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Way to Reduce Electron Dose in Pseudo Atomic Column Elemental Maps by 2D STEM Moiré Method

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Atomic column elemental mapping, by EELS and/or EDS, is useful for material research, because we can clarify the elemental specie and atomic site simultaneously in these maps [1]. Though, the method requires high electron dose density on a sample, since the elemental signal is weaker than the image signal due to small ionization cross section for core electrons of atoms. Many efforts to acquire more elemental signal has been made for improvement of X-ray detection system in recent years, that is, the system with multiple silicon drift detectors (SDD) that have large sensor area, has been realized. And total solid angle of X-ray detection is > 1.75 sr in a detection system with two SDD detectors [2]. However, this improvement is insufficient for very electron-beam-sensitive materials. We need to find other way to have an atomic column elemental map with low dose density on samples.
We recently succeeded in displaying the pseudo atomic column elemental map utilizing a 2D STEM moiré by EDS [3] and EELS [4]. This method allows the atomic column mapping with the dose density < 1 % of one for the conventional method, since a pixel interval to draw the pseudo lattice fringe is sparser than that for a real lattice. We already reported the results on typical ceramics (Si_3N_4) [5] and very fragile mineral (Aquamarine: $Be_3Al_2Si_6O_{18}$: Fe^{2+}) [6].

In moiré method (see Fig. 1), a probe skips a number of unit cell as the pixel interval ($\boldsymbol{d_{rmoire}}$) have the relations $\boldsymbol{d_{rmoire}} = \boldsymbol{d_{lattice}} + \boldsymbol{\Delta_{moire}}$, where $\boldsymbol{d_{rmoire}}$ is a pixel interval in moiré method and $\boldsymbol{d_{lattice}}$ is a width of unit cell of a sample. And in conventional method $\boldsymbol{d_{rdirect}} = \boldsymbol{\Delta_{direct}}$, where $\boldsymbol{d_{rdirect}}$ is a pixel interval in conventional method. In this paper, we explain the way how to increase the dose reduce rate more than the rate by typical 2D STEM moiré method reduces.

Figure 2 shows the pseudo atomic column elemental maps of an aquamarine (oriented to the [0001]), which is very fragile for electron beam irradiation and has a hexagonal lattice in the [0001] direction. The dose density on the sample was 5.72 x 10^6 electrons / nm^2 with the total analysis time of 2089 sec and the probe current of 24 pA. The equivalent dose density, if the analysis was performed by the conventional direct method with the same pixel resolution, is estimated to be 4.42 x 10^{10} electrons / nm^2. Therefore, we could reduce the dose density to be 1/7700 for this experiment. Figure 3 shows the 2D unit cell of the sample for 2D STEM moiré method. To reduce the dose density, we used a super cell that has 4 times larger than the smallest unit cell of the sample crystalline. We also used a Cartesian unit cell instead of rhombus-shaped unit cell, which is used for crystallography, because the scanning system of the microscope cannot make a hexagonal pixel grating.

In conclusion, we explained the way to reduce dose onto the sample more than ever for analysis of very beam-sensitive samples by setting multiple-sized Cartesian unit cell for 2D moiré, and we succeeded in obtaining a pseudo atomic column elemental map for aquamarine.

References

[1] E Okunishi et al, Microsc. Microanal. **12 (S2)** (2006), p.1150.

[2] S Kawai et al, Microsc. Microanl. **20 (S3)** (2014), p.1150.
[3] Y Kondo and E Okunishi, Microscopy **63 (5)** (2014), **p.** 391.
[4] E Okunishi and Y Kondo, Microsc. Microanl. **22 (S3)** (2016), p.264.
[5] E Okunishi and Y Kondo, European Microscopy Congress 2016: Proceedings (2016), p.555.
[6] Y Kondo et al, European Microscopy Congress 2016: Proceedings (2016), p.542.

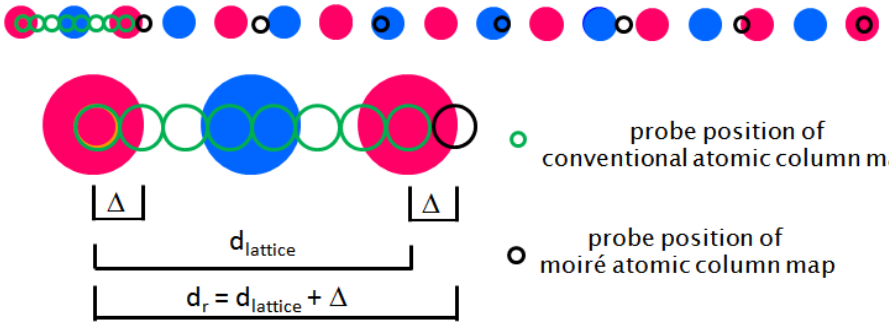

Figure 1. Pixel intervals in moiré method (shown as black circle) and direct method (shown as green circle). The pixels in moiré method point positions equivalent to those in direct method.

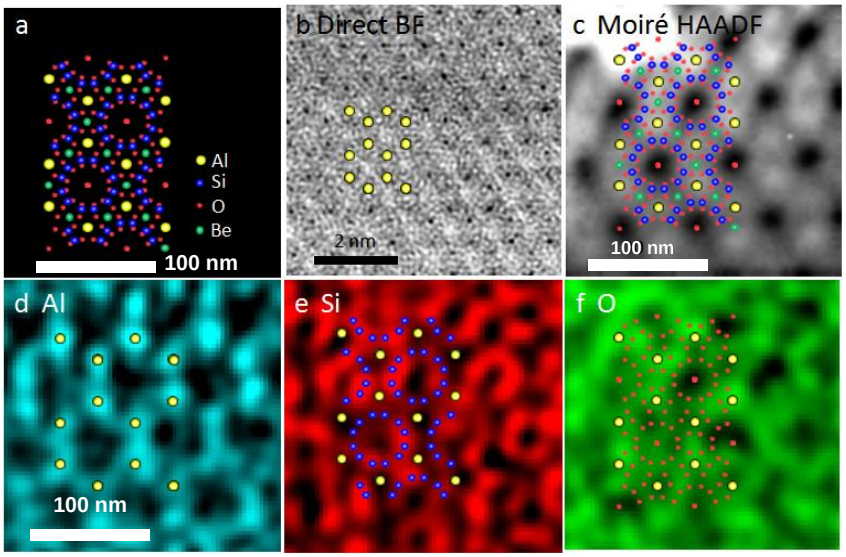

Figure 2. Pseudo moiré HAADF image, conventional direct BF image and 2D moiré atomic column elemental maps of aquamarine ($Be_3Al_2Si_6O_{18}$: Fe^{2+}). a: crystal structure, b: direct BF image, c: simultaneously obtained 2D moiré HAADF image, d-f: pseudo moiré atomic column elemental maps of Al, Si and O. We used JEM-ARM300F with cold field emission gun working at 300 kV.

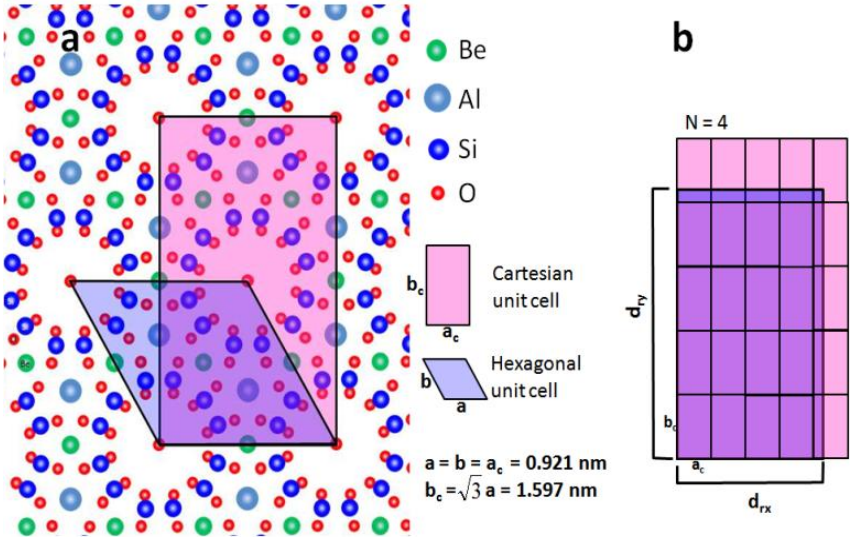

Figure 3. (a) The unit cell for STEM moiré method on 2D projected atomic site map of aquamarine ($Be_3Al_2Si_6O_{18}$: Fe^{2+}). We must use Cartesian unit cell for STEM moiré method, since the pixel positions are on the Cartesian grid, in general scanning system. (b) In the case that N is not 1, a pixel intervals are close to Na_c or Nb_c. In this configuration, the dose density reduced more than one in N = 1.

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