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DESIGN OF A FLAT FIELD SPECTROMETER FOR SOFT X-RAY EMISSION SPECTROSCOPY

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A spectrometer for soft X-ray emission spectroscopy was designed and constructed. The aims of this spectrometer are high detection efficiency and realizing the energy resolution of 1000 ($E/\Delta E$) in a compact size. To satisfy these requirements, a flat field spectrometer with a varied line spacing grating and a CCD detector was chosen. Furthermore, the configuration without the entrance slit was employed. For compact flat field spectrometers, the spectral dispersion on the detector is sometimes smaller than the spatial resolution of existing detectors restricting maximum energy resolution. The spectral dispersion on the detector was optimized to balance with the spatial resolution of CCD. Optical performance was tested with a ray-tracing program. The spectrometer covers 250–900 eV, with two gratings. Taking into account the spatial resolution of a CCD detector, the maximum energy resolution of the spectrometer is estimated to be about 1000 ($E/\Delta E$) for the beam size of 10 μm. Using the constructed spectrometer, test experiments were performed at the beamline BL27SU in SPring-8.

1. Introduction

Soft X-ray emission spectroscopy (SXES) is a powerful tool for studying the electronic structure of various materials,[1] including solution and wet samples. We have designed and constructed a new spectrometer aiming to do experiments on adsorbate, solution, and biological samples. As the intensity of soft X-ray emission from adsorbate and solution samples is low, and biological samples are easily damaged by irradiation of soft X-rays, a spectrometer with high detection efficiency is required.

Spectrometers for SXES are usually of the Rowland mount type, with a constant line spacing spherical grating.[2] This type of spectrometer needs to set all optical elements on the cylindrical surface defined by curvature of the grating. Therefore, the detector must also be aligned at grazing incidence to fit the cylindrical surface in the soft X-ray region.

In addition, detectors are typically planar, so that they can never fit the Rowland circle at all points. To access another energy region, the detector must be moved along the Rowland circle with a mechanical stage with high precision.

For the detection of soft X-rays, MCP (micro channel plate) and CCD (charge coupled device) are normally used. The spatial resolution of CCD is higher than that of MCP. Concerning the detection efficiency in the soft X-ray region, Skinner and Schwob reported that CCD has higher efficiency in comparison with MCP even at grazing incidence.[3] They have also pointed out that the sensitivity decreases at shallow incident angle due to the absorption in the insensitive surface layer. In order to fully utilize the high efficiency of CCD, another type of spectrometer with a large incidence angle at CCD is preferable.

Flat field spectrometers using spherical VLS (varied line spacing) gratings[4–6] are suitable for CCD because the incident angle at CCD can be optimized by changing optical parameters of the grating. Furthermore, this type of spectrometer has a nearly straight focal curve and matches well with the flat surface of the CCD detector. Simple linear motion of the detector is enough to access other energy regions.

Consequently, the combination of a flat field spectrometer and a back illuminated CCD detector was chosen for our high efficiency spectrometer system. The optical parameters were optimized to maintain the detection efficiency and the energy resolution of 1000 in the energy range of 250–900 eV. The energy resolution was estimated using a ray-tracing program. Test experiments using synchrotron radiation were also performed. A schematic view of the constructed spectrometer is shown in Fig. 1.

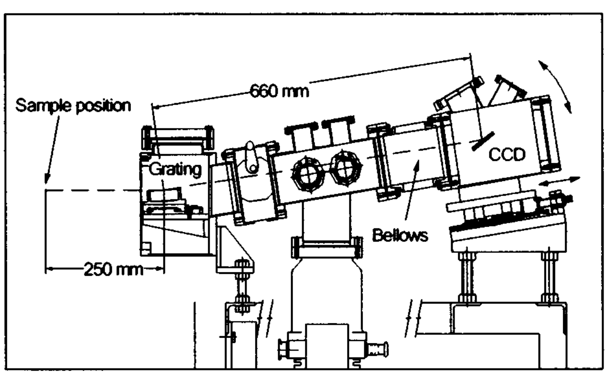


Fig. 1. Schematic view of the spectrometer. The CCD chamber is possible to rotate and slide along the direction indicated by arrows for initial adjustments.

2. Optimization of the Optical Parameters

The groove spacing of a spherical VLS grating is described as[7]

$$d(w)d_0\Big/\left(1+\frac{2b_2}{R}w+\frac{3b_3}{R^2}w^2+\frac{4b_4}{R^3}w^3+\cdots\right), \quad (1)$$

where d_0 is the groove spacing at the center of a grating, R is the radius of the spherical grating surface, and b_2, b_3, b_4 are parameters for correcting aberrations.

Using the $F_{20} = 0$ condition of the light path function,[8] the focal curve along the direction of spectral dispersion can be described as

$$r' = \frac{rR\cos^2\alpha}{r[\cos\alpha+\cos\beta-2b_2(\sin\alpha+\sin\beta)]-R\cos^2}, \quad (2)$$

where r' is the focal distance from the center of the grating, α is the incident angle of the grating, β is a diffraction angle of the grating, and r is the distance between the light source and the grating center (see Fig. 2).

If the parameters of r, $r_0{}'$, α and β_0 are fixed temporarily, the radius of the grating is expressed as

$$R_0(b_2) = \frac{\cos\alpha+\beta_0+2b_2(\sin\alpha+\sin\beta_0)}{\dfrac{\cos^2\alpha}{r}-\dfrac{\cos^2\beta_0}{r'_0}}, \quad (3)$$

and becomes a function of b_2. Substituting R_0 for R in Eq. (2), the focal curve becomes a function of b_2.

Integration of the deviation distance between the focal curve and the detector surface for the required energy range has a local minimal value for the

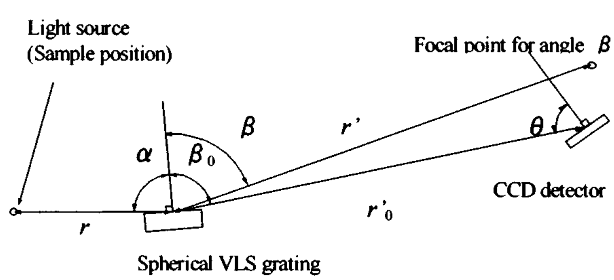


Fig. 2. Optical parameters of a flat field spectrometer.

Table 1. Detailed parameters of the spectrometer.

	r	r'	α	β_0	R	θ	Size	d_0	b_2	b_3	b_4
Grating 1 (250–450 eV)	250	660	87	−84.58 (600 eV)	7067.10	67.94	54 × 26	1500	−4.22	−45.6	−3221
Grating 2 (400–900 eV)	250	660	88	−83.58 (525.65 eV)	11243.51	62.88	54 × 26	2400	−10.69	40	−8291
units	mm	mm	degree	degree	mm	degree	mm	lines/mm	–	–	–

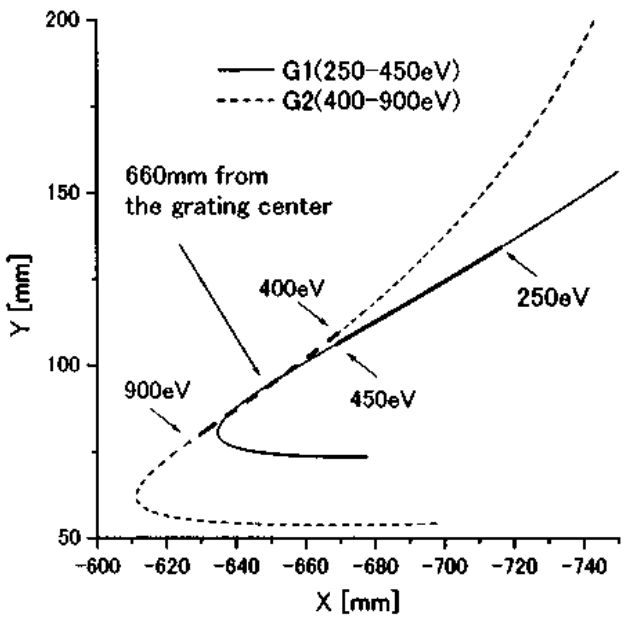
optimized b_2 parameter, and can be used to judge the straight focusing characteristics. Other VLS grating parameters, b_3 and b_4, are determined to keep the energy resolution in the required range.

The optimized optical parameters are summarized in Table 1. In order to gain the detection efficiency, the configuration without the entrance slit was employed, because the source size of 10 μm can be achievable at the recent high performance soft X-ray beamline. The shorter distance r is preferable to gain the acceptance and the detection efficiency, and was determined as 250 mm considering the physical restriction of the existing vacuum chamber. Angular acceptance of the gratings in vertical direction is 11 mrad (grating 1) and 7.6 mrad (grating 2). The shorter distance r' is also desirable to make the spectrometer compact. The spectrometer is required to cover the energy range of 250–900 eV with the energy resolution ($E/\Delta E$) of 1000. In order to satisfy these conditions, two gratings with the parameters shown in Table 1 are equipped. The incident angles θ at CCD are 67.94° and 62.88° for gratings 1 and 2, respectively.

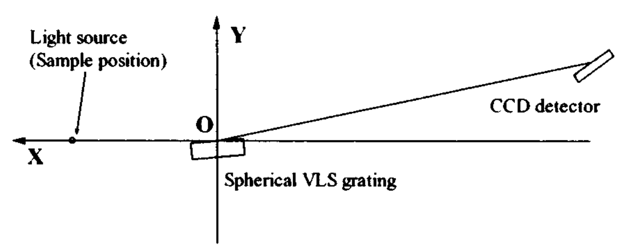
3. Performance of the Spectrometer

Figure 3 shows the focal curves for two gratings. It should be noted that each grating has the nearly straight focal curve in the energy range of 250–450 eV (grating 1) and 400–900 eV (grating 2), respectively. The aimed energy region can be accessed easily by a simple linear motion of the detector after the initial adjustment of the CCD chamber (see Fig. 1). When the grating is exchanged, we must readjust the CCD chamber. However, this procedure is easily done by rotating the chamber because the focal curves of two gratings have a crossing point at 660 mm from the grating center (see Fig. 3).

Optical performance was checked with a personally developed ray-tracing program. Calculated spot diagrams on the detector surface are plotted in

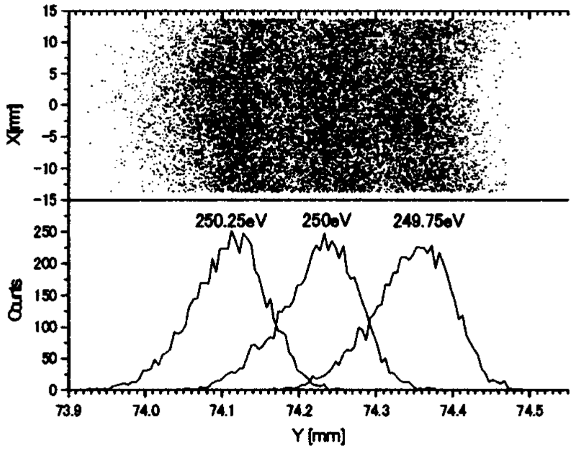


(a)

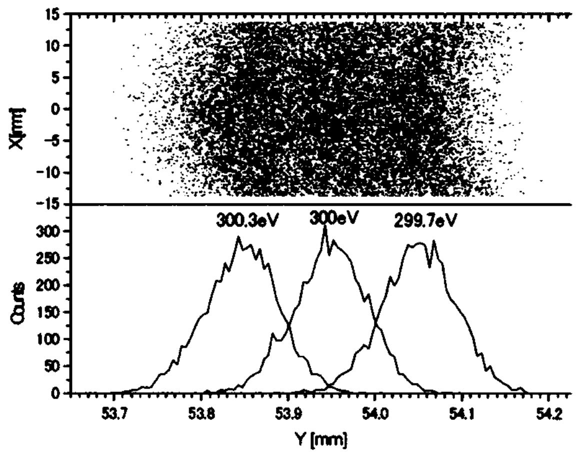


(b)

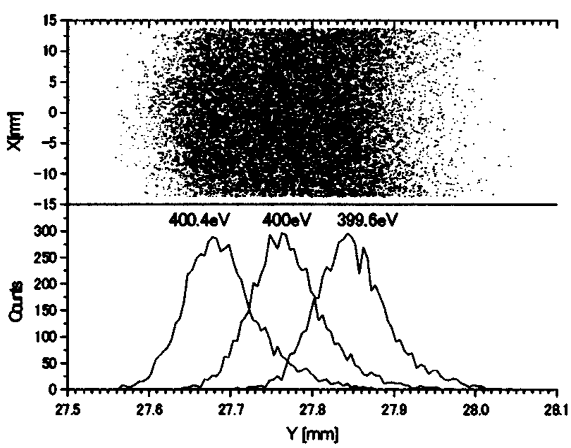
Fig. 3. (a) Focal curves of the flat field spectrometer (narrow lines) and the detector surface (bold lines). Focal curves of gratings 1 and 2 have a cross point at 660 mm from the center of the grating. (b) X and Y axes of coordinates for calculations of focal curves.



(a)

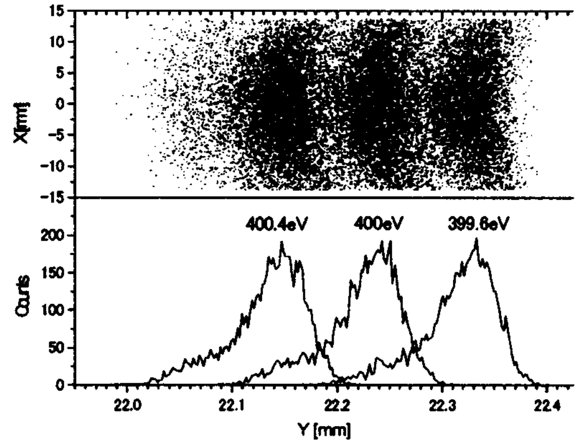


(b)

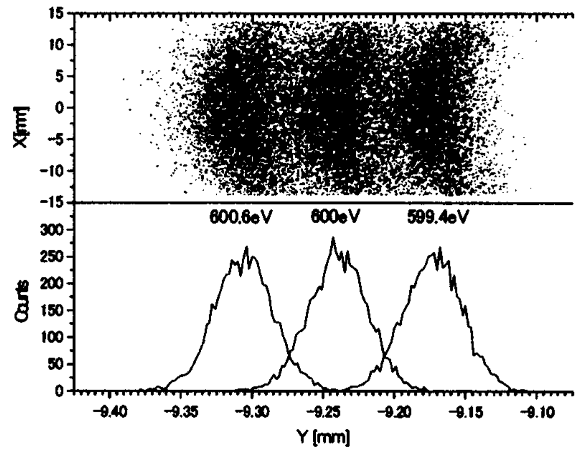


(c)

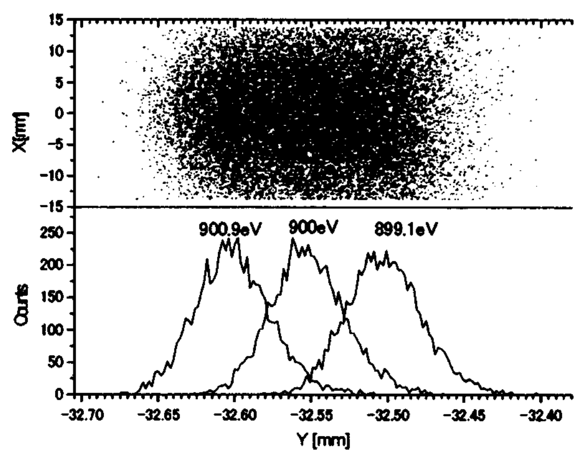
Fig. 4. Calculated spot diagrams on the detector surface for grating 1 at photon energy of (a) 250 eV, (b) 300 eV and (c) 400 eV. The range of the X axis is defined by the detector width (27.5 mm).



(a)



(b)



(c)

Fig. 5. Calculated spot diagrams on the detector surface for grating 2 at photon energy of (a) 450 eV, (b) 600 eV and (c) 900 eV. The range of the X axis is defined by the detector width (27.5 mm).

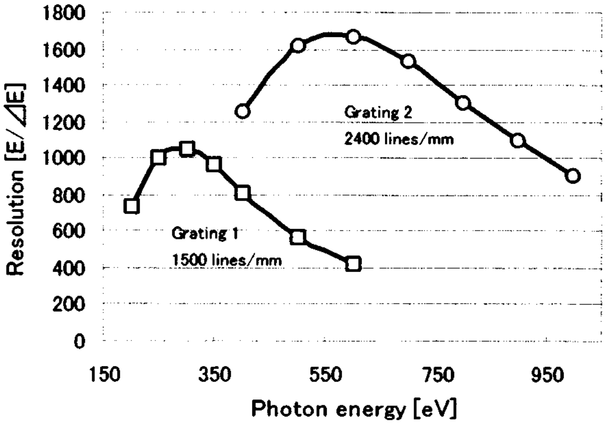


Fig. 6. Estimated energy resolution of the optical system for the beam size of 10 μm. The contribution of the spatial resolution of the detector is not included in this calculation.

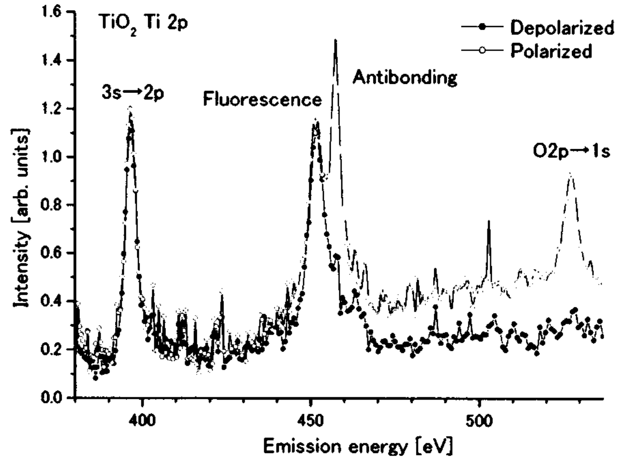


Fig. 7. Soft X-ray emission spectra of TiO_2 at the Ti 2p edge. The O 2p $\rightarrow$ 1s peak is caused by second order light of the beam line spectrometer.

Figs. 4 and 5. The energy resolution of our optical system was estimated from these spot diagrams and is more than 1000 in the almost energy range of 250–900 eV, as shown in Fig. 6. It is very important that the spectral dispersion matches the spatial resolution of the detector. From the line profiles in Figs. 4 and 5, we can judge that the FWHMs of each profile correspond to the total width of at least 5 pixels of CCD with the pixel size of 13.5 μm. Taking into account of the spatial resolution of CCD, the energy resolution is estimated at about 1000 ($E/\Delta E$) for the spot size of 10 μm.

4. Test Measurement of the Spectrometer

We have tested the performance of the spectrometer by measuring the soft X-ray emission spectra of TiO_2 at the Ti 2p edge. The experiments were performed at the beamline BL27SU,[9,10] in SPring-8, where vertically and horizontally polarized light can be switched by changing the undulator gap. Our CCD detector has 2048×2048 pixels with a pixel size of 13.5 μm, its detection area is 27.6 mm $\times$ 27.6 mm. The detector is cooled down to 173 K using liquid nitrogen to reduce the thermal noise. Preliminary results are shown in Fig. 7. The spectral feature is essentially the same as the published one.[11,12] The estimated energy resolution ($E/\Delta E \approx 100$) is not high, but is reasonable because the actual beam size measured with a pinhole was about 100 μm, which was ten times worse than the expected value. It is hard to mention quantitatively the detection efficiency of our present system, from these spectra in comparison with the other system, such as a Rowland type spectrometer with a MCP detector. However, it should be emphasized that the acquisition time for each spectrum was only 10 min.

5. Conclusion

A flat field spectrometer for soft X-ray emission spectroscopy was designed and constructed. This spectrometer covers 250–900 eV with two gratings. The maximum energy resolution of the spectrometer is estimated to be about 1000 ($E/\Delta E$) for the beam size of 10 μm, taking the spatial resolution of a CCD into account. Test experiments were performed at the beamline BL27SU in SPring-8. The soft X-ray emission spectra of TiO_2 at the Ti 2p edge were obtained with a short acquisition time. The achieved energy resolution was not high due to the unexpected large spot size of 100 μm. The smaller beam size of 10 μm will certainly be achieved soon, and it will improve the energy resolution.

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