Pseudo Atomic Column EELS & EDS Mapping of Silicon Reconstructed With K and L Electrons Using STEM-Moiré Method.

Eiji Okunishi¹ and Yukihito Kondo¹

Nowadays, an atomic column elemental mapping is popular method to determine an atomic position and species simultaneously [1] owing to radically innovated instrumentations such as aberration correctors [2], highly sensitive detectors [3] and highly refined analysis software. The field to apply the method is expanding, since the results of the method is intuitive with high spatial resolution. One of challenges for communization of this method is to overcome the sample damage caused by electron beam irradiation, since it need more electron dose than imaging due to small cross section of ionization of atoms. To reduce the damage, many attempts are tried and succeeded such as improvement of analytical sensitivity of X-ray detectors [3] and use of lower accelerating voltage [4].

On the other hands, the characterizations of the atomic column elemental mapping, such as spatial resolution or quantitative capability, have been pursuing to educe its advantages and disadvantages [5]. For example, delocalization of analysis, which is one of the important physical parameter, is determined by accelerating voltage of a primary electron and absorption edge energy of a sample. For the experimental estimation, it is ideal to perform under condition of no sample damage, since the damage makes displacement of atoms, resulting in a large experimental error.

The STEM moiré method is already successfully applied to measure the strain of the semiconductor device in very short time (totally a few minutes) [6]. An advantage for the STEM moiré method is that low electron density due to its sparse interval of scanning pixels is required for the analysis. We have tried to apply the STEM moiré method to reduce an electron beam density and obtained a pseudo atomic column map [7]. With the STEM moiré method, we can obtain an atomic column map under an electron density as < 1 % low as one used in conventional method (direct method [1]).

We used an aberration corrected microscope (JEM-ARM200F) with cold FEG, electron energy loss spectrometer (EELS) (Gatan Quantum ER) and silicone drift energy dispersive x-ray spectrometer (EDS) for this experiment operated at 200 kV. The sample was Si (110) with the thickness ≈ 250 nm and analytical signals were detected with EELS (for L shell electrons) and EDS (K shell electrons) simultaneously. To compare the result on K or L shell electrons, the elemental map was reconstructed with EDS for K shell and EELS for L shells.

Figures 1 (a), (b) and (c) show the simultaneously obtained pseudo atomic column maps of Si using HAADF signal (a), K shell X-ray detected by the EDS (b) and L shell electrons detected by EELS (c). In HAADF image, the dumbbell in Si (110) sample is clearly shown as in direct atomic resolution HAADF image. The elemental maps also show the atomic location of Si atoms. The magnification of these maps is estimated to be 10 x. The number of pixel of the map is 96 x 96, resulting in pixel resolution of 29 pixels for $a_{Si} = 0.58$ nm. The dose density on the sample over analysis time for moiré method is estimated to be 10^6 electrons / nm². The dose density for equivalent pixel resolution for the direct method is estimated to be 10^8 electrons / nm². Therefore, the total dose density is reduced to be 1%. Figure 2 shows the signal comparison of Si Ka by EDS, L_{2,3} electrons by EELS and HAADF after

^{1.} JEOL Ltd., 3-1-2 Musashino Akishima Tokyo, Japan.

background subtraction and normalization on line shown in Fig. 1(a) as blue rectangle. They show similar profiles. We expect that the resolution or delocalization of these signals is determined by electron trajectories in the Si crystalline, not by delocalization of the signals. For, further understanding of the delocalization could be demonstrated by thinner samples.

References:

- [1] E Okunishi et al, Microsc. Microanal. **14** (S2) (2008), p. 1372-1373.
- [2] M Haider et al, Ultramicroscopy. **75** (1.1) (1998) p.53-60.
- [3] S Kawai et al, Microsc. Microanal. 20 (S3) (2014), p. 1150-1151.
- [4] K Suenaga et al, Nature chemistry. **1** (5) (2009), p.415-418.
- [5] LJ Allen et al, MRS Bulletin. 37 (2012), p.47-52.
- [6] N Endo and Y Kondo, Microsc. Microanal. **20** (S3) (2014), p.1068-1069.
- [7] Y Kondo and E Okunishi, Microscopy. **63** (5) (2014), p. 391-395.
- [8] D Su and Y Zhu, Ultramicroscopy. **110** (3) (2010), p.229-233.
- [9] Y Kondo and N Endo. Kenbikyo. **49** (3) (2014) p.226-230.



Figures 1. (a) - (c) show the simultaneously obtained pseudo moiré atomic column maps of Si. These maps are composed of (a) HAADF image signal and (b) $K\alpha$ shell X-ray detected by EDS and (c) L shell (L_{2,3}) detected by EELS.

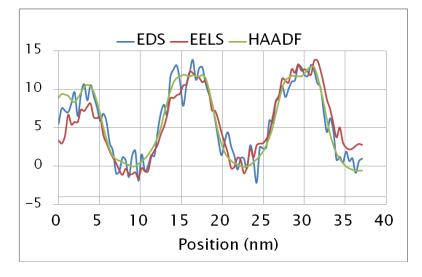


Figure 2. Signal comparison after background subtraction on line shown in Fig. 1(a) as blue rectangle.

EDS Si Ka = 1740 eV, EELS $L_{2,3}$ = 100 eV, HAADF, Eo = 200 kV