Performance evaluation of a new soft X-ray double crystal monochromator beamline, BL-13

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Abstract

A new soft X-ray double crystal monochromator beamline was designed and constructed at BL-13 last year. Two toroidal mirrors were installed in this beamline, one for producing a quasi-parallel beam and another for focusing the beam at the sample position. The use of the two toroidal mirrors allows us to obtain high energy resolution spectra without expensive parabolic mirrors and focus the beam at the sample position in a size of 3.0 mm^H x 1.5 mm^V. The grazing incidence of X-rays to the first toroidal mirror was set to 0.6° with respect to the mirror surface. This setup extends the available energy up to 5000 eV.

1. Introduction

The X-ray absorption fine structure (XAFS) spectroscopy has been developed in the last decades as a powerful tool to investigate electronic and geometric structures around a specific element of interest in a sample [1]. One of the advantages of the XAFS spectroscopy is that the amount of chemical species in a sample can be quantitatively and separately determined owing to the characteristic spectral features depending on chemical species. Another advantage is that XAFS can be applied to any sample without restriction of crystallinity and phase (solid, liquid and gas). The soft X-ray region (1000 ~ 4000 eV) includes Na, Mg, Al, Si, P, S, Cl K-edges and Zn to Sb L-edges. These elements are included in many materials of interest. Recently, lithium ion battery (LIB) has been widely studied for automobile application such as electric vehicles [2,3]. These elements are also included in LIB as a main constituent. Therefore, it increases the demand of XAFS measurement.

In the SR center, the soft x-ray double crystal monochromator beamline, BL-10 is in full use. To satisfy further demands of users for a beamline covering the soft x-ray region, we have designed and constructed a new soft x-ray double crystal monochromator beamline, BL-13 last year. In this year, we have performed several performance tests of BL-13. The obtained results indicated that XAFS spectra with higher energy resolution and higher intensity of incident X-rays can be obtained at BL-13 by introducing quasi-parallel beam for monochromatizing crystals and small incident angle to mirror

2. Design concept

BL-13 has been designed under the following three concepts,

1) All of the beamline and XAFS instrument should be set in-between 2.2 m and 8 m from the source point.
2) BL-13 should have better performance than the existing beamline, BL-10.
3) The available energy range should cover up to 5000 eV.

3. Specification of the beamline

The layout of the beamline is shown in Figure 1 and the specifications of two toroidal mirrors are listed in Table 1. A water cooled 4 quadrant slits and Be filter is located at 2.4 m from the source point. The first Ni coated Si toroidal mirror, whose effective size is 500 mm^L x 40 mm^W, is located at 3 m and deflects the SR beam downward by 1.2° . This mirror can accept an SR beam of as large as 10 mrad^H x 2.5 mm^V. The deflected beam shape and intensity is monitored at 3.5 m from the source point. The quasi-parallel beam is introduced to Golovchenko-type double crystal monochromator (TOYAMA Co. Ltd.). We employed KTP(011), InSb(111), Ge(111), Si(111) and Si(220) as monochromatizing crystal pairs in order to cover the wide energy range. The monochromatized beam is deflected upward by 1.2° and focused by the second Ni coated SiO_2 toroidal mirrors, whose effective size is just same as the first one. The monochromatized beam is monitored at 5.8 m and focused at the sample position apart by 6.5 m from the source point. The XAFS spectra can be obtained in two different modes simultaneously; the partial fluorescence yield (PFY) mode with a silicon drift detector and the total electron yield (TEY) mode (sample drain current mode).

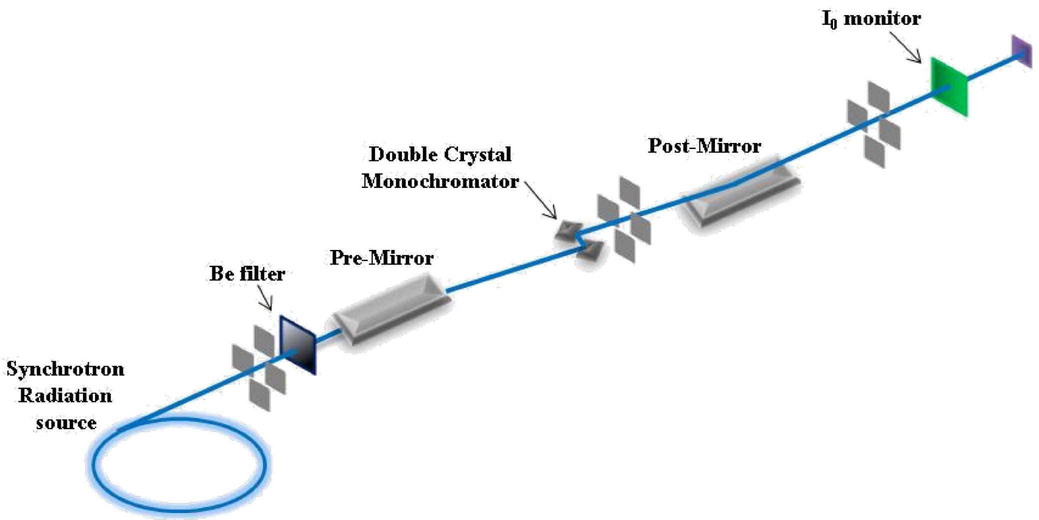


Fig.1 The layout of the soft X-ray double crystal beamline, BL-13

Table 1 Specifications of the two toroidal mirrors

	Pre-focusing mirror	Post-focusing mirror
Mirror size	500 mm^L x 50 mm^W x 30 mm^D	500 mm^L x 50 mm^W x 30 mm^D
Effective area	500 mm^L x 40 mm^W	500 mm^L x 40 mm^W
Tangential radius	557012 mm	288101 mm
Sagittal radius	62.15 mm	32.09 mm
Material	Si	SiO_2
Distance from the source point	3000 mm	5300 mm
Focal point	∞	6800 mm

4. Optics of the beamline

For the crystal diffraction, it is an ideal condition to use a parallel incident beam, since any converged and diverged beam causes a low energy resolution. To obtain high energy resolution, we have installed two toroidal mirrors; one for producing a quasi-parallel beam and another for focusing the beam at the sample position. Figure 2 shows the phase space diagrams of three kinds of optics; parabolic mirror, 1:1 focusing toroidal mirror and 1:∞ quasi-parabolic toroidal mirror. Here, we simulated by the program, SHADOW [4] and the SR beam divergence is $8mrad^H$ x $2mrad^V$. In the phase space, x and x' denote position and dispersion in the horizontal direction, respectively, while z and z' denote them in the vertical direction. The degree of parallelism of the 1: ∞ toroidal mirror is much more improved than the 1:1 toroidal one. It also found that degree of parallelism of 1: ∞ toroidal mirror is same as that of parabolic mirror apparently.

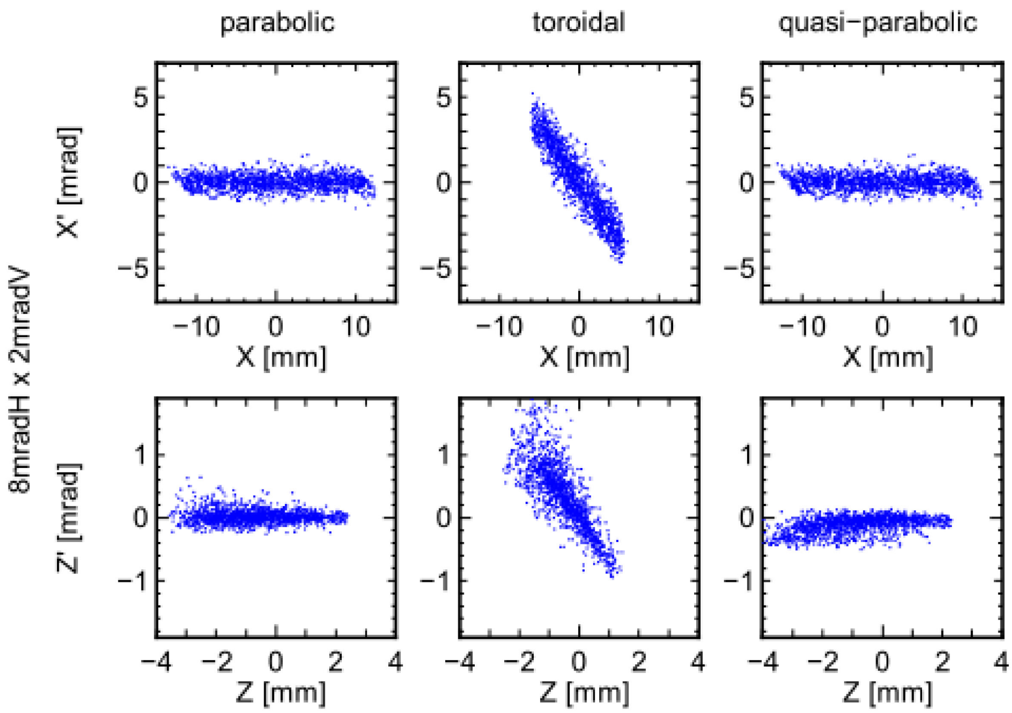
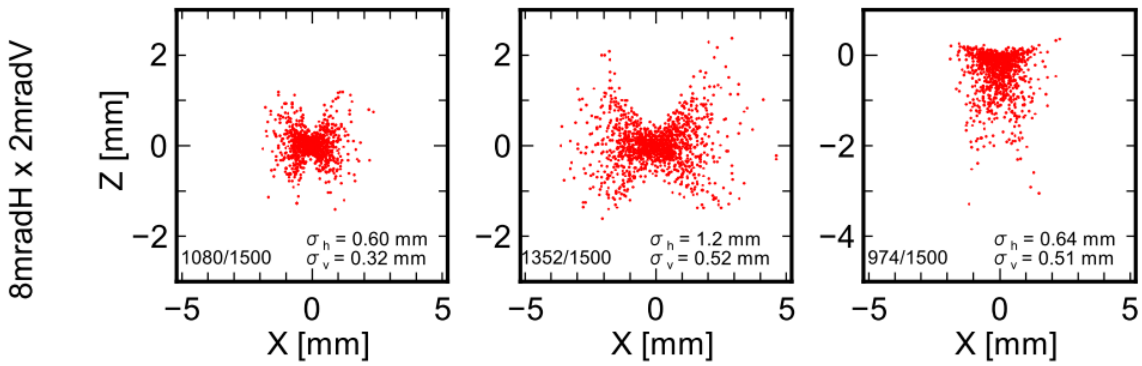


Fig.2 Phase space diagrams of the SR beam at monochromator position for parabolic mirror, 1:1 toroidal mirror and quasi-parabolic toroidal mirror from left to right. The SR beam divergence is $8mrad^H$ x $2mrad^V$.

Another important parameter is the beam size at the focal point. Figure 3 (a) shows beam profile simulated by SHADOW. The set of the quasi-parabolic mirrors gives almost same beam width comparable with that of parabolic mirrors. Figure 3 (b) shows the intensity distribution in horizontal direction of a beam at the sample position. Here, we obtained this spectrum by scanning ϕ0.1 mm Au wire and incident X-ray energy is 2000 eV. We used gauss function to fit this spectrum and estimated $2\sigma = 1.20$ mm. This value is almost same as simulated data. Above results indicate that this optical system gives high energy resolution without using parabolic mirror and small beam size at the sample position.

(a)



(b)

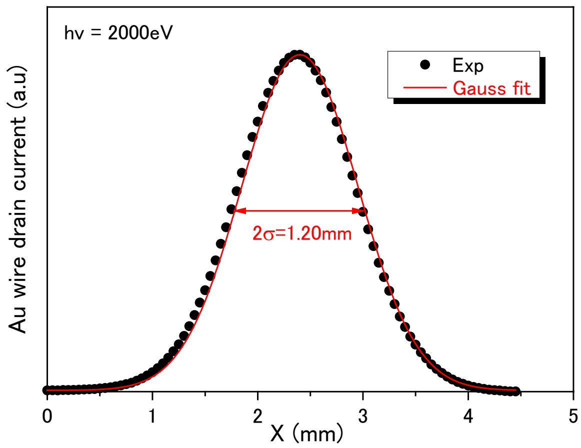


Fig.3 (a)Simulated X-ray beam profiles at the focal point for parabolic mirror, 1:1 toroidal mirror and quasi-parabolic toroidal mirror from left to right. The SR beam divergence is $8\mathrm{mrad}^{H} \times 2\mathrm{mrad}^{V}$. (b) The intensity distribution in horizontal direction of a beam at the sample position. Red curve (thick) indicates the best-fitted spectrum.

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5. Performance comparison with BL-10

Figure 4 (a) - (d) show Mg K-edge spectra of MgO, Si K-edge of Si(111) wafer, S K-edge of K_2SO_4, Ca K-edge of $Ca(OH)_2$, respectively. Each spectra were obtained by using KTP(011) (Figure 3(a)), InSb(111) (Figure 3(b)(c)), Si(220) (Figure 3(d)) as monochromatizing crystal pairs, respectively. All spectra were taken with TEY mode and obtained spectra (red lines) are compared with those taken in BL-10 (black lines). The main peaks observed at BL-13 are sharper than that taken in BL-10 for all spectra. These results demonstrate that XAFS spectra with higher energy resolution can be obtained at BL-13, possibly because we introduced quasi- parallel beam optics for monochromatizing crystals.

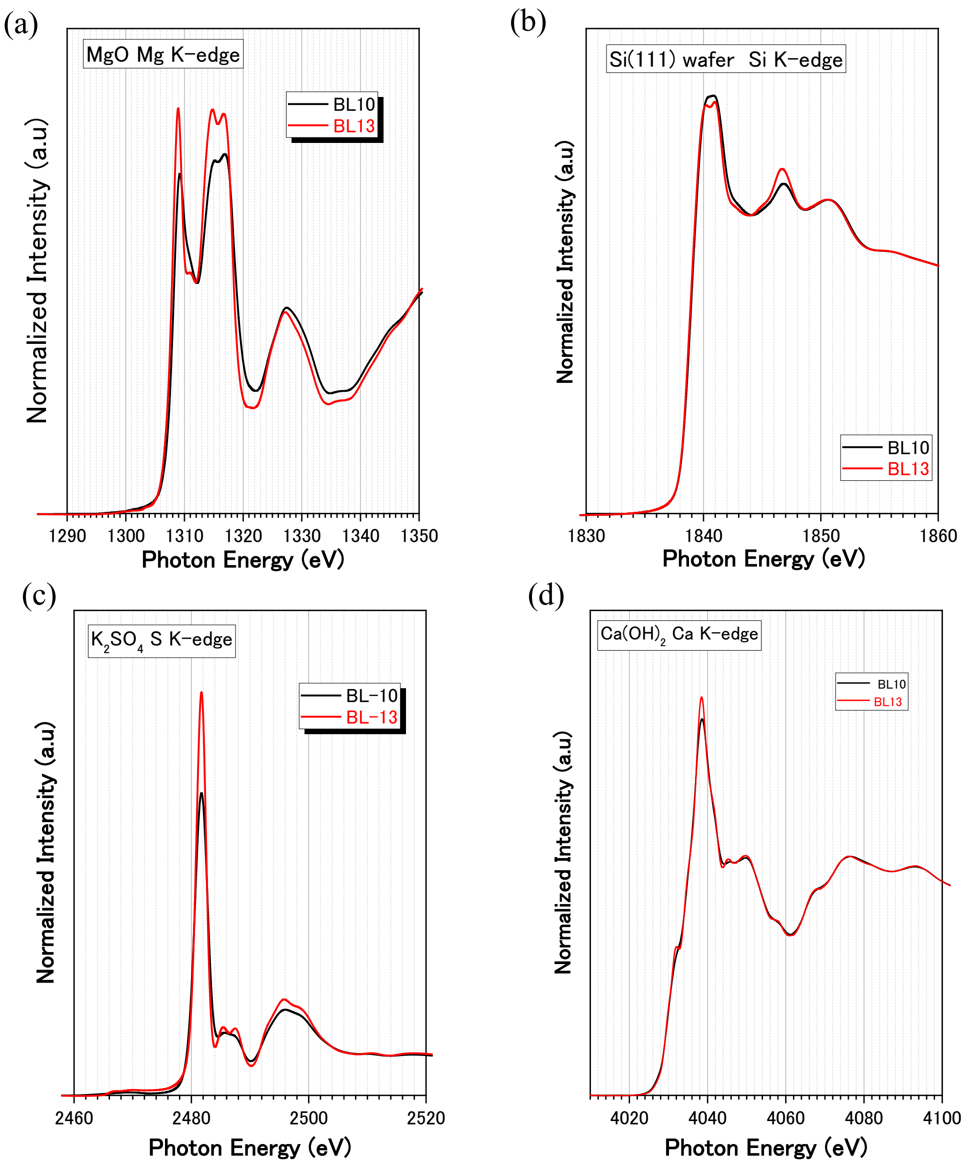

Fig.4 (a) Mg K-edge XAFS spectra of MgO (b) Si K-edge XAFS spectra of Si(111) wafer, (c) S K-edge XAFS spectra of K_2SO_4, (d) Ca K-edge XAFS spectra of $Ca(OH)_2$. All spectra were taken with TEY mode and obtained spectra (red lines) are compared with those taken in BL-10 (black lines).

We also confirmed that BL-13 can be used to measure XAFS spectra in the higher energy region (around 5000 eV). In higher energy region, the reflectivity of Ni coated toroidal mirror in BL-13 is higher than that in BL-10, since the incident angle to toroidal mirror at BL-13 is smaller than that at BL-10. Therefore we can expect to obtain high intensity in this energy region. Figure 5 shows Ti K-edge spectrum of anatase TiO_2 powder with TEY mode. Si(220) were used as monochromatizing crystal pairs. Figure 5 indicates that it is possible to measure the XAFS spectra at BL-13 in higher energy region, while it can't measure at BL-10 because intensity of incident beam is too small.

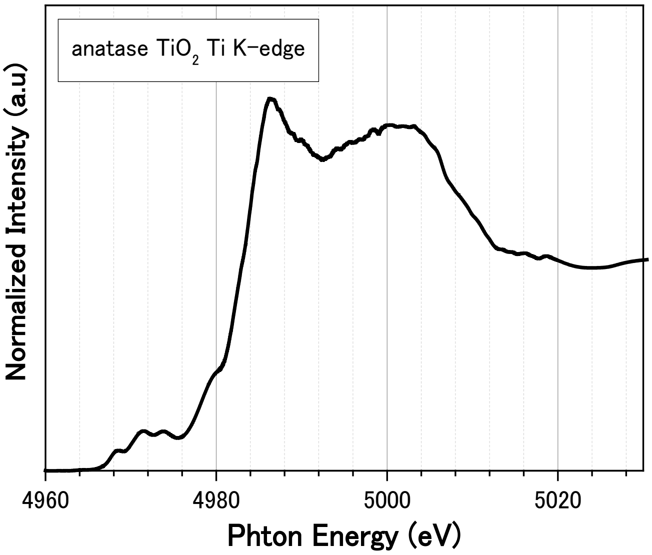

Fig.5 Ti K-edge spectrum of anatase-TiO_2 taken at BL-13 with TEY mode.

Finally, we checked intensity of incident x-ray beam. Figure 6 shows photon energy distributions of sample drain current from In plate. We used InSb(111) as monochromatizing crystal pairs and obtained distribution are compared with that taken in BL-10. It is surprising that the sample drain current at BL-13 is larger than that at BL-10 despite the reflectivity of mirror by BL-13 is smaller than that by BL-10. This is because the accepting angle of the first mirror in BL-13 is larger than that in BL-10. Surface contamination of the mirror of BL-10 is also considered as another reason.

6. Conclusions

We have designed and constructed a new soft X-ray double crystal monochromator beamline at BL-13 last year. By using two toroidal mirrors, we can produce a nearly parallel beam and obtain higher energy resolution by using perfect crystals. We can obtain the XAFS spectra around 5000 eV by small incident angle to mirrors. We also have obtained higher

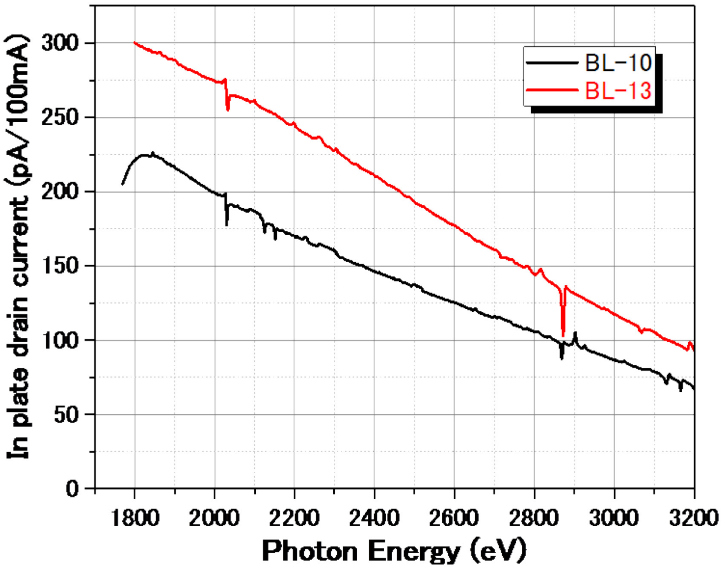

Fig.6 Photon energy distribution of sample drain current from In plate. InSb(111) is used as monochromatizing crystal pairs. Obtained intensity is normalized by SR ring current (100mA).

intensity at sample position by setting the first mirror at 3 m from the source point. Furthermore, thanks to the parallel beam, we can make the whole beamline as short as 7 m.

BL-13 has been open to users since last September.

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