The application of 19-element Ge solid state detector to polarization-dependent total-reflection fluorescence XAFS measurement

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Introduction

PTRF-XAFS (polarization-dependent total-reflection fluorescence XAFS) is a powerful method to investigate the surface structure of model supported catalysts where metal or metal oxides are dispersed on oxide single crystals. We have carried out PTRF-XAFS analyses on Mo/TiO₂(110), Cu/TiO₂(110) and Ni/TiO₂(110), and found that they have anisotropic structures. Because the amount of surface species is very small, a multi-element solid state detector with good energy resolution and large solid angle is preferable in order to detect such minor species. We have applied a newly developed 19-element Ge solid state detector (Canberra) to PTRF-XAFS measurements.

Experimental

We measured Ni K-edge XAFS spectra to investigate the local structure around Ni atoms dispersed on TiO₂(110) single crystal (20 mm × 20 mm). Ni was deposited by a dropwise impregnation method using a diethylether solution of Ni(DPM)₂ complex. We used a 19-element SSD to detect the fluorescence X-ray from Ni. We also used a digital control system called DXP (XIA, Inc.) to adjust the conditions of detector such as gain, filtering parameters and so on. The DXP has advantages in operation and optimization of the measurement conditions.

Results and discussion

Figure 1 shows EXAFS oscillations for Ni/TiO₂(110) which were measured using (a) 19-element SSD and (b) NaI(Tl) scintillation counter (SC). The quality of the spectra measured by SSD was dramatically improved. The SSD can provide a better spectrum in less time than the SC.

Another advantage in applying the multi-element SSD to PTRF-XAFS measurement is that we can ignore the effects of diffractions from the substrate. In PTRF-XAFS measurement, incident X-rays are sometimes diffracted by the single crystal substrate and come into the detector, which disturb the spectra. We can distinguish the fluorescence X-ray from diffraction lines utilizing a good energy resolution of the SSD. If much larger diffractions which cause a pile-up of the detector come into the detector, we can cancel out the diffractions simply by removing the signals from the elements which suffer from diffractions. Figure 2 shows the signals from each element. Diffractions hit 2 elements, but others did not suffer from the diffractions. Thus we summed up the signals without the effect of diffractions.

![Figure 1 EXAFS oscillations for Ni/TiO₂(110) measured with (a) 19-element SSD and (b) scintillation counter.](image1)

![Figure 2 Output signals from 19-element SSD. (5 among 19 elements)](image2)