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Soft X-Ray Absorption Spectroscopy with Variable Surface Sensitivity using Fluorescence Yield Detection

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Abstract. Surface sensitivity in the soft X-ray absorption spectroscopy by the fluorescent X-ray yield detection under grazing exit condition is discussed. It is experimentally demonstrated at Si *K*-edge that the sampling depth can be controlled by changing exit angles in the vicinity of the critical angle for total reflection.

1. INTRODUCTION

For the XAFS measurements of bulk solid materials in the soft X-ray region, total electron yield (TEY) technique has been mostly employed but fluorescent X-ray yield (FY) detection has recently been proposed as an alternative [1,2], where surface sensitivity is discussed considering the penetration depth of the emitted photons. On the other hand, it was shown in the hard X-ray region that FY is applicable to surface probe if the observation angle is less than the critical angle (θ_c) for total reflection [3]. This report aims to clarify experimentally how the surface sensitivity of FY varies under extreme grazing exit condition in the soft X-ray region.

2. EXPERIMENTAL SET-UP

Figure 1 schematically illustrates the experimental set-up. A silicon wafer with 195 Å thick surface oxide was chosen as a typical sample to evaluate surface sensitivity. Soft X-ray beams monochromatized with a pair of InSb(111) crystals at PF-BL-11B [4] illuminate the sample at nearly normal incidence. Horizontal beam size was set to 3 mm by the incidence slit S_i . Silicon *K* FY was monitored with a gas-flow proportional counter (PC) [5]. The sample was precisely rotatable with a stepping motor around the vertical axis so that the exit angle (θ_E) of the fluorescent X-rays can be controlled. Observation angle spread of PC was set to about 2.2 mrad by the 0.5 mm opening exit slit S_E at a distance of 220 mm from the incident beams. TEY was simultaneously recorded by the drain current from the sample with a picoammeter.

Figure 1: Schematic diagram showing the experimental set-up.

Figure 2: Exit angle dependence of FY intensity.

Although it is hard to know the absolute value of θ_E in the present experimental condition, it was estimated from the variation of FY intensity with rotating the sample. Figure 2 shows the θ_E dependence of the FY intensity, where θ_E is expressed relative to an unknown value of α . By comparing this curve with the calculation ($\theta_c \approx 0.9^\circ$) and previous experiments for a Si wafer [6], α was estimated to be zero.

3. RESULTS AND DISCUSSION

Figure 3 shows the Si K-edge FY XANES spectra at various grazing exit angles. The first two peaks at 1842.5 eV and 1848.5 eV are well known as characteristic features of the bulk silicon and surface oxide, respectively. It is clearly seen that the intensity of the oxide peak does not follow the total FY intensity as the exit angle decreases. This indicates the sampling depth of FY changing with θ_E . In order to compare surface sensitivity between FY and TEY, these spectra were normalized by the edge jump (Fig. 4). It should be mentioned that the pre-edge background was subtracted for only the TEY spectrum. As expected, the surface sensitivity of FY changes drastically as θ_E crosses the critical angle θ_c . It becomes higher than TEY under extreme GE condition ($\theta_E < \theta_c$). In this sense, previous estimation of the information depth in Ref. [2] is insufficient. On the other hand, FY at a larger grazing exit angle is more bulk sensitive than TEY. Thus variable surface sensitivity is realized by this GE-FY detection method.

Extreme GE condition has some advantages as a surface probe compared to other previous methods such as "Total Reflection" (TR) method and Auger Electron Yield (AEY) detection. It is inevitable to correct anomalous absorption effects in TR method and surface sensitivity cannot be adjusted because the kinetic energy as well as the escape depth of the Auger electrons are specified for elements.

Figure 3: Si K-edge FY XANES spectra at GE condition.

Figure 4: Normalized XANES spectra by FY and TEY.

It is well demonstrated that the FY detection can be more and less surface sensitive than TEY by slightly changing the grazing exit angle around the critical angle for total reflection. This controllability of surface sensitivity is quite useful in XAFS study for both the surface and bulk properties as well as depth-profile analysis.

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