Intensity measurement of equal thickness fringes in wedge-shaped crystal images and estimation of crystal potential

Koji Nishio[1], Toshiyuki Isshiki[1] and Makoto Shiojiri[1,2]

[1]Department of Electronics and Information Science, Kyoto Institute of Technology, Matsugasaki, Sakyo-ku, Kyoto 606-8585, Japan
[2]Department of Anatomy, Kanazawa Medical University, Uchinada, Ishikawa 920-0293, Japan

Summary: Quantitative measurement of intensity profiles of equal thickness fringes has been carried out in TEM images of wedge-shaped crystals using a slow-scan CCD camera (SSC) and an imaging plate (IP). The intensities are measured in bright and several dark field images for [100] GaAs, InP, and Si and [$1\bar{1}0$] MgO, with and without an energy slit having ± 5 eV energy width for incident electrons. Calculated diffraction intensities are compared with the experimental data to investigate suitable complex potential.

1. Introduction

Equal thickness fringes in a wedge-shaped crystal image are a direct representation of dynamical electron diffraction intensity. Therefore, many investigations were carried out on the basis of the measurement of the equal thickness fringe intensities, and they have contributed to the improvement of the dynamical electron diffraction theory and the evaluation of the structure factor of crystals. These experiments were, however, performed under the two-beam excited Bragg condition, and not under the multi-beam excited zone axis condition which is used in the usual HRTEM work. In this paper, zero-loss filtered intensity profiles as well as unfiltered ones of equal thickness fringes of several crystals under the zone axis condition are provided [1, 2]. Quantitative HRTEM depends entirely on the simulation, which contains various approximation and might have bugs or errors. An objective inspection of the programs is indispensable for its accuracy, by means of the comparison between the present kind of experimental data and the simulated results. The simulated results are checked with respect to the obtained experimental data, using various crystal potentials.

2. Experimental

GaAs, InP, and Si single crystals cleaved along the {110} planes and MgO single crystals, having the {100} surfaces, prepared by burning Mg in air were used. Observed conditions are summarized in Table 1. The crystal orientations were exactly adjusted using its diffraction patterns including Kikuchi lines. The edge of the specimen along the [001] axis has 90° wedge-shape, thus the thickness can be derived as a function of the distance from the edge. The magnification of the images was calibrated using lattice images recorded under the same condition. The intensity of the equal thickness fringes was normalized with respect to the incident beam intensity which was measured in a vacuum region.

3. Results and discussion

Figure 1 shows intensity profiles of equal thickness fringes in GaAs and InP crystal images. Since inelastically scattered electrons contribute to the intensity, it has been revealed that the correspondence of the calculated intensities which ignored the inelastic scattering to the experimental profiles is limited to thinner region than 20 nm in specimen thickness [1].

Figures 2 and 3 indicate the zero-loss filtered (thick solid lines) and unfiltered (thin solid lines) intensity profiles of equal thickness fringes of Si and MgO, respectively. Measurement of the zero-loss filtered intensities using an energy filtering TEM enables us to cut the contribution of inelastically scattered electrons mainly due to plasmon excitation out of the profiles.

Comparison between the experimental profiles and the ones calculated with our multi-slice program [3] was carried out for Si and MgO, by using various structural parameters such as the scattering factor, the temperature factor and the absorption coefficient given as the ratio of imaginary part of the complex potential to the real part. It has been confirmed that a Bethe program [4] produces almost the same results as those by our program [2]. In the calculations neglecting inelastic scattering for

Si, the positions of peaks almost agree with the experimental one but the intensities of them disagree. The best agreement was obtained by introducing a complex potential of $(1+0.04i)V_{real}$ and a temperature factor of 4.5×10^{-3} nm^2 (dashed lines in Fig. 2). For MgO crystal, the calculation with a potential due to neutral atoms could not account for the experimental result, giving poor agreement in the positions as well as the intensities of peaks. The agreement improves when the calculation was performed using the complex potential, taking account of a mixture of the both scattering factors for neutral atoms and ions (dashed lines in Fig. 3).

Acknowledgements: The authors would like to express their thanks to Drs. E. Okunishi, T. Oikawa and M. Kawasaki, JEOL Ltd., and Dr. H. Kakibayashi, Hitachi Ltd., for their experimental support.

Table 1 Observed conditions.

Specimen	GaAs, InP	Si	MgO
Orientation	[100]		[110]
Dif. spot	000, 002, 022, 040	000, 022, 040	000, 111, 002, 220, 113, 222, 004
TEM / Rec.	H-800 / SSC	JEM-2010FEF, JEM-2010EF / IP	
Energy filter	—	Zero-loss filter (±5 eV), unfilter	
Accel. volt.	175 kV	200 kV	
Mag.	60,000	$\geq 200,000$	

References

[1] K. Nishio, H. Kakibayashi, T. Isshiki, H. Saijo and M. Shiojiri, *J. Elec. Micros.*, **43** (1994) 198.

[2] K. Nishio, T. Isshiki and M. Shiojiri, *J. Electron Microscopy* (submitted).

[3] K. Nishio, T. Isshiki, H. Saijo and M. Shiojiri, *Ultramicroscopy*, **54** (1994) 301.

[4] P. A. Stadelmann, *Ultramicros.*, **21** (1987) 131.

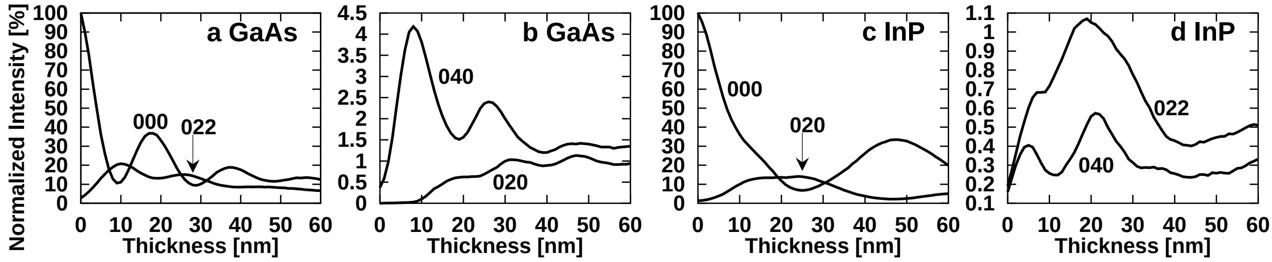

Fig. 1 Intensity profiles of bright field, and 020, 022, and 040 dark fields of GaAs (a, b) and InP (c, d), respectively.

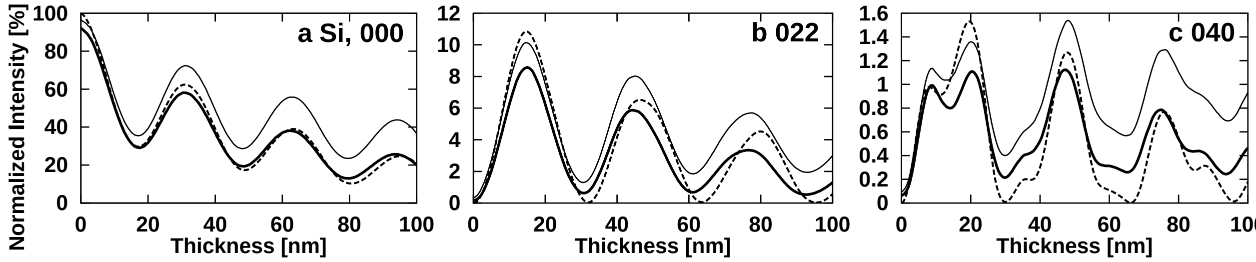

Fig. 2 (a-c) Intensity profiles of bright field, 022 dark field and 040 dark field of Si with (thick solid) and without (thin solid) an energy slit of ±5 eV in width, and corresponding calculated profiles (dashed), respectively.

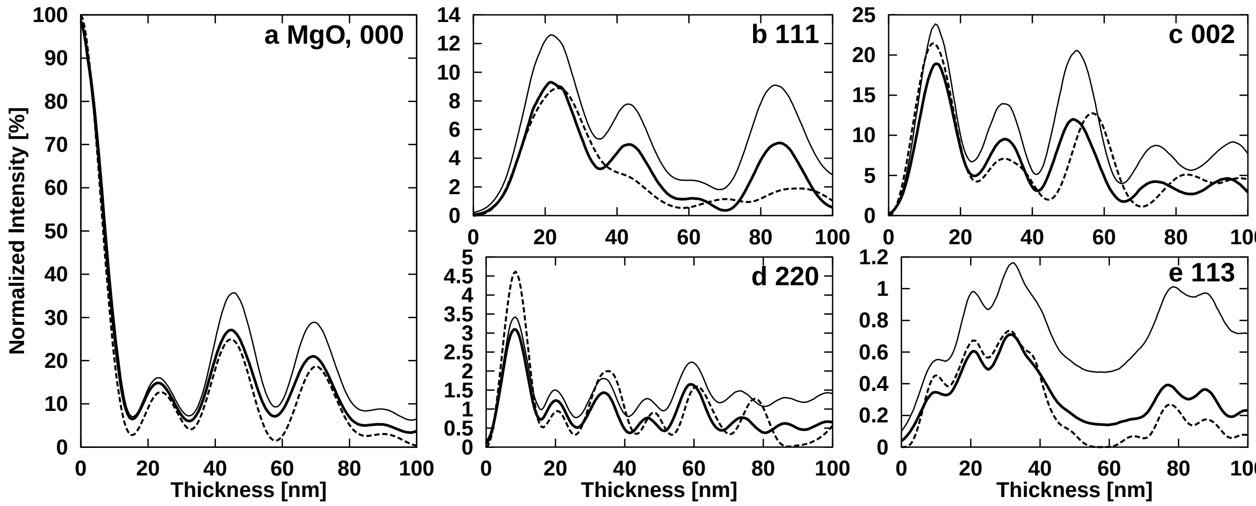

Fig. 3 (a-e) Intensity profiles of bright field, and 111, 002, 220, and 113 dark fields of MgO with (thick solid) and without (thin solid) an energy slit of ±5 eV in width, and corresponding calculated profiles (dashed), respectively.