BL13XU: X-ray Diffraction Measurement of Reconstructed Surface

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1. Introduction

The undulator beamline BL13XU[1,2] is dedicated to studies of surface/interface structures using surface X-ray diffraction. Precise structure determination of a crystal surface can be done by measuring crystal truncation rod (CTR) scatterings, which originate from termination of the surface. In the beamline practical, we measure CTR scatterings from a reconstructed surface in ultra-high vacuum (UHV) with a 1-ton UHV chamber mounted on a 2+2 type diffractometer (See Fig.1).

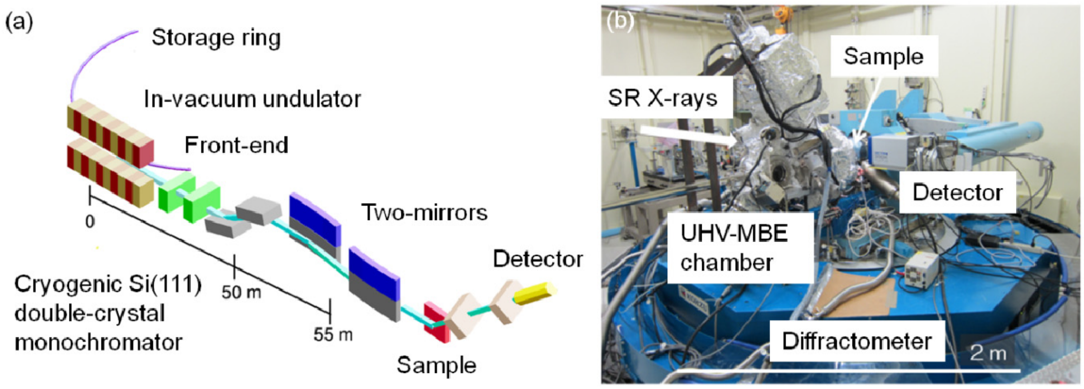

Figure 1. (a) Schematic layout of BL13XU. (b) Surface diffractometer combined with an UHV-MBE chamber.

2. Surface X-ray Diffraction

X-ray diffraction has contributed for decades, as well as electron diffraction, to structure analysis of surfaces and interfaces since brilliant synchrotron sources became available, which overcome a drawback of weak interactions of X-rays with matters and then make X-ray diffraction surface sensitive. A simple kinematical interpretation of X-ray scattering make it possible to determine constellations of surface atoms precisely. Excellent guides of surface X-ray diffraction (SXRD) can be found in the literatures[3-6].

In SXRD, diffraction conditions are described as those for two-dimensional lattices as shown in Fig.2, and satisfied where either CTRs or fractional-order rods (FORs) intersect the Ewald sphere. CTR has contributions from both surface layers and the inner crystal whose periodicity perpendicular to the surface is truncated. In the kinematical expression, by summation of contributions to X-diffraction over a half infinite lattice plane, we get CTR intensity I_{CTR} as follows with neglecting a constant of proportion,

$$F(\boldsymbol{K}) \;=\; \int_{\mathrm{cell}} \rho(\boldsymbol{r})\, e^{2\pi i \boldsymbol{K}\cdot\boldsymbol{r}}\, d\boldsymbol{r}. \qquad (1)$$

$$I_{\mathrm{CTR}}(l) \;=\; \left| \frac{F^{B}_{hk}(l)}{1-e^{-2\pi i l}} + F^{S}_{hk}(l) \right|^2 \qquad (2)$$

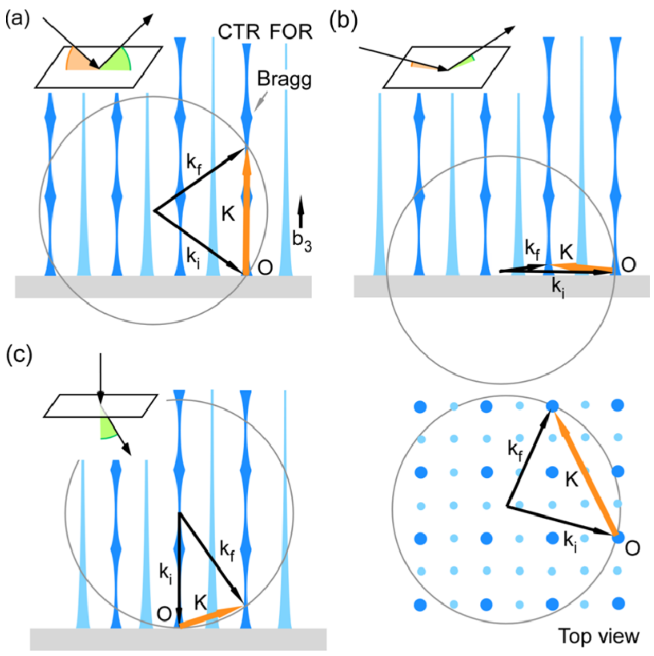

Figure 2. Reflection geometry for (a) out-of-plane, (b) in-plane surface X-ray diffraction, and (c) transmission geometry. Gray circles denote Ewald spheres.

Here, crystal structure factor $F(\boldsymbol{K})$ is the Fourier transform of charge density $\rho(\boldsymbol{r})$ in an unit cell. $\boldsymbol{K}$, $\boldsymbol{r}$ represent respectively a reciprocal space vector with the indices *hkl* and a position vector in the unit cell, and $F_{hk}{}^{B}(l)$, $F_{hk}{}^{S}(l)$ are structure factors of bulk crystal and surface layers, respectively. Bragg conditions are satisfied when l is integer and the right side of eq. (2) diverges. However, the CTR intensity enough away from the Bragg point compared to the Bragg width is well explained by the kinematical theory. This condition is almost always satisfied in SXRD. On the other hand, FOR is originating from a surface super-lattice with a lager unit cell in a different symmetry from that in the crystal by surface reconstruction in general.

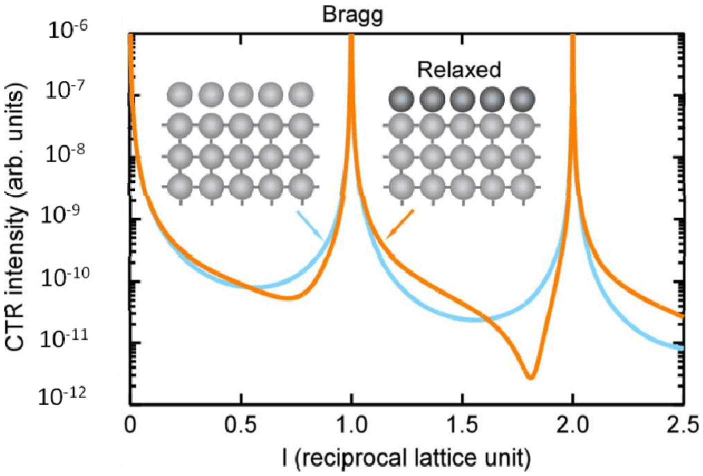

Figure 3. Calculated intensity of crystal truncation rod (CTR) scattering from a simple cubic crystal with a surface.

CTR is sensitive to a surface structure as shown in Fig.3 since contributions to CTRs from an inner crystal and its surface become comparable according to leaving away from the Bragg point. The blue curve is a CTR profile from the ideal crystal that surface atoms are in the same positions as they would be in the bulk. The orange curve shows a CTR profile from a crystal in which the top layer is relaxed inward by 7%.

3. Beamline Practical

We make in-situ observations of CTR scatterings from the Si(111)-$\sqrt{3} \times \sqrt{3}$-B reconstructed surface with the UHV-MBE chamber mounted on the S2+D2 diffractometer. Silicon surface is one of the most intensively studied surface as a prototypical semiconductor surface. This evidence comes from not only scientific interests to understand behaviors of semiconductor surfaces but also requirements from the industrial side to need making technological progress for developing reliable and/or novel-concept semiconductor electronic devices. According to device size small to nanometers, where surface states of electrons play a fundamental role in device performance, scientific knowledge becomes indispensable. Therefore, to know atomic constellations on surfaces is a fundamental issue in understanding physical and chemical properties on the surfaces at all.

Our plan of the practical is as follows:

1) Introduction of the beamline including optics, surface x-ray diffraction, the control software (SPEC) [7], and scientific activities at BL13XU.

2) Sample alignment: Alignment of the optical surface normal by observing laser reflection from a sample surface, and determination of a crystallographic sample orientation by detecting at least two Bragg reflections from the substrate.

3) Sample cleaning by heating a high boron-doped substrate at 1050℃ several times

4) Measurement of SXRD (CTR scatterings) at room temperature.

5) Data analysis: Deriving structure factors from the observed data by using correction factors, and making a structure analysis using the least-square fitting code developed at BL13XU.

References:

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