Preliminary wavelength-dispersive X-ray fluorescence experiments with 75.5 keV X-rays

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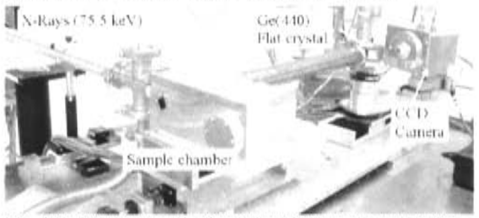
One of the biggest advantages of using a brilliant X-ray source in the high-energy region is the capability of exciting K electrons for heavy elements [1,2]. Although X-ray fluorescence (XRF) analysis with an energy-dispersive Ge detector is powerful [2], another interesting way is to perform further higher-resolution experiments to explore chemical environments and electronic structures, in particular 4f electrons for lanthanides.

The primary X-ray photons used are 10^{th} harmonics of an undulator beam, and the energy is 75.5 keV. As shown in Fig.1, the instrument is an ordinary X-ray spectrometer, which combines a flat Ge crystal (50×25×2(t) mm, for most cases, (440) or (660) symmetric reflections are used) and a Peltier cooled CCD camera (576×384 pixels, pixel size 22μm×22μm, equipped with fiber optics and a Gd_2O_2S:Tb scintillator, Princeton). A CdZnTe detector, placed at the CCD camera position, was employed to adjust the crystal. Once X-ray fluorescence peaks from the sample were found, the camera system was set there again to start the exposure. Typical exposure time was 30 min~2h.

Figure 2 shows an example of XRF spectra obtained by a CCD camera. The sample measured is cerium oxide (pellet of 10 mm dia., 5 mm thickness). One can see the energy resolution is ~85 eV at Ce $K\alpha_1$ (34,719.7 keV). Although this is much better than a normal Ge detector, it is still far from our goal (~40 eV) and is not sufficient to discuss any chemical effects. Another problem is the possible imperfection of the shielding against the high-energy X-ray background. The electronic noise of the CCD camera also degrades the spectra, because fairly lengthy exposure is needed at the moment. Further improvements to the instrumentation are now under way.

References
[1] M.Harada and K.Sakurai, Spectrochim. Acta, **B54**, 29 (1999)
[2] I.Nakai, Y.Terada, M.Itou and Y.Sakurai, J. Synchrotron Rad. **8**, 1078 (2001).



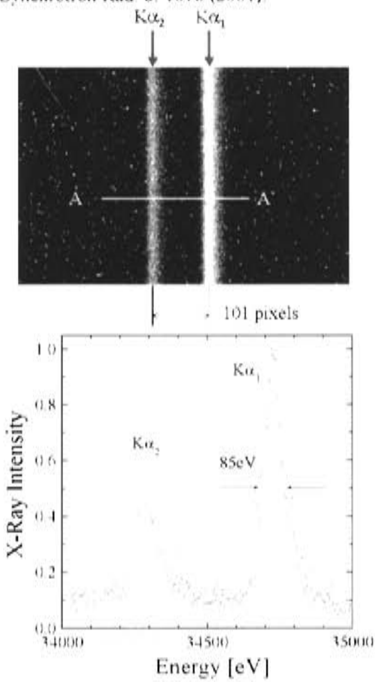


Fig. 1 (left) Wavelength-dispersive XRF spectrometer for high-energy X-ray region. The distance between the sample and the Ge crystal is ca. 730mm. A CCD detector is placed ca. 200mm from the Ge crystal. Beam size: 0.3mm(H) × 1mm(V).
Fig. 2 (right) Ce Kα spectra obtained by a CCD camera. A raw image (top) and the intensity profile along A-A' (bottom)

X-ray luminescence holography using ZnO epitaxial thin film

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X-ray absorption fine structure (XAFS) studies using X-ray excited optical luminescence (XEOL) have been conducted by some researchers. The XEOL can be used also for atom-resolved X-ray holography. In this study, changes of the XEOL intensities for ZnO were measured with varying the sample orientation.

The measured sample was ZnO/MgO/c-Al_2O_3 epitaxial thin film (520nm). Incident energy was 15.0 keV. The data was collected in the so-called inverse mode. The XEOL of ZnO was detected by a photon counter (H7155-20). In addition to this, the Zn Kα (8.64keV) X-ray fluorescence hologram was measured. Countrates of the XEOL and the X-ray fluorescence were about two and one millions cps respectively. These intensities were measured as a function of azimuthal angle ϕ and polar angle θ within the ranges of $0^\circ \le \phi \le 360^\circ$ and $0^\circ \le \theta \le 70^\circ$. The exit angles of the XEOL and the X-ray fluorescence were fixed at 45°.

Figures 1 (a) and (b) show the holograms by the fluorescence and XEOL, respectively. In both patterns, X-ray standing wave (XSW) lines are observed clearly. However, the XSW lines patterns for both the holograms are different. Strong XSW lines are indicated by break lines in Fig. 1. From lattice spacings (1.625Å, 1.412Å) calculated from both the strong XSW lines, it was found that break lines in Fig. 1 (a) and (b) correspond the reflection index of 2-1-10 of ZnO film and 30-30 reflection of Al_2O_3 substrate, respectively. The 2-1-10 direction of ZnO film was parallel to the 30-30 direction of Al_2O_3 substrate in the present sample. This result indicates that the luminescence arose from the ZnO layer close to the substrate.

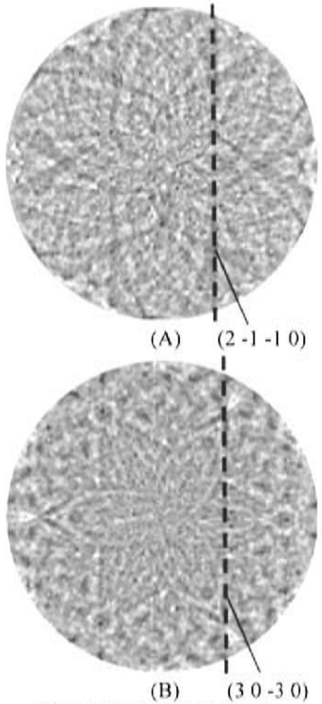

Fig.1 Holograms at 15keV
(A):XFH,(B):XLH.