Studies on the Extremely Soft X-ray Absorption Spectrometry at BL-2

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Abstract

We measured visible photoluminescence spectra under extremely soft X-ray excitation at BL-2. MgO single crystal emits a broad band at 728 nm and α-Al_2O_2 single crystal shows a sharp band at 614 nm and a broad band at 700 nm. In the case of ZnS(Ag), the intensity of photoluminescence was increased with increasing the excitation energy from 50 to 750 eV, especially above 600 eV.

BL-2 is a Rowland mount type beamline with a 1200 groves/mm concave curved grating, and the Rowland radius is 9581.6±31.7 mm and the incidence angle is 89.103±0.005 deg. BL-2 provides us with photons of the energy of 47 to 700 eV, but the actually useful energy region is 50 to 250 eV because of the carbon contamination on optical elements and the energy resolution. The real radius of the grating is 1500 mm, therefore the present optical setting is not the best, but good absorption data of lithium, boron, magnesium and aluminum could be obtained. BL-2 will have more and more enough ability for extremely soft X-ray total electron yield (TEY) measurements and for other optical experiments after precise optical setting by reconsidering electronic devices.

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1. Introduction

Electromagnetic waves are very effective and interesting probes in order to understand the rotation and vibration of molecules, the electronic structure or atomic distribution of solids, etc. Synchrotron light source provides high quality electromagnetic waves from far-infrared light to hard X-ray. In 1996, AURORA was installed, and the construction of BL-2 was started to use in the X-ray absorption spectroscopy (XAS) and X-ray emission spectroscopy in the extremely soft X-ray region. From 2003, it was only used for the XAS investigation.

BL-2 is composed of the quite simple optical system: one bent mirror, an incident slit, a concaved grating and an outgoing slit. The bent mirror is focusing 1.0 mrad extremely soft X-ray in the horizontal direction at the incident slit and removes higher energy X-ray. Two slits and the grating make a Rowland circle, the ideal radius being 3000 mm. The energy region provided at BL-2 is from 50 to 500 eV, therefore it covers the lithium, beryllium, boron, carbon, nitrogen K-edge. On the other hand, the vacuum is not reached at the level of ultra-high vacuum, therefore optical components suffer carbon contamination. It has a serious problem to investigate the carbon or nitrogen region because of the contamination.

We had an opportunity for four days trial experiments at BL-2, and have succeeded in measurements of several wonderful L-edge spectra by total electron yield (TEY) method and visible luminescence spectra under extremely soft X-ray excitation.

2. Standard Spectra at BL-2

Samples were powders of lithium halides, beryllium foil, hexagonal type boron nitride (h-BN) powder, single crystals of lithium fluoride, magnesium oxide and sapphire (α-Al_2O_3). Fig. 1 shows Li K-edge XANES spectra of lithium halide powder samples that were annealed at 423 K for several hours in order to dry. Tsuji et al. reported that the energy of a peak for an atom shifts according to electron negativity of a neighboring atom, and they expected that the peak energy for LiCl is in the region of 60.7 and 60.9 eV[1]. This is good agreement of our new results for Li K-edge XANES experiments of lithium halides.

Fig. 2 shows Li, Be and B K-edge XANES spectra of LiF powder, Be foil and h-BN powder. From the maximum positions of the sharp resonance peaks, the roland radius can be estimated to be 9581.6$\pm$31.7 mm, and the incident angle to the concaved grating to be 89.103 deg.

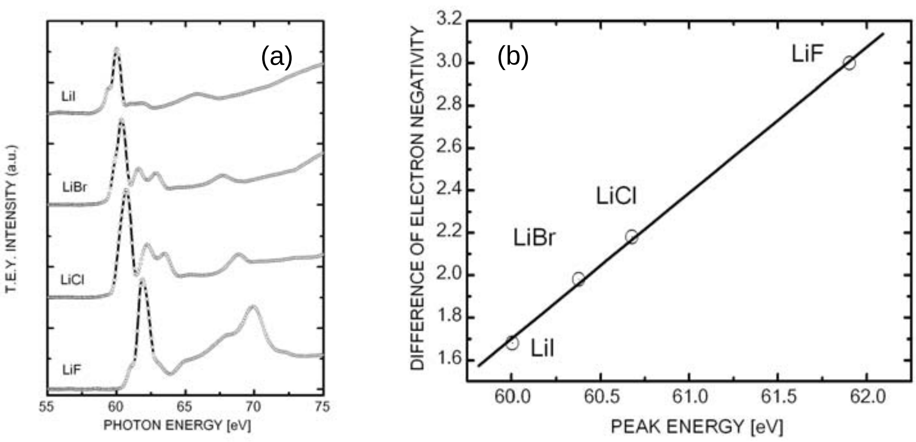

Fig. 1. XAS spectra by TEY method. (a) Lithium K-edge XANES of lithium halide powders, (b) relation between the energy of the first resonance peak and the electron negativity difference.

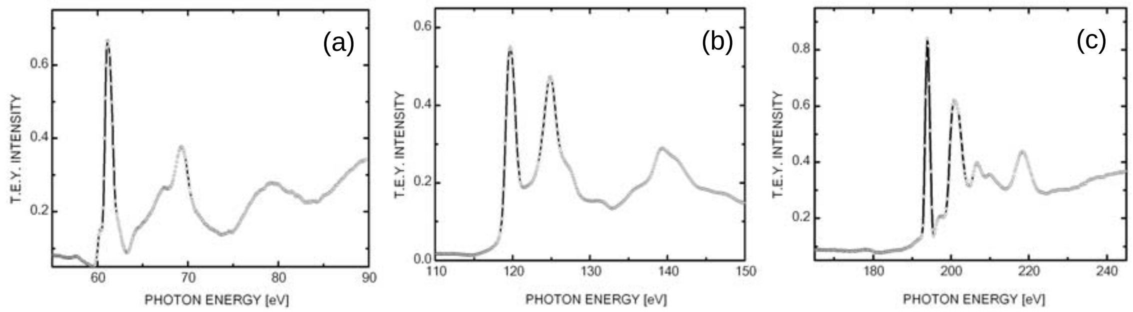

Fig. 2. XAS spectra by TEY method. (a) Lithium K-edge XANES of LiF powder, (b) beryllium K-edge XANES of Be foil, and (c) boron K-edge XANES of h-BN powder.

In these trial experiments, we obtained some XAS spectra of single crystals. The samples of single crystals were lithium fluoride, magnesium oxide and sapphire, and XAS measurements were done by TEY method for Li K-edge, Mg L-edge and Al L-edge, respectively. The details of the peak assignments were discussed by Tanaka, et al. [2]. From the comparison between Figs. 2a and 3a, the intensity ratio of the peak at 62 and 70 eV is much larger in the single crystal sample than the powder sample.

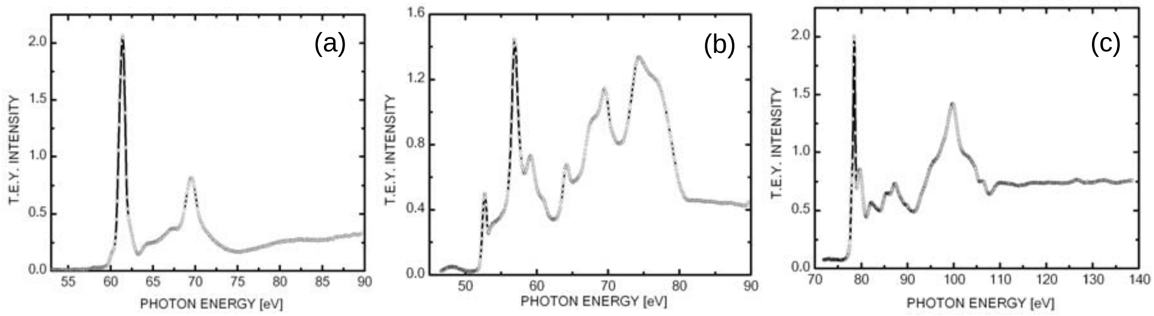

Fig. 3. XAS spectra of single crystals. (a) Lithium K-edge XANES of LiF single crystal, (b) magnesium L-edge XANES of MgO single crystal, and (c) aluminum L-edge XANES of sapphire (α-Al_2O_3) single crystal.

3. Visible luminescence spectra under extremely soft X-ray excitation

Zinc sulfide doped with silver, ZnS(Ag), is a famous fluorescent material and used for the cathode-ray tubes of color televisions. The luminescence intensity is very strong and shows blue color, and it is also well known that it emits under X-ray excitation. We have used ZnS(Ag) powder as an X-ray fluorescent material to check the beam position, etc. However, the luminescent spectrum excited by extremely soft X-ray has not been reported. Fig. 4 shows visible luminescent spectra of ZnS(Ag) excited by extremely soft X-ray. The shape of the emission band is not changed with the change in the excitation energy, but the intensity is increasing with an increase in the excitation energy.

Fig. 5 shows visible luminescence spectra of a green fluorescent glass, MgO and sapphire single crystals under extremely soft X-ray excitation. The fluorescent glass shows green luminescence under extremely soft X-ray excitation as well as the case of UV excitation, and the main peak is located at 540 nm. It was also observed that MgO single crystal emits a broad band, but the main peak in the vicinity of 200 nm could not be observed because of the restriction of the performance of optical instruments used. Sapphire also emits visible bands, the peaks of which are located at 600 and 700 nm.

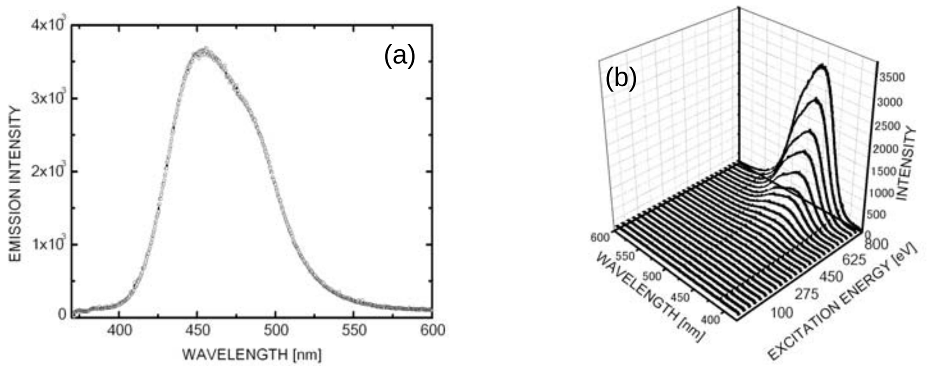

Fig. 4. Visible luminescence spectra of ZnS(Ag) powder under extremely soft X-ray excitation. (a) Excitation energy = 700 eV and (b) excitation energy = 50-700 eV.

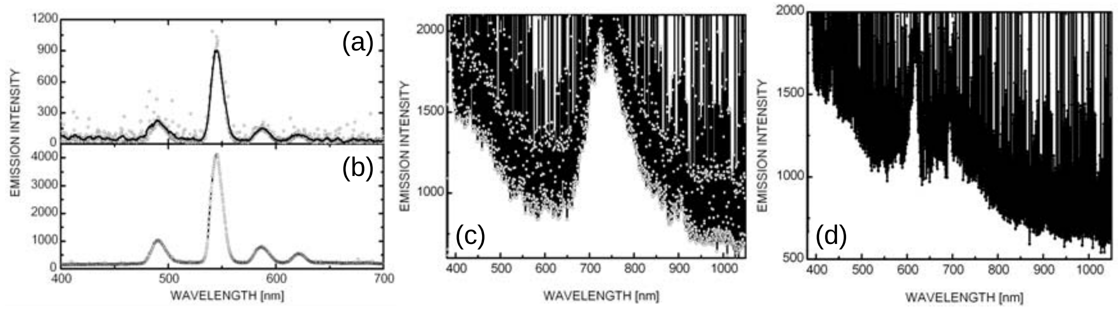

Fig. 5. Visible luminescence spectra under extremely soft X-ray excitation of 700 eV for (a) a green glass, (c) a MgO single crystal, and (d) a sapphire. Fig. 5(b) is a luminescence spectrum of the green glass excited at 365 nm.

4. Conclusion

BL-2 is an extremely soft X-ray beamline with the possibility of developments by the correction of the optical setting and by the settlement of the carbon contamination. The present setting of the beamline is not the best, however good absorption data of lithium, boron, magnesium and aluminum could be obtained. Visible photoluminescence spectra under extremely soft X-ray excitation could also be obtained. These experimental results show only a part of the beamline performances and new usages. BL-2 must be evolving by ceaseless efforts and provides users with new and interesting data.

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