order diffraction is negligible in the carbon window region. However, a finite background was found for 290–300 eV, where the C 1$s-\sigma^*$ absorption appears to saturate. Thus, the only possible reason is the scattered lower energy lights.

Figure 4. Spectral transmittances of a Co/C multilayer and a free-standing Cu film, and spectral reflectance of Ni at a 4.5° grazing incidence.
Absorption features of the Ni and Cu L$_{2,3}$-edges from a 40 nm thick Ni monolayer deposited on a SiO$_2$ substrate in a 4.5° grazing incidence reflection geometry and a 500 nm thick free-standing Cu film in normal incidence transmission geometry are also shown in figure 4(b). All the results show that the monochromator will operate with a 1% spectral accuracy in the 200–1000 eV range.

4. Reflectance measurements of Co/C multilayers
Co/C multilayers ($D = 2.30$ nm, $N = 175$) were simultaneously deposited on two Si wafers. Co/C multilayers ($D = 2.28$ nm, $N = 130$) were simultaneously deposited on one of the first Co/C multilayers and on a clean Si wafer. The spectral reflectances of the three samples measured at a 5° angle of incidence are shown in figure 5(a). The multilayer period thicknesses were designed so that the transmittance peak of the second multilayer should agree with the reflectance peak of the first multilayer. This produces a small sub-peak and increases the integral reflectance slightly. The difference between the spectral reflectances of the second multilayer and the double multilayer was clearly observed. When the detector was replaced by an AXUV100Ti/Zr/Al (IRD Inc.) detector, the reflectances increased as shown in figure 5(b) possibly because the thicker coating reduced the lower energy impurity. Thus, a careful choice of filters is important for accurate reflectance measurements.

![Figure 5](image-url)

Figure 5. Spectral reflectances of Co/C multilayers measured using an (a) AXUV100Ti/C2 and (b) AXUV100Ti/Zr/Al photodiode.

5. Conclusions
We have programmed a monochromator operation optimized in the 250–300 eV region at BL-11D, PF. A spectral resolution and spectral accuracy of 0.1% were achieved. No higher order diffraction components were observed but a lower energy impurity was found. We have also programmed a monochromator operation above 700 eV. The operable range of the monochromator has been extended to 200–1000 eV.

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References
[1] PF Act. Rep. 2000 A 2001 #18 69–71
[2] Hatano T, Kondo Y, Saito K, Ejima T, Watanabe M and Takahashi M 2002 Surf. Rev. Lett. 9 587–91
[3] Hatano T, Harada T, Matsushita T, Arakawa E and Higashi Y 2010 AIP Conf. Proc. 1234 663-6
[4] Artyukov I A, Feschenko R M, Vinogradov A V, Bugayev Ye A, Devizenko O Y, Kondratenko V V, Kasyanov Yu S, Hatano T, Yamamoto M and Saveliev S V 2010 Micron 41 722–8