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Supporting information for article:

Bent Crystal Laue Analyser Combined with Total Reflection Fluorescence-X-ray Absorption Fine Structure (BCLA+TRF-XAFS) and its Application to Surface Studies

Yuki Wakisaka, Bing Hu, Daiki Kido, Md. Harun Al Rashid, Wenhan Chen, Kaiyue Dong, Takahiro Wada, Bapurao Bharate, Quiyi Yuan, Shingo Mukai, Yasuo Takeichi, Satoru Takakusagi and Kiyotaka Asakura

S1. Design of the Home-made BCLA

A schematic of the home-made BCLA is shown in Fig. S1. The principle of the design is that the thin Si crystal is not only bent to the logarithmic spiral shape by the side frame, but the beams are positioned with a logarithmic spiral shape. The beams are set at the Mo Soller slit positions to reduce diffraction loss by the beams. The 150 × 40 mm^2 Si (111) with thickness of 0.05 mm was used for Laue analyzer. The reflection plane was another (111) plane tilted with 70.5° from the crystal surface normal. We made the BCLA expressed as $r=\rho_0\cos(\chi-\theta_B)\exp(\tan(\chi-\theta_B)\theta)$ with the curvature of ρ_0=100mm and χ(asymmetry angle) =19.5°. θ_B is the Bragg angle.

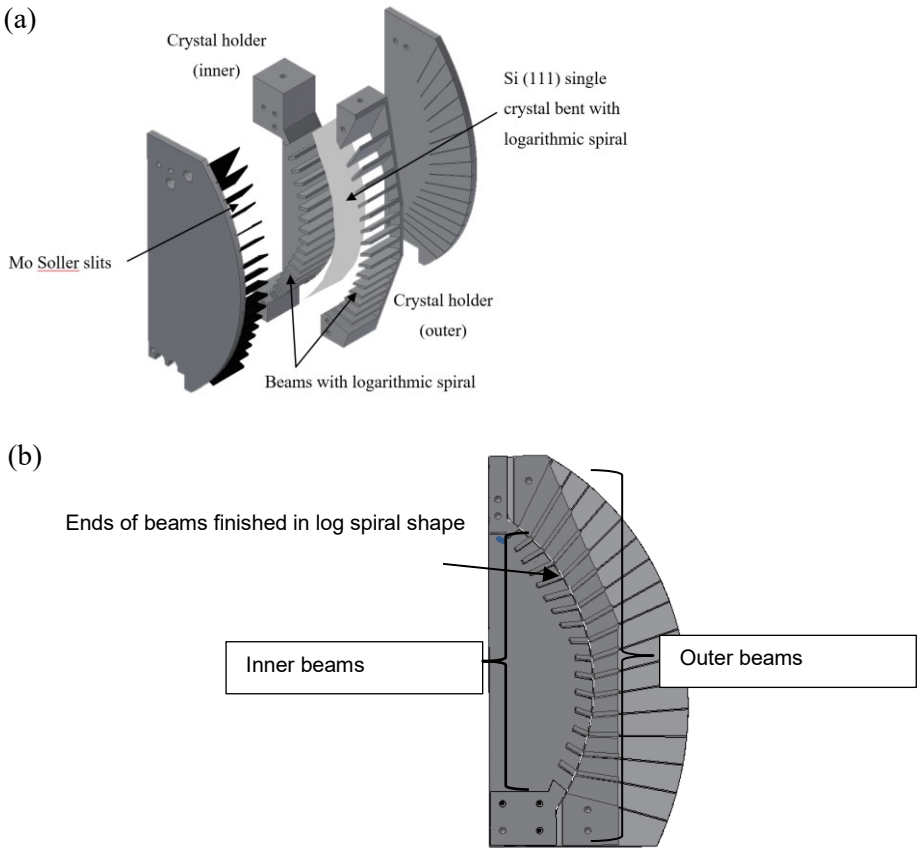

Figure S1 Home-made BCLA. (a) overall structures of home-made BCLA and (b)Side view. The ends of the log-spirally shaped inner and outer beams sandwiched the thin Si crystal.

S2. Principle of the BCLA

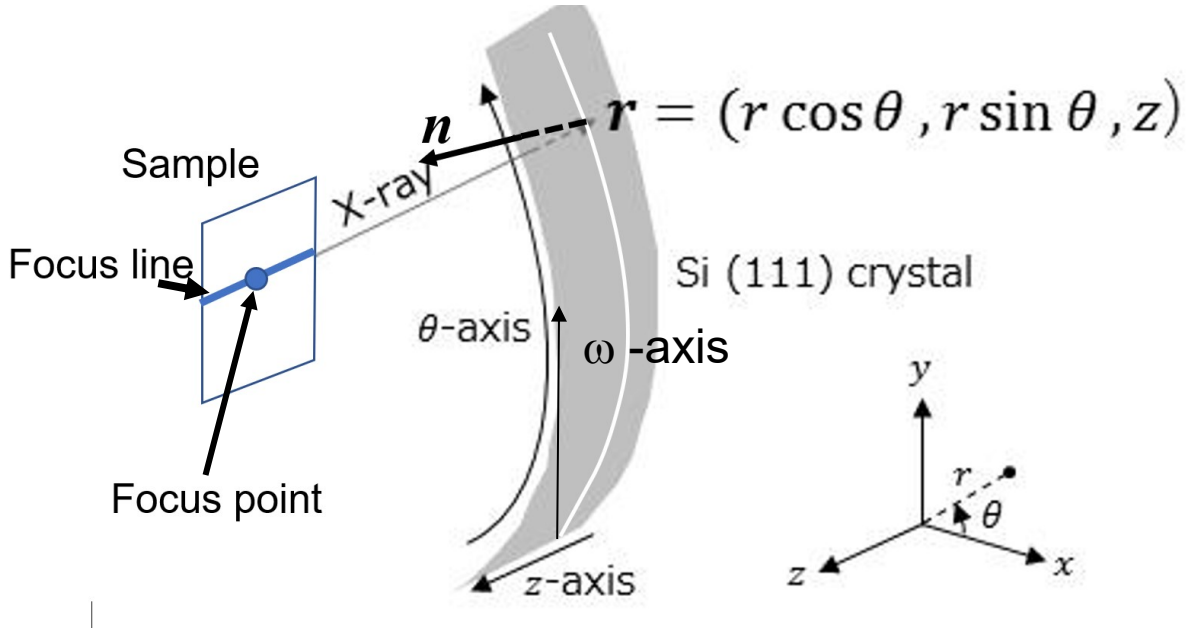

Figure S2 BCLA and focal point. ***n*** is the normal vector to the crystal surface and ***r*** is the radial vector from the focal point on the sample. The angle between the two vectors ***n*** and ***r*** is the same along the white curve on the crystal. The white curve is the intersectional curve of the BCLA crystal surface and the plane created by ***n***, ***r***, and the focal point. The blue line on the sample is the focal line of the BCLA crystal. When the X-ray footprint and focal line agree, the whole BCLA can accept X-rays with a constant wavelength.

S3. 45° incidence and grazing (parallel) arrangement of the BCLA

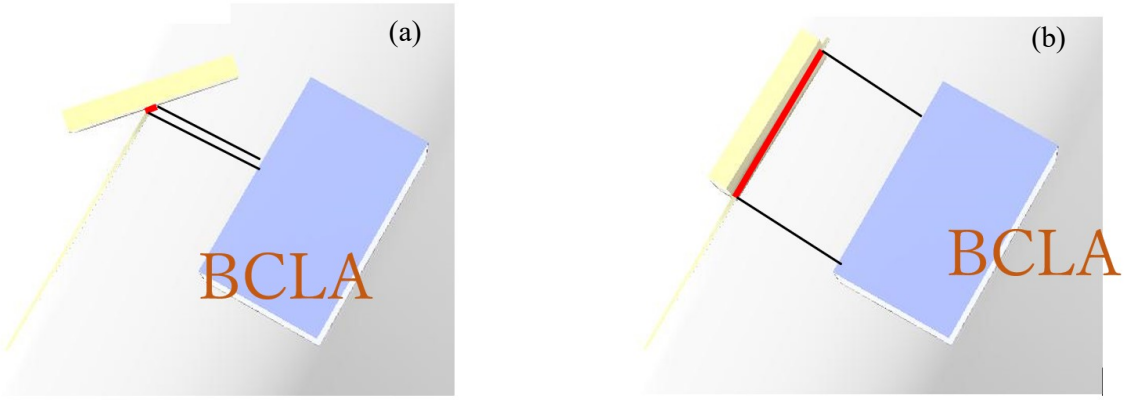

Figure S3 Relation between the sample and BCLA. (a) 45° incidence case. The BCLA is set parallel to the beam. (b) Grazing incident case. The BCLA is parallel to the surface. The BCLA can accept the X-ray fluorescence coming from the long footprint (red line) if the BCLA focal line (Fig. S2) and the incident X-ray footprint agree.

S4. CV of Pt / Au(111)

The Cyclic Voltammetry (CV) measurement was possible in the cell at the XAFS measurement position (the sample was about 1mm away from the window) as shown in Figure S4(a). The CV curve was similar to that measured at the position we usually used for electrochemical treatment (the sample was 3mm away from the window) as shown in Figure S4(b).

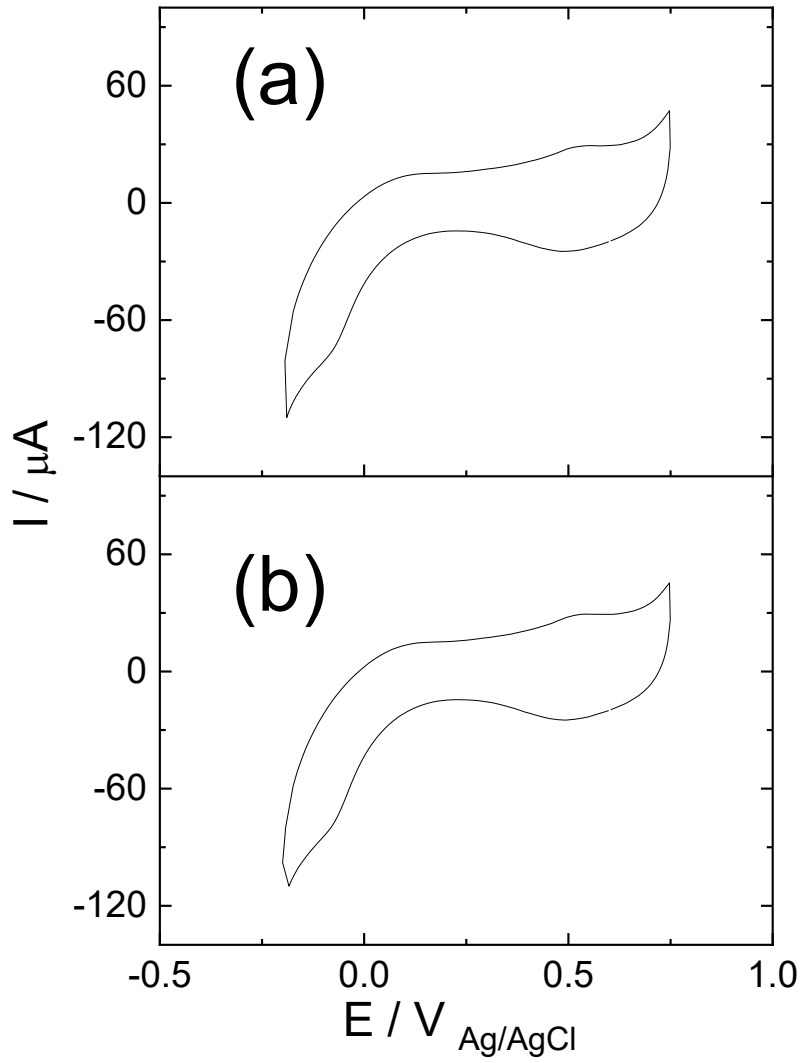

Figure S4 CV (scan rate =50 mV / s) of the 0.1ML Pt on Au(111) in the PTRF-XAFS measurement cell (a): The sample to the mylar window is 1mm(XAFS measurement position.) (b): The sample to the mylar window is 3 mm (electrochemical treatment position)

S5. Comparison of EXAFS with and without XAFS

We obtained the data above the Au edge using BCLA or so-called the range extended XAFS. Figure S5 shows the comparison between the observed and fitted data corrected and uncorrected spectra shown in Figure 6. Table S1 shows the fitting results in both spectra. Both are not so different because the S/N ratio is not so good above k~9.5 A^{-1}. If we use the third generation SR with a low emittance, the range extended XAFS by BCLA will have merits.

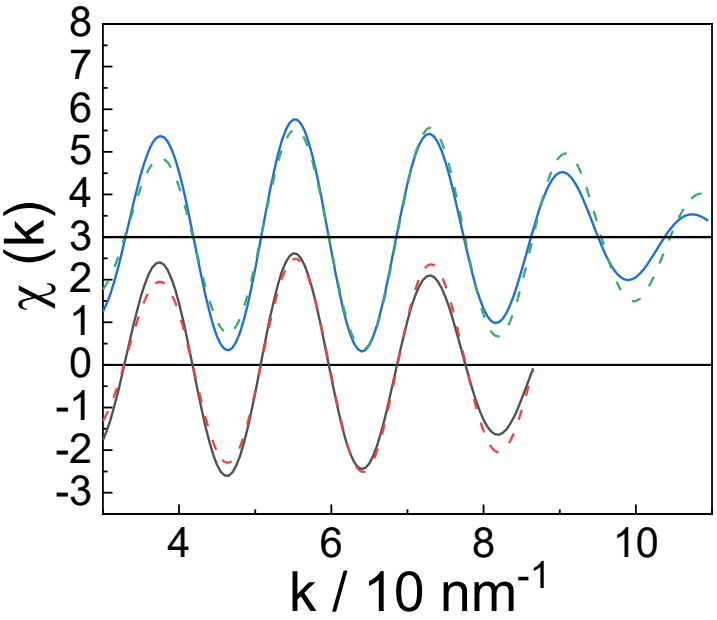

Figure S5 Comparison of observed (solid lines) and curve fitted (broken lines) data for uncorrected(bottom) and corrected ones.

Table S1 Curve fitting results of two corrected and uncorrected spectra. (CN= Coordination number) Pt-O bonding was assumed.

	CN	r / 0.1 nm	DE / eV	DW / 0.1 nm	R / %
Uncorrected (30-90 nm^{-1})	5.5± 0.5	2.05 ± 0.03	27 ± 5	0.09 (fixed)	2.7
Corrected (30-105 nm^{-1})	5.6± 0.5	2.06 ± 0.02	28 ± 5	0.09 (fixed)	3.7

S6. The removal of Au L a fluorescence by limiting the region of interest (ROI) of the multichannel analyser (MAC) for the SDD signal and the range extended XAFS

Figure S6 shows the fluorescence XAFS spectra with Pt-Au powder sample (Concentrations is 0.006 mol and 0.0006 mol for Au and Pt respectively.) When the ROI was set at 9180-9340 eV as shown in Figure S6b , we could get the Pt EXAFS without interference from the Au L□ shown below. Although the method was effective against the removal of Au fluorescence, yet it is not effective to remove the elastic scattering x-ray of the flat crystal under the solution because of the strong elastic scattering from the solution that causes the serious counting loss of SDD. Consequently, the BCLA is necessary to carry out the *in situ* measurement of total reflection fluorescence XAFS.

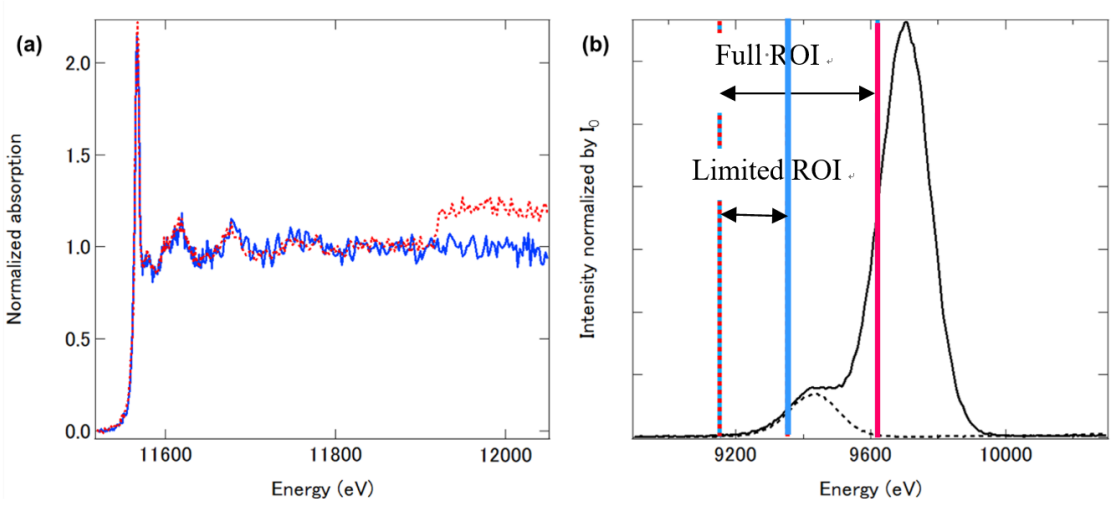

Figure S6 The XAFS spectra of PtAu particles detected by SDD(a). The selection of ROI in the MCA output. (b) Blue and red lines are corresponding to 9340 eV and 9620 eV, respectively.