Development of highly stable Bragg polychromator for energy dispersive XAFS

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Abstract. A highly stable Bragg polychromator for energy dispersive XAFS (DXAFS) systems was developed at the bending magnet beamline BL28B2 at SPring-8. The bending device is a four–point-type bender. Additional stoppers for the crystal are attached at both ends of the bender to maintain the position and curvature of the crystal in the bender. The crystal was indirectly cooled in a copper grain bath with good thermal contact, the temperature of which was precisely controlled using water cooling. A performance test of stability of the new Bragg polychromator showed that the energy drift in the DXAFS spectra was reduced to less than 0.01 eV for 2 hrs.

1. Introduction
In energy dispersive XAFS (DXAFS) measurements, the temporal stability of a polychromator is an important factor for measuring high-quality spectra. Recently, Mairs et al. developed the Bragg polychromator which have the high stability of the crystal bending shape and the high repeatability of the adjustment of the focal spot [1]. At the DXAFS station at BL28B2, SPring-8, a Bragg-type polychromator was developed and has been used in the energy region from 6 to 12 keV [2]. It consists of a crystal-bending device to obtain a variable curvature but has no cooling devices. However, an energy shift and/or fluctuation in the DXAFS spectra were observed during long-term measurements due to mechanical and thermal instability of the bent crystal in the polychromator. We therefore newly developed a Bragg-type polychromator having a highly stable bending and cooling mechanism.

2. Design of the Bragg polychromator
The newly developed Bragg polychromator consists of a four-point bender as a bending mechanism and a cooling bath for a crystal (Fig. 1), which are housed in a helium gas flow chamber under atmospheric pressure. The crystal is a symmetrically cut Si (111) with dimensions of 200(w) x 40(h) x 1(t) mm. The bender has an adjusting mechanism to correct the twist of the crystal. We first developed a 4 point bender made of 4 cylinders, but curvature of the crystal could not be maintained due to the slip of the crystal in the bender. To solve this problem, we adopted the design of the bender made of 2 cylinders and 2 plane surfaces with stoppers. Two stoppers for the crystal are installed at both ends of...
the bender; one is movable and the other is fixed. The position of the movable stopper is adjusted to fix the end of the crystal by hand after adjusting the bending of the crystal.

The crystal was indirectly cooled with a newly developed bath. A liquid InGa eutectic alloy is often used as a heating medium of indirect-cooling bending devices due to its excellent thermal conductivity [3, 4]. This alloy, however, is highly corrosive and difficult to be removed once adhered. If the liquid InGa eutectic alloy spills out from the bath, it will inflict damage on the surrounding surface. We used copper grains with an average grain diameter of 0.5 mm (Kojundo Chemical Laboratory Co.) as a substitute for the liquid InGa eutectic alloy, which are much easier to handle and achieve good thermal contact between the crystal and the water-cooling bath. The cooling water is circulated in flow channel in the bath (Fig. 1(c)). The lower part of the crystal (ca. 5 mm wide) was immersed in the copper grain bath. The crystal temperature could be controlled between 26 and 30°C within an accuracy of 0.01°C by using water circulated in the bath.

3. Performance of the Bragg polychromator

3.1. Long-term mechanical stability of the crystal bending shape

Figure 2 shows the effectiveness of the stoppers for the bent crystal of the polychromator bender on the long-term stability of the focused beam profiles. In Fig. 2(b), the focused beam size in the horizontal direction of 22 μm (FWHM) is achieved with energy bandwidth of 450 eV at 8 keV. The focused beam profile with no stoppers significantly expanded with 24-hour continuous bending even under no heat load by X-ray irradiation on the crystal (Fig. 2(a)). This indicates that the curvature of the bent crystal gradually decreased due to the slip of the crystal in the bender. The bent shape of the crystal was significantly stabilized by installing stoppers on the polychromator bender (Fig. 2(b)).
3.2. Stability of the crystal temperature

Figure 3 shows the effects of crystal cooling on the stability of the crystal temperature during X-rays irradiation. The temperature of the crystal was measured using thermistor elements attached to the back surface of the crystal. The temperature of the crystal without cooling (without the copper grains in the cooling bath) increased to 31.05°C after 20 min then gradually increased until 31.1°C (dotted line). In contrast, by cooling the crystal, the crystal temperature reached a steady state at 29.04°C within 20 min and its fluctuation was suppressed within ± 0.01°C.

3.3. Stability of the absorption edge position in DXAFS spectra

Figure 4 shows the fluctuation of the Ni K-edge position in the DXAFS spectra of the Ni foil during X-ray irradiation. Without cooling, the Ni K-edge position gradually shifted toward high energy after
20 min. With cooling, the DXAFS spectral shape and curvature of the crystal reached a steady state within 20 min, which is the same tendency observed in the stability of the crystal temperature. As a result, the energy fluctuation in the DXAFS spectra decreased to 0.007 eV (FWHM).

![Figure 4. Ni K-edge energy of Ni foil in DXAFS spectra with (black) and without cooling of crystal during X-rays irradiation (gray)](image)

4. Conclusion and further improvement plan

We developed a highly stable Bragg polychromator for DXAFS measurements at the bending magnet beamline BL28B2 at SPring-8. Long-term stability of the curvature of the crystal and reduction in the energy drift below 0.01 eV were achieved by introducing newly developed equipment, such as a stopper for the crystal and cooling device with copper grain bath.

The DXAFS station at BL28B2 has a beam shutter upstream of the polychromator. The polychromator is irradiated intermittently with intense white X-rays for each measurement. Thus, it is quite important to cut the time needed for stabilizing the X-ray beams incident on a sample for efficient use of the beam time. We plan to develop a high-speed stabilizing system for reaching steady state within a few minutes regarding crystal temperature. We also plan to develop a remote control device to adjust the stopper position without opening the helium gas flow chamber.

References

[1] T. R. Mairs J. Borrel and O. Mathon Diamond Light Source Proceedings 1 MEDSI-6 2011 1-4
[2] K. Kato, T. Uruga, H. Tanida, S. Yokota, K. Okumura, Y. Imai, T. Irie, Y. Yamakata, AIP Conf. Proc. 879 2007 1214-1217
[3] A. San-Miguel M. Hagelstein J. Borrel G. Marot M. Renier J. Synchrotron Radiattion 5 1998 1396-1397
[4] F. Baudelet Q. Kong L. Nataf J.D. Cafun, A. Monza S.Chagnot and J.P. Itié High Pressure Research 31 2011 136-139