Mg and Al K-edge XAFS measurements with a KTP crystal monochromator

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There has been a strong demand for monochromator crystals with a large lattice spacing in•order to measure EXAFS spectra at the Mg and Al K-edges. We have introduced a pair of KTP crystal at BL1A of the UVSOR facility and examined its performance in the energy range between 1200 and 2000eV. This crystal monochromator can supply stable photon beams enough to measure the EXAFS spectra for the Mg and Al K-edges. Advantages of the KTP crystal are discussed in comparison with a YB_{66} crystal monochromator.

Keywords: KTP; crystal monochromator; EXAFS; Mg and Al K-edges.

1. Introduction

There has been a strong demand for monochromator crystals with a larger lattice spacing than the Si, Ge and InSb crystals widely used in the soft X-ray region, especially in order to get a wide energy range covering the Mg (1305eV) and Al (1560eV) K-edges and their EXAFS (extended X-ray absorption fine structure) region. As crystal monochromators to get near-edge spectra at the Mg and Al K-edges, beryl (10-10) [2d=15.965Å] and quartz (10-10) [2d=8.512Å] have been used, respectively; however, the energy range (*k* range) is limited for EXAFS study because beryl and quartz contain Al and Si, respectively. Furthermore, serious degradation in reflectivity and rocking curve width is often induced as radiation damage by intense non-monochromatized beams, especially in high-energy synchrotron radiation facilities.

In recent years, a YB_{66} (400) [2d=11.72Å] crystal monochromator has been introduced to measure EXAFS for the Mg, Al and Si K-edges (Rowen et al., 1993; Wong et al., 1994; Kinoshita et al., 1998; Smith et al., 1998). There is no absorption edge originating from the elements contained in the YB_{66} crystal from 1200 eV up to the Y L_{III} edge of 2080 eV; therefore, the YB_{66} (400) crystal is very attractive for the Mg and Al EXAFS measurements. However, unfortunately, this crystal has a serious problem; two positive glitches at 1386 and 1438eV are caused by sharp increase in reflectivity of the (600) reflection at the Y $L_{II,III}$ edges (Tanaka et al., 1997). To solve such a problem, a cut-off mirror is employed (Tanaka et al., 1997; Smith et al., 1998), and proves to work effectively for EXAFS measurement; however, it is still hard to completely exclude these glitches.

At the last XAFS conference held in Chicago (XAFS-X, 1998), Rogalev et al. reported that the KTP (011), $KTiOPO_4$, [2d=10.95Å] crystal can be used in the crystal monochromator at the ESRF ID12A circularly polarized undulator beamline. They showed its good performance at the Cl K-edge (2820eV). The KTP (011) crystal has a little shorter lattice spacing than YB_{66} (400), but it covers the Mg and Al K-edges, and has no absorption edge from 1200 eV up to the P K-edge of 2145eV.

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In the present work, we have introduced the KTP (Crystal Laser Co., France) crystal at the soft X-ray double crystal beamline BL1A of the UVSOR facility (Hiraya et al., 1992) and examined its performance and applicability to the EXAFS measurement for the Al and Mg K-edges. Our storage ring is being operated at a rather low energy (750MeV), and the BL1A utilizes radiation from a bending magnet, whose critical energy is 424 eV. The radiation damage proves to be less serious for beryl and quartz crystals; therefore, we can also expect a small radiation damage to the KTP crystal.

2. Performance of KTP crystal

Figure 1 shows throughput spectra of the KTP (011) double crystal monochromator. The solid line indicates the photon flux measured by the Si photodiode (IRD Co.). The Si K-edge jump at around 1840 eV originates from the photodiode itself. The total electron yield spectrum of Au plate (broken line) is also shown as a reference. It should be noticed that there is no structure up to the P K-edge of 2140eV. This is one of some advantages of KTP in comparison with YB_{66}. The averaged photon flux in the 1200-2000 eV range is roughly estimated about $5x10^8$ photons/sec/100mA in the UVSOR bending magnet beamline. We already have examined performance of the YB_{66} crystal; the throughput of the present KTP double crystal monochromator is at least ten times larger than that of YB_{66}. This is the second advantage of KTP. The energy spread (FWHM) of the monochromatized beam is estimated to be about 0.5~0.8 eV by using rocking curves in the 1200-2100 eV range, and is comparable to that of YB_{66} (Rowen et al., 1993). We have to mention radiation damage of the KTP crystal. At the ESRF ID12A, the damage was not noticeable (Rogalev et al., 1998): on the other hand, the present first crystal showed the damage to a certain extent at the BL1A, similarly to the case of beryl and quartz crystals. We cannot discuss the difference in radiation damage, because the radiation damage depends on character of the crystals, e.g., impurities, surface roughness, absorbance of the incident light, and

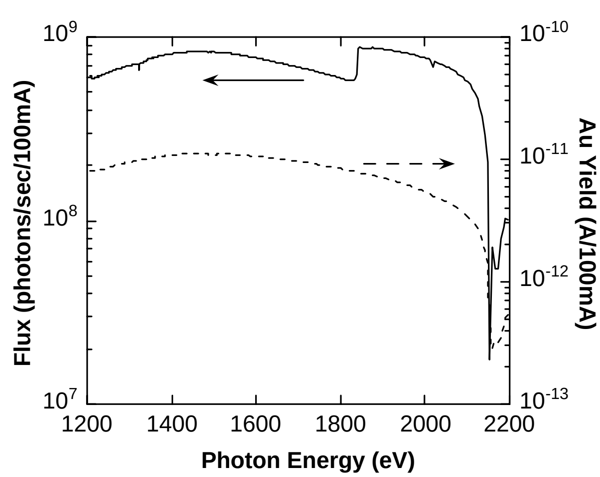

Figure 1
Throughput spectra of a KTP (011) crystal monochromator at BL1A of the UVSOR facility. The solid line indicates the photon flux measured by a Si photodiode. The sample drain current from a Au plate (broken line) is also shown as a reference.

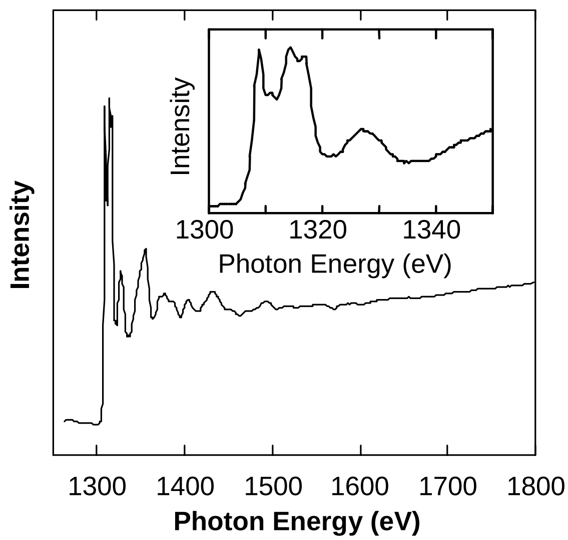

Figure 2
Mg K-edge XAFS spectra of MgO measured by monitoring sample darin current.

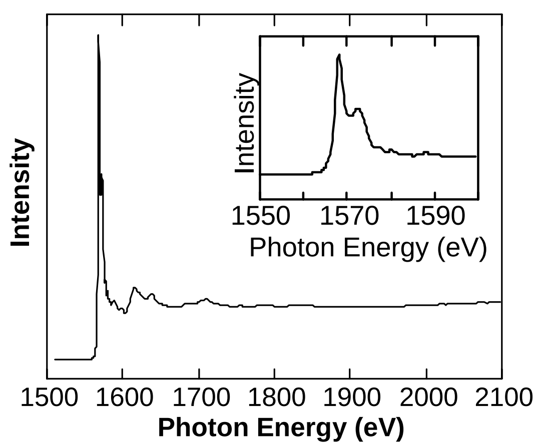

Figure 3
Al K-edge XAFS spectra of α-Al_2O_2 measured by monitoring sample drain current.

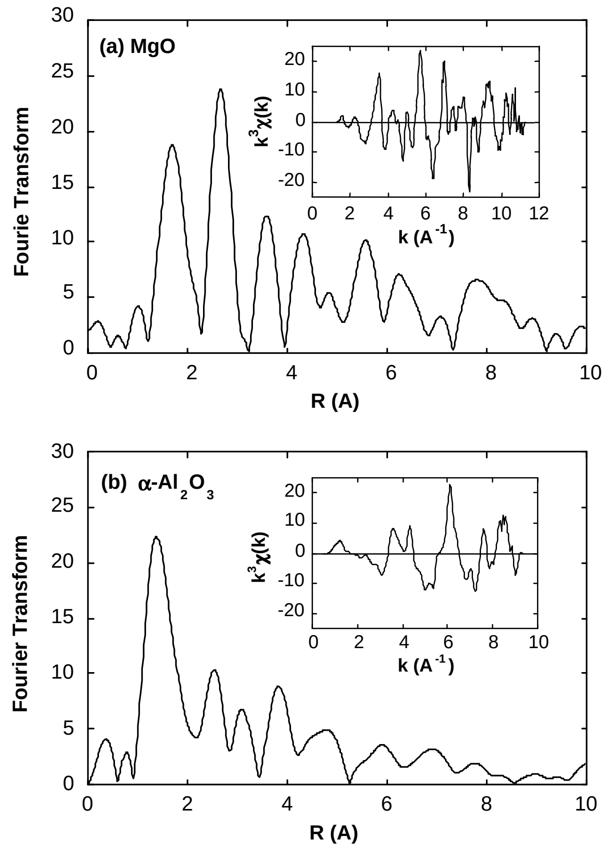

Figure 4
EXAFS oscillations $k^3\chi(k)$ of MgO and α-Al_2O_2 and their Fourier transforms without any phase-shift correction.

so on. Fortunately, the damage was not so serious and we could measure EXAFS spectra as shown in the following section.

3. Mg and Al K-edge XAFS spectra

Figures 2 and 3 show the Mg and Al K-edge XAFS (XANES and EXAFS) spectra of MgO and α-Al_2O_3, respectively, which were measured by monitoring the sample drain current. Both the XANES spectral shape is identical to the corresponding spectra measured with YB_{66} because the energy resolution is comparable to each other. In the EXAFS spectra shown in Figs. 2 and 3, the energy range is considerably extended in comparison with the spectra measured with the beryl and quartz monochromator. In the latter case, Mg and Al K-edge EXAFS are limited up to 1560 eV at the Al K-edge and 1840 eV at the Si K-edge. Figure 4 shows k^3-weighted EXAFS oscillations and their Fourier transforms without any phase-shift correction. Compared with the phase-corrected results of YB_{66} (Wong et al., 1994), the distance shown in the Fourier transform is different from the present one, but the radial distribution is quite similar even for higher shells.

4. Summary

We have introduced a pair of KTP (011) crystal at BL1A of the UVSOR facility and examined its performance in the energy range between 1200 and 2000eV. The KTP crystal monochromator proves to supply stable photon beams and the energy resolution is estimated to be about 0.5-0.8eV. The KTP has two excellent advantages in comparison with YB_{66}. First, there is no absorption structure originating in the elements contained in KTP in the energy range to be measured; a cut-off mirror system is not necessary. Secondly, a higher throughput is available. By use of the KTP monochromator, reliable EXAFS spectra for the Mg and Al K-edges are observed for a wide *k*-range.

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