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Investigation of Diffraction Condition and Convergent Probe Effects on Inelastic Mean Free Path Determination by Using a Cone-Shaped Silicon Crystal

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As an important parameter in foil thickness measurement by electron energy-loss spectroscopy (EELS), the inelastic mean free path (λ) not only varies with the collection angle (β) of scattered electrons, but also depends on diffraction conditions of crystalline materials and the convergent angle (α) of the incident electron beam. In general, λ decreases with increasing β. However, details of α and dynamical diffraction effects on the variation of λ are not very clear, though some work has been done on this topic [1, 2]. For a diffraction condition on a zone-axis with many beams within the collection aperture, the intensity ratio of inelastic and elastic scattering in the acquired EEL spectrum, i.e., I_{in}/I_{el}, can be expressed as:

$$\frac{I_{in}}{I_{el}} = \frac{I_{in}(0)}{I_{el}(0)} \cdot \frac{1+\sum_{\theta\neq 0}\omega(\theta)}{1+\sum_{\theta\neq 0}b(\theta)} \tag{1}$$

where $I_{in}(0)$ and $I_{el}(0)$ are the inelastic scattering intensity and elastic scattering intensity contributed from around the transmitted beam. $\omega(\theta)$ and $b(\theta)$ represent the relative contributions to the inelastic scattering and elastic scattering, respectively, from around a diffracted beam with Bragg angle θ. The intensity ratio, I_{in}/I_{el}, determines the value of λ.

Utilization of a cone-shaped Si crystal prepared by a focused ion beam (FIB) [3], as shown in Fig.1, can overcome the difficulty in examining the dynamical diffraction effect on λ. In the experiment, the cone axis was aligned to the A-tilt axis so that the sample thickness (t) passing through the cone axis was always equal to the projected cross-section diameter (w), regardless of the A-tilt. The effect of misalignment between the cone axis and the A-tilt axis on a sample thickness measurement can be estimated as $t = w/\sqrt{1-\sin^2\delta\sin^2\alpha'}$, where δ is the angle between the two axes and α' is the angle of A-tilt. This effect is negligible with careful alignment of the two axes and slight A-tilt.

As shown in Fig.2, three types of diffraction conditions, many-beam, two-beam, and single-beam, were used in this study. To achieve these diffraction conditions, the A-tilts are 6^o, 3^o, and 11^o, respectively, with almost no B-tilt ($<2^o$). To evaluate the dynamic diffraction effect, the convergent angle α = 6.4 mrad was used, while α = 15 mrad was used to study the effect of the convergent probe. For each α, different values of collection angle β from 4.6–95 mrad were used by varying the camera length and the spectrometer entrance aperture. All experiments were conducted at 200kV and the EEL spectra were acquired with a diffraction pattern on screen. As shown in Fig.3a, the effects of diffraction conditions on λ are negligible at large collection angles. Even at low collection angles, they only make moderate differences (less than 6%). These results suggest that the transmitted beam in Eq. (1) plays a dominant role in determining λ, regardless of diffraction conditions. On the other hand, the convergence of the probe only affects λ by reducing its value when $\alpha>\beta$ (Fig.3b). For $\beta>\alpha$, α has no effect on λ. The value of λ depends on β only and starts to saturate at $\beta \approx$ 20mrad. The results shown in Fig.3b support those reported previously [1].

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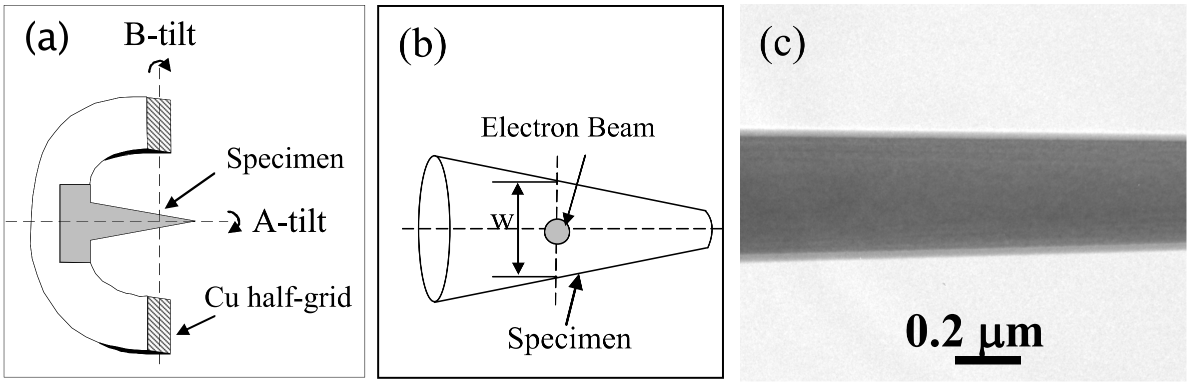

Fig.1 A cone-shaped specimen, (a) position alignment; (b) thickness measurement; (c) a TEM image.

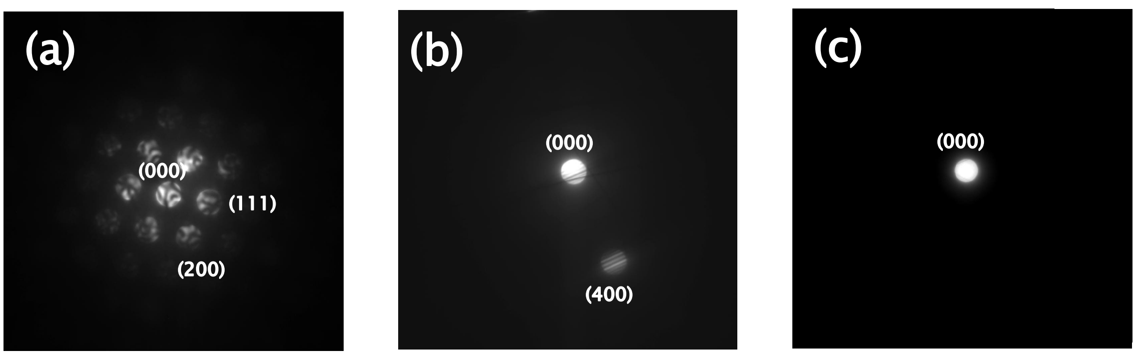

Fig.2 Diffraction conditions, (a) many-beam on (110) zone; (b) (400) two-beam; (c) (000) single-beam.

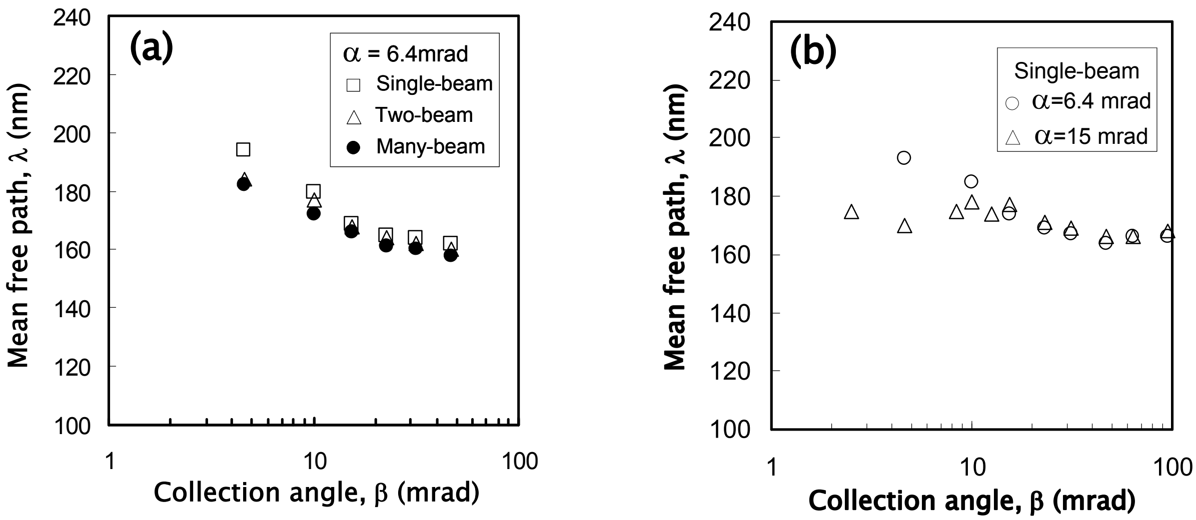

Fig.3 Measured λ values at sample thickness t = 450 nm with different collection angles, (a) effect of diffraction condition; (b) effect of convergent probe.

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