Evaluation of Window Material for Operando Double-Edge DXAFS Experiments

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The wavelength-dispersive XAFS (DXAFS) technique is a powerful tool for in-situ and operando experiments because of its ability to the real time observation. We have recently developed a double-edge DXAFS system, which consists of two polychromators and a wide photodiode array detector [1]. It is possible to detect simultaneously the chemical state of two different elements, such as Fe and V, using the double-edge DXAFS system, and such measurements are important for the development of a next-generation secondary battery.

The design of the electrochemical cell is significant for operando experiments for the stable operation of the electrochemical procedures. The electrochemical cell is usually fixed by some window plates to keep the thickness. Be plate and Kapton film are useful window materials for the X-ray experiments. The X-ray transmittance and the physical strength are the requirements as the window material In addition, it is important that the small-angle scattering is negligible [2] for the DXAFS experiments, which deforms the absorption spectrum. In this study, the effect of Be and polyimide window upon the XANES spectra at V and Fe K edges has been evaluated at BL-5. The Si(111) polychromators with the curvature of 1.8 m and 3.0 m were used for the XANES measurement at the V and Fe K edge, respectively.

The X-ray transmittance are shown in Fig. 1 for Be and polyimide with the thickness of 0.5 mm. Figure 2 shows XANES spectra of Fe and V foil measured with and without the window materials. The edge jump of Fe measured with Be was slightly larger than the others. It is caused by Fe contained in Be as an impurity (see Fig. 1(A)). The shape and the energy resolution of XANES spectra were equivalent, therefore the X-ray scattering from these window materials is negligible. The X-ray transmittance of polyimide is considerably low at the V K edge, as shown in Fig. 1, and the S/N ratio of V measured with polyimide was thus obviously lower than the other two. The use of polyimide is not practical for the measurement at the V K edge.

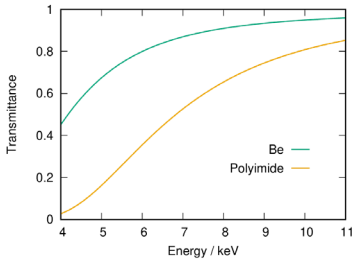

Fig. 1 X-Ray transmittance of Be and polyimide window with the thickness of 0.5 mm.

In conclusion, it has been revealed that the small-angle scattering of Be and polyimide windows is negligible for the double-edge DXAFS experiment. However, the impurity of Be and the low transmittance of polyimide at lower X-ray energies need to consider when choosing the window materials.

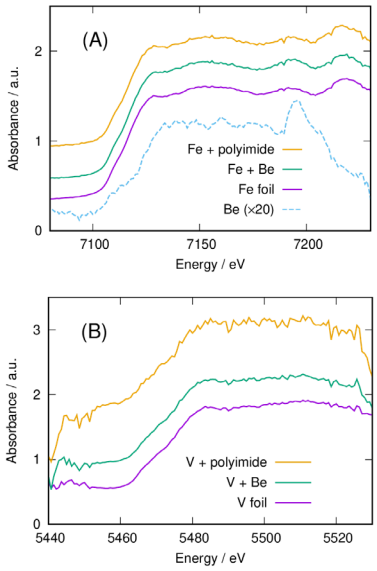

Fig. 2 XANES spectra of Fe foil (A) and V foil (B) with and without Be and polyimide window.

References

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