XAS Measurement for a Small Sample at BL-8

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BL-8 SORIS Beamline has some experimental apparatuses capable of performing x-ray absorption spectroscopy (XAS), photoelectron spectroscopy (PES), medium energy ion scattering (MEIS), molecular beam epitaxy (MBE), reflection high energy electron diffraction (RHEED), and infrared heating, so we can prepare samples in ultrahigh vacuum (UHV) and analyze them *in situ*. Especially for XAS measurement, photon energy range from about 10 eV to 900 eV is available with both total electron yield (TEY) and partial electron yield (PEY) methods. The electron yield is a surface sensitive experimental method; the detection depth of TEY is several nm and PEY is about 1nm. Therefore, we can measure chemical states of materials including carbon, nitrogen and oxygen in the region near the surface. However, carbon and oxygen are also representative contaminations adhering to the surface. Surface contaminations can be removed by sample cleaning such as sputtering methods, but if the beam size is bigger than the sample size, materials in the out regions of samples induce X-ray absorption. In this case, there may be a risk that absorption spectra distort. Therefore, we have introduced a pinhole plate to make beam size smaller and investigated the effect of XAS measurement for small samples. The plate has a pinhole of about 2 mm in diameter. The beam size at sample position without the pinhole was about ~3 mm(H) × 1 mm(V), and the pinhole made it about 1 mm(H) × 1 mm(V). The sample drain current with the pinhole became about one sixth compared with that without the pinhole.

We have investigated a small single-crystal diamond (100) sample with the size of 3 mm × 3 mm, which was produced by high-pressure and high-temperature method. It was put in a perforated Cu holder and fixed with glue including carbon – nitride bonds (in order to polish the diamond surface after the measurements). Figure 1 shows C *K*-edge XAS spectra of the small diamond sample with or without the pinhole. The pinhole makes spectra of diamonds ridge and dip structure [1] within the energy range of about 300 to 305 eV clear. The intensity of a small 286.4eV peak appeared without the pinhole, while it did not with the pinhole. This peak was assigned to C≡N bond [2]. Thus, it is indicated that elements in the out of region of samples made XAS spectrum obscure. These results revealed that XAS measurement become possible to get data clearly for small size sample about 1 mm × 1 mm. We are also able to measure XAS and PES data with choosing a spot in 1 mm^2 units for our samples at BL-8.

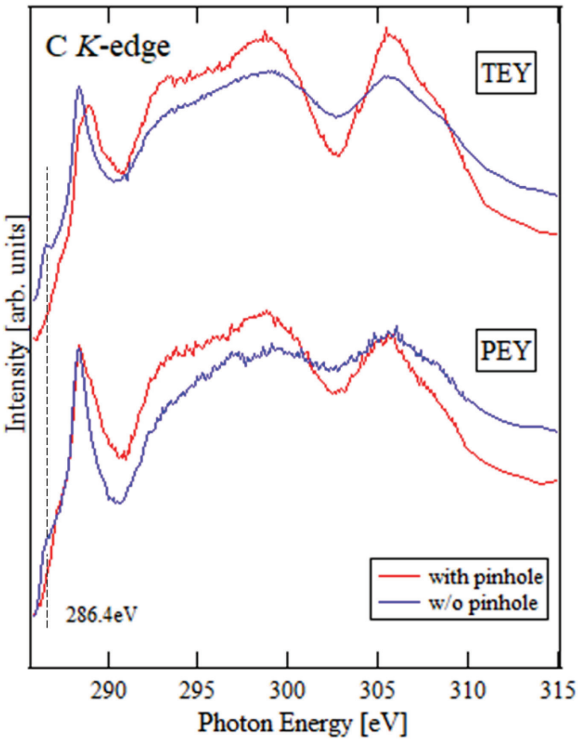

Fig. 1 C *K*-edge XANES spectra of a diamond sample with or without a pinhole measured with the TEY and the PEY methods. Diamond shows a typical structure at the region of about 300 eV to 305 eV. The measurement with a pinhole has no peak at about 286.4 eV

References

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[2] C. Walter, K. Kummer, D. Vyalikh, V. Bruser, and K.D. Weltmanna, J. Elec. Soc. **160**, 1088 (2013).