

(BL4A1)

Al thin film deposition on Si substrate surface stimulated by monochromatized SR

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We constructed a multilayered-mirror (MLM) monochromator beam line at the UVSOR for the purpose of SR stimulated process studies. Based on our previous experiments of Al thin film deposition using dimethylaluminum hydride (DMAH),¹ we consider a total photon flux density higher than 10^{18} photons/cm² as a criterion for the necessary total flux, in the SR stimulated process experiments. This means that a monochromatized-photon flux larger than 10^{13} - 10^{14} photons/s is needed, if we consider a reasonable irradiation time (a few tens of minutes to a few hours) and an irradiation area ($\geq 3 \times 3$ mm²) on the substrate surface applicable even to the device fabrications.

The output beam of the constructed beam line was applied to the Al thin film deposition using DMAH low temperature condensed layer. Details about the sample treatments are described in ref. 1. Si (100) substrate treated by conventional wet process was introduced into the reaction chamber. After cooling to about 100K, the DMAH gas was introduced and deposited on the substrate (10–20 monolayers), then, the monochromatized SR beam ($\theta = 50^\circ$, 84 eV) tuned to the Al 2p core electron excitation was irradiated for about 56 minutes at the average ring current of 100 mA. (This corresponds to the calculated total photon flux of 5×10^{17} photons/cm² on the sample surface). After the irradiation, the substrate temperature was increased to the room temperature to remove the unreacted DMAH, the composition of the deposited film was measured by XPS. As shown in Fig. 1, the deposited Al thin film was observed visually, although the thickness was not measured. An interesting point is the C concentration of the deposited film. The XPS spectra observed for the DMAH low temperature condensed layer is shown in Fig. 2A, and those for the deposited Al film is shown in Fig. 2B. By assuming C/Al=2 for DMAH condensed layer, the composition C/Al of the deposited film is estimated to be about 0.5. This value is quite small compared with those of films obtained by the white or filtered white SR beams irradiations. This may be due to that the Al-C bond is preferentially broken by the Al core electron excitations. To discuss the excitation energy dependence, much more detailed experiments are necessary. The data shown in Fig. 2, however, indicates clearly that the MLM monochromator beam line constructed have a sufficient performance as an SR stimulated process beam line.

Reference

1) Y. Imaizumi, Y. Tusaka, T. Urisu, and T. Ibuki, *Jan. J. Appl. Phys.* **35**, 6588 (1996)

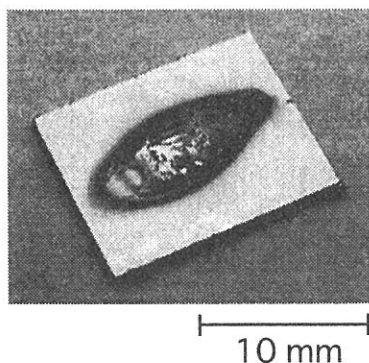


Figure 1. Photograph of the deposited Al thin film.

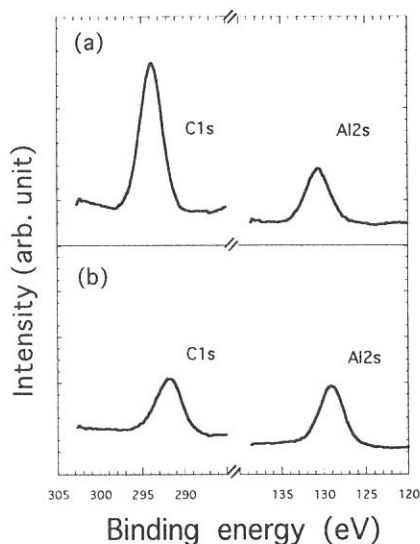


Figure 2. The observed XPS spectra for (A) the DMAH condensed layer at 105K and (B) the deposited Al thin film.

(BL4A1)

Performance of the Multilayered-mirror monochromator and the beam line BL4A1

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In this paper, we describe the performance of a double crystal type MLM monochromator combined with an appropriate thin-film filter that reduces the total reflection component, developed for SR stimulated process experiments. The MLM monochromator was installed in a beamline (BL4A1) of the UVSOR storage ring in the Institute for Molecular Science (IMS). The photon flux and the monochromaticity of the output beam were experimentally evaluated for the case of using Mo/Si MLMs combined with a C filter.

We first calculated the photon flux of the output beam as a function of photon energy, as shown in Fig. 1, taking into consideration the acceptance angles and reflectivity of the pre-mirror, the reflectivity of two MLMs calculated by assuming an ideal structure, and the transmission of the C filter. The transmission (84.4%) of the mesh holding a thin C film was also considered in addition to the transmission of the 120-nm-thick C filter. The characteristics of the output beam from the MLM monochromator beamline were estimated using Mo/Si MLMs and the C filter. A 120 ± 24 -nm-thick C filter was set behind the MLM monochromator to reduce the low energy background existing less than 40 eV. A 150 ± 30 -nm-thick Al filter was set at the downstream of the C filter to evaluate the monochromaticity of the output beam by measuring the transmission spectrum of the Al filter near the Al $L_{2,3}$ absorption edge. The intensities of the beam at the downstream of the C and the Al filter were measured by the Si photodiode detector as well as by the Au detector.

Next, we measured the dependence of the photon flux on θ using the Si photodiode detector and compared it to the calculation, as shown in Fig. 2. The calculated flux is obtained by integrating the photon flux spectrum in the photon energy range from 0 to 2 keV for various θ values in Fig. 1. The monochromator does not operate at the normal incident angle side ($\theta \leq 10^\circ$) because of the significant reductions of the MLM reflectivity and the transmission of the C filter. It also does not operate at the grazing incident angle side ($\theta \geq 55^\circ$) because of the significant increase of the background. If we require for the photon flux to be more than 1.0×10^{12} photons/s and for the background to be less than 15% (in the case of using Mo/Si MLMs combined with the C filter), the incident angle range which gives a practical output beam is determined to be between 10° (55 eV) and 55° (93 eV), as shown by dotted lines a and b in Fig. 3. The maximum intensity of the

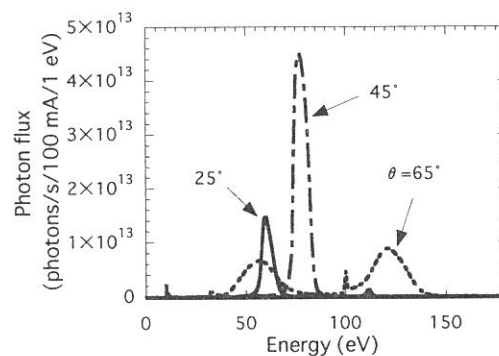


Fig. 1. Calculated spectra of the photon flux of output beam of the MLM monochromator beamline (BL4A1) using Mo/Si MLMs and a C filter (thickness 120nm).

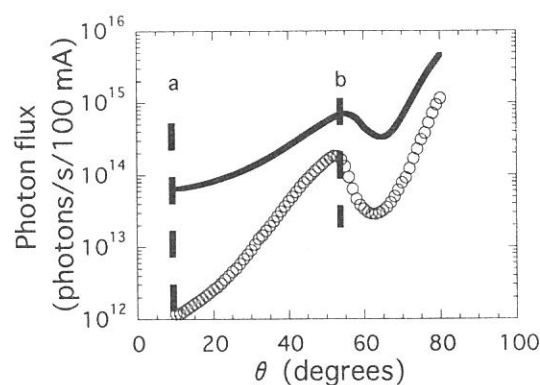


Fig. 2. The dependence on the incident angle θ of the photon flux of output beam. The solid line shows calculated values and “○” shows measured values. The dotted lines a and b show the working region of the monochromator.

photon flux of the output beam is obtained at about $\theta = 55^\circ$. The reduction of the photon flux over $\theta = 55^\circ$ is due to the absorption of Si (Si $L_{2,3}$) used as a MLM material. The measured photon flux at 55° was about 24% of the calculated value as shown in Fig. 3. If we attribute this difference of the measured value and the ideal value equally to the incompleteness of the pre-mirror and two MLMs and possible cut of the beam edge by the C filter, then the MLM's reflectivity would be estimated to be about 10% of the ideal value. We think this is a reasonable value, considering that we did not pay special attention to obtain MLMs with high reflectivity.

To evaluate the spectrum width, the contribution of the higher-order photon, and the low-energy background caused by the total reflection, the transmission characteristics of the monochromator output beam for the Al filter near the Al $L_{2,3}$ absorption edge were measured as a function of θ . The results are compared with the calculation in Fig. 3. The calculated transmission was obtained by taking the ratio between the integrated photon flux behind the C filter and that behind the Al filter for each θ value. In the calculation, the thickness of the Al filter was varied within the catalog value 150 ± 30 nm to fit the calculation to the experiments. The broken curve in Fig. 3 was calculated for a thickness of 180 nm. Considering the error of the optical constants with MLM and filter materials and the structural incompleteness of the MLM, for which precise evaluations are difficult, the calculated results agree well with the experimental results. Thus, we conclude that the spectrum width should be almost equal to the calculated width (5 - 9 eV in 55 - 93 eV peak energy range), and the background is almost equal to the level predicted by the calculation.

To confirm these conclusions, we measured the photo-emission spectra of Ta, which is expected to give two dominant peaks (4f and 5d) close to the Fermi edge for an excitation energy of 50 - 100 eV, using the monochromator output photons as an excitation light source. A CL150 analyzer and a HAC300 controller (VSW Inc.) equipped in the XPS chamber were used in this measurement. After acetone and methanol washing, the Ta substrate was introduced into the XPS chamber and heated up to about 850 K for 4 hours. The observed photo-emission spectra, excited by an output beam of $\theta = 20^\circ$, without filters

(a) and with the C filter (b), are shown in Fig. 4. The clear peak assigned to the Ta 5d bands is observed in Fig. 4 (b), but not in (a). This indicates that the C filter sufficiently reduces the low-energy background. We confirmed that the present MLM monochromator using a Mo/Si MLM in combination with a C filter works well as predicted by the calculation in the energy range from 55 eV ($\theta = 10^\circ$) to 93 eV ($\theta = 55^\circ$). In this range, the photon flux of output beam was $1.0 \times 10^{12} - 1.5 \times 10^{14}$ photons/s, the spectrum width was 5 eV ~ 9 eV, the low-energy background was less than 7.2%, and the higher-order (second-order) photon background was less than 12%. We conclude that the MLM monochromator beamline (BL4A1) constructed here performs sufficiently well for studying the excitation-energy dependence in the SR stimulated processes.

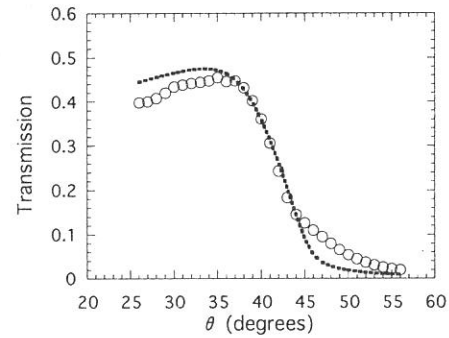


Fig. 3. The transmission of the Al filter in the vicinity of the Al $L_{2,3}$ absorption edge measured (“○”) by using output beam of the monochromator as a function of θ . The dotted line represents calculated values. (An Al filter thickness of 180 nm and the transmission of 74.8% for the mesh holding the thin Al film are assumed.)

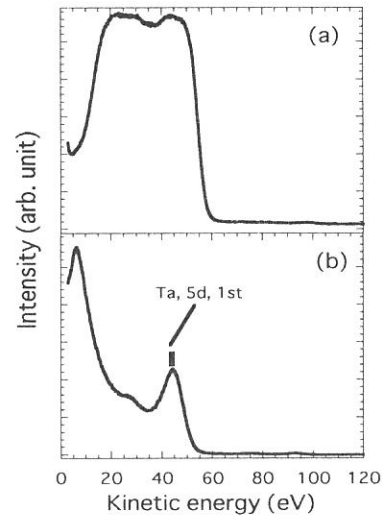


Fig. 4. The photo-emission spectra of Ta excited by the output beam from the MLM monochromator. Mo/Si MLMs are used and $\theta = 20$ degrees: (a) the case without the C filter and (b) the case with the C filter. “1st” means the peak excited by the first-order photon component of the MLM monochromator output.