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Observation of Anomalous Kikuchi Patterns by Column-by-Column CBED

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The recent development of the spherical aberration correction of the electron microscopes allows us to form an electron beam with a diameter less than 0.1 nm. The sub-angstrom probe is used for ultra high resolution imaging in the bright-field and dark-field of scanning transmission electron microscopy and for electron energy-loss spectroscopy with atomic resolution, that is column-by-column EELS. In this study, we apply the sub-angstrom probe for observing the probe-position dependence of Kikuchi pattern.

MgO, which has the NaCl structure, was used as a test specimen. Incident direction is in the [110] orientation. Fig. 1 shows a schematic drawing of the atomic arrangement of MgO projected in the [110] orientation. The projected structure has two kinds of atom columns composed of Mg and O, and two kinds of atom planes composed of Mg (solid lines) and O (dashed lines).

Simulation of Kikuchi patterns was carried out by the multislice method, where a thermal vibration of atom was introduced by the frozen phonon approach [1]. Fig. 2(a) and 2(b) show diffraction patterns simulated with the probe located at Mg site and O site. Kikuchi band due to the {110} planes is indicated by arrow and dashed lines. Moreover, the 110, the 001 and the 222 bands are indicated by dashed lines. When the probe locates at Mg site, the 111 band is brighter than the 222 band. In contrast, it shows week the 111 band intensity relatively compared to the 222 band when the probe is positioned at O site. The change of the intensity can clearly be seen in the ratio pattern shown in Fig. 2(c). However, the other Kikuchi bands show little change for instance the 110 band or the 001 band.

Experimental patterns were taken by using a transmission electron microscope, JEOL JEM-2100F equipped with a CEOS aberration corrector for the probe forming lens system, operated at an acceleration voltage of 200 kV. The incident probe size with a convergence semi-angle of 24 mrad was about 0.1 nm. An MgO single crystal was crushed and dispersed on a holy carbon film on a Cu grid. Fig. 3(a) and Fig. 3(b) show two typical diffraction patterns. The characteristic of the 111 and 222 bands show good correspondence to Fig. 2(a) and Fig. 2(b). That is, the 111 bands in Fig. 2(a) and Fig. 3(a) show higher intensity than the 222 bands, and the 111 bands in Fig. 2(b) and Fig. 3(b) show darker than the 222 bands. Therefore, we consider that Fig. 3(a) is obtained when the probe is positioned at Mg site, and Fig. 3(b) is obtained when the probe is positioned at O site. Fig. 3(c) shows a ratio pattern of Fig. 3(a) to Fig. 3(b). The difference in the 111 band between the two patterns is clearly seen in Fig. 3(c). The beam-position dependence of the 111 band intensity can be interpreted by electron channeling and the reciprocity theorem.

References

[1] R. F. Loane, P. R. Xu and J. Silcox, Acta Cryst. (1991), A47: 267-278.

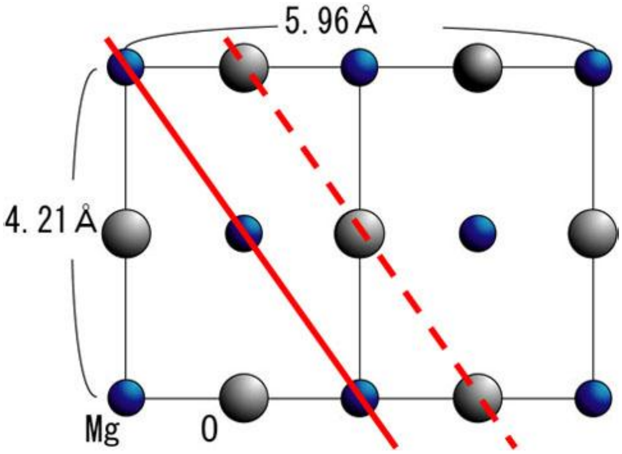

Fig. 1. Projected figure of MgO [110].

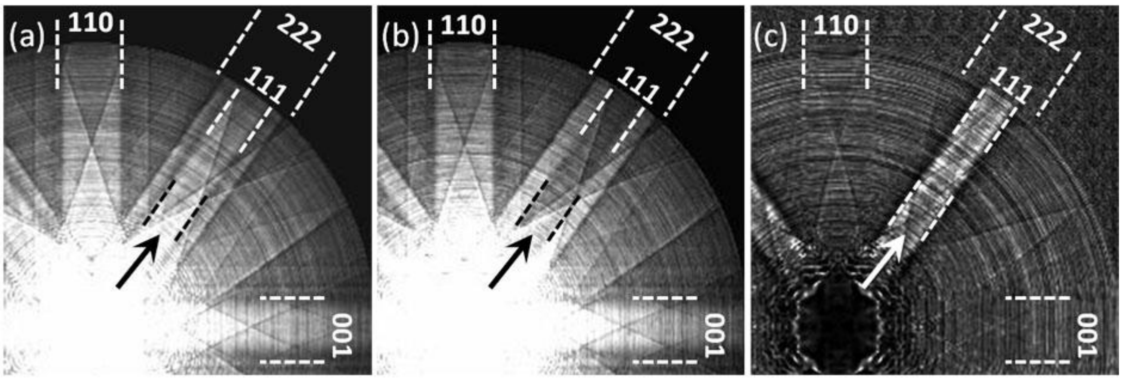

Fig. 2. Simulated diffraction images. 111 band is indicated by arrow. (a) Probe locate at Mg site, (b) Probe locate at O site, (c) The ratio pattern of (a) to (b).

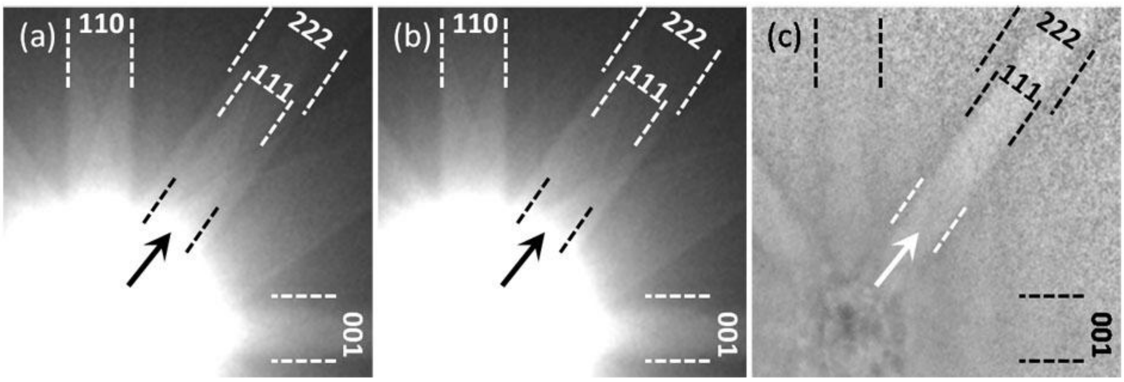

Fig. 3. Experimental diffraction images. 111 band is indicated by arrow. (a) Probe locate at Mg site, (b) Probe locate at O site, (c) The ratio pattern of (a) to (b).