BL13XU: Training for Surface X-ray Diffraction Measurements

Beamline BL13XU is dedicated to the study of surface/interface structure using diffraction and scattering techniques, which is specifically called surface x-ray diffraction. Precise determination of the surface-atom arrangement typically calls for recording several hundred rocking curves of in-plane diffraction and collecting intensities of crystal-truncation-rod (CTR) scattering emanating from a reconstructed surface or surface-adsorbate system in ultra-high vacuum (UHV).

Such measurements require us to precisely control two angular parameters: The incident angles from the physical surface of the sample and Bragg planes of interest. Thus it is necessary to orient the physical surface and crystallographic planes of the sample in UHV.
The intensities are extremely weak compared with that obtained from a thin film, and of course, a bulk crystal. Thus surface x-ray diffraction measurements require not only a powerful x-ray source like the SPring-8 standard undulator, but also proper control of a BL monochromator and mirrors to make the incident-beam intensity maximum and stable.

The plan for training at the Cheiron School is to learn how to orientate the sample in UHV and successively make surface x-ray measurements. We will study the Cu(001) surface after monolayer heavy water (D_2O) dosing to investigate the sites of adsorption of water.
CTR scattering is a powerful technique which also yields information on atomic relaxation of the substrate due to D_2O adsorption. The principal facility for this training is a 1-ton UHV chamber mounted on a 2 + 2 type diffractometer for high angular resolution measurements.
We will quickly show how to align the beamline optics to tune the incident x-rays to the desired energy as well.

Schedule:

1) Guidance (15 min)

2) Introduction of beamline BL13XU [1][2] . (30 min)

Quick tour from the optics hutch to experimental hutch 3 to watch the Lq.

N_2 cooled monochromator, mirrors for rejecting higher hermonics, and typical facilities like diffractometers for in-air or in-solution measurements and for ultra-high vacuum measurements.

3) Brief review on surface x-ray diffraction (30 min)

Presentation on what surface x-ray diffraction is and what kind of information we can achieve. [3,4,5] For example, results and outcomes obtained at BL13XU.[2]

4) Quick show for tuning the beamline optics using the BL13XU workstation (15 min)

i) SPring-8 standard undulator,

ii) Lq. N_2 cooled monochromator.

iii) Mirrors for rejecting higher hermonics.

5) Alignment of surface diffractometer (60 min)

i) Diffractometer control with software SPEC [6].

ii) Sample alignment by a laser method

iii) Alignment of surface diffractometer with a pinhole assembly.

Lunch time (60 min)

6) Determination of orientation matrix for the sample surface (60 min)

7) Measurement of surface diffraction data on Cu (001)(180 min)

i) Checking the reconstruction of the surface using LEED.

ii) Dosing of monolayer D_2O and Checking the reconstruction of the surface using LEED.

iii) Measurement of CTR of the absorbate-surface system.

8) Dicussion (30 min)

References:

[1] BL13XU outline, URL:http://www.spring8.or.jp/wkg/BL13XU/instrument/lang-en/INS-0000000394/instrument_summary_view
[2]URL:http:http://www.spring8.or.jp/wkg/BL13XU/solution/lang-en/
[3] I.K. Robinson and D.J. Tweet, Reports on Progress in Physics, 55 (1992) 599-651.
[4] E. Vlieg, Surface Science, 500 (2002) 458-474.
[5] J. Als-Nielsen and D. McMorrow, "Elements of Modern X-ray Physics" (John Wiley and Sons, 2001).
[6] SPEC software, URL: http://www.certif.com/