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# Development of Double-Crystal DXAFS Instrument

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## Introduction

The time-resolved X-ray absorption spectroscopy is a powerful technique to determine the local structure and the electronic state of a target element during fast reaction processes. The dispersive XAFS (DXAFS) method is the only technique to apply to single-action processes, which are difficult to repeat many times. The detector-limited time-resolution of the DXAFS technique is overcome by the utilizations of the XSTRIP detector [1] and the pulse characteristics of PF-AR, and it has been achieved to perform the time-resolved applications in sub-nano second time region. On the other hand, the quality of XAFS spectrum obtained by the DXAFS instrument is largely improved by the removal of X-ray scatterings which is generated by air in the X-ray path, the polychromator crystal, the powdered sample, etc. We have recently developed a new DXAFS instrument with two crystals, the double-crystal DXAFS instrument, to remove such scatterings. In this report, the details and the performance are summarized for the new double-crystal DXAFS instrument.

## Development

The double-crystal DXAFS instrument is mainly composed of a slit for the incident white X-ray, a cylindrically bent crystal as the first polychromator, a variable-bent crystal as the second polychromator, and a linear detector to measure the dispersed X-ray intensities, as shown in Fig. 1.

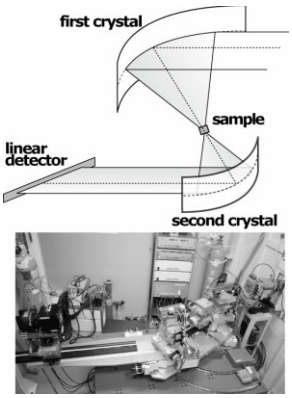


Fig. 1. Concept of the double-crystal DXAFS instrument and its photograph developed at NW2A. The white X-ray is irradiated to the first bent crystal to produce the dispersed X-rays. A sample is placed at a focal point of the dispersed X-rays, and the transmitted X-rays are monochromatized by the second bent crystal. The position-sensitive linear detector records the X-ray intensities to obtain the XAFS spectrum.

To prevent the thermal instability of the first crystal, we use a crystal holder, in which the thermostat water is circulated, with the fixed radius for the cylindrical bent. The In–Ga liquid metal is used to achieve the thermal contact between the crystal and the holder. The second crystal is bent using the four-point-supported bending mechanism [2] to adjust flexibly the incident angle of the dispersed X-rays to the crystal surface.

## Results and Discussion

Fig. 2 shows the intensity profile of a slit blade measured with the single-crystal and the newly developed double-crystal DXAFS instruments for some powdered samples. Many powdered samples produce the small

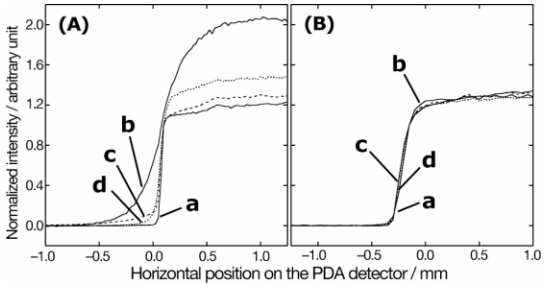


Fig. 2. Normalized intensity profile of a slit blade measured with the normal single-crystal (A) and double-crystal (B) DXAFS instrument. The sample is none (a), $Al_2O_3$ (b), MgO (c), and BN (d).

angle scatterings, leading to the tailed intensity profile as given in Fig. 2(A). Such scatterings cannot satisfy the diffraction condition at the second crystal, and they are perfectly removed as shown in Fig. 2(B) in the case of the double-crystal DXAFS instrument.

The removals of such excrescent X-rays improve the quality of the XAFS spectrum as shown in Fig. 3, in which the K edge absorption spectra of Cu foil are compared. The XAFS spectrum obtained with the double-crystal DXAFS instrument is perfectly in

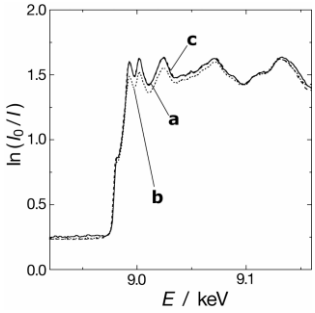


Fig. 3. The XAFS spectra of Cu foil measured with the double-crystal DXAFS instrument (a), the single-crystal DXAFS instrument (b), and the conventional step-scanning system (c). The crystal surface is Si(111).

agreement with that measured at the conventional step-scanning beamline. A blunt feature observed just after the absorption edge in the case of the single-crystal instrument is considered to be due to the scatterings reaching to the linear detector.

## References

[1] Y. Niwa *et al.*, *PF Act. Rep.*, **23**, 253 (2006).
[2] P. G Allen, S. D. Conradson, *J. Appl. Cryst.*, **26**, 172 (1993).

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