High-Tension Determination of Transmission Electron Microscopes by means of Convergent-Beam Electron Diffraction (CBED)

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MOTIVATION

CBED is a well-established method for local strain and lattice parameter measurements [1-4]. Application of line detection algorithms based on Hough transformation allows HOLZ-Line detection with accuracy below one pixel [3, 5] and thus line detection does not contribute significantly to the resulting accuracy of lattice parameter measurement. The main sources of errors, physically limiting the accuracy, are the image noise [5] and the systematic errors. The latter include HT drift during experimental time-slice and thickness determined shift of HOLZ lines [6]. In this work we evaluate and compare the long term HT stability for CM-20 and TITAN 80-300 microscopes for the range of operating conditions. Absolute value of HT is determined by CBED method exploiting Si [230] zone as a standard. As far as absolute values are measured, the a priori knowledge of the measurement errors becomes a key issue. This roadmap for thorough evaluation of measurement accuracy including SNR related errors and thickness correction is suggested and approved.

METHODS

Experimental Setup

- Analytical sample: Si plane-view sample of [110] orientation, lattice constant 5.431Å to get large areas without defects.
- Analytical projection: [230] incident direction.
- Exposure techniques: CM-20 (200kV): LACBED was used to filter out thermal diffuse scattered electrons to increase SNR. TITAN (80kV/300kV): Normal CBED was used to filter out inelastic scattered electrons and increase SNR. Images were 2D filtered by using GIL.

Prerequisites

- Absolute HT value (energy at sample location) of interest to lattice parameter determination by comparing HOLZ-Line positions of experimental and calculated CBED patterns.
- Reduction of systematical errors. Usage of dynamical calculations and consideration of the thickness depending HOLZ-Line position by thickness correction.
- Errors of HT values important to evaluate influence of HT accuracy on lattice parameter determination. Therefore the total error of the experimental image was calculated considering SNR.

Workflow

- HT determination by calibration
- Error calculation
- Thickness correction

RESULTS

CT Bars for CM-20 and TITAN Microscope

- Errors of the HT values in diagram 1: ±55V.
  A: Large HT down-drift directly after HT started. After 7h HT stable.
  B: C: As expected in respect of A, HT stable and constant until HT-drift switch-off. After HT switching HT jump (~100V) between A and B probably because of temperature changes between different days.
- Errors of the HT values in diagram 2: ±34V.
  D: Small up-drift (~50V) reaching HT Level of E. HT oscillations observed until 2h after switch-over.
  E: Very stable HT run without drifts and constant within the errors.
- Errors of the HT values in diagram 3: ±12V.
  F: Small down-drift (~40V) with HT oscillations until 3h after switch-over. Afterwards HT-Level of G reached. HT very stable without drifts.
  G: As expected in respect of F, HT very stable run without drifts and constant within the errors.

Comparison of CBED and Zero-Loss-Peak Drift

- Simultaneously recorded drift of ZLP plotted in addition to CBED data.
- Only changes of the ZLP known, not the absolute HT values.
  First ZLP position calibrated to 0V and fitted to the first value of the CBED run.
  ZLP drift confirms HT down-drift determined by CBED. The peaks in the ZLP run indicate calibration problems of the GIF during HT oscillations.

Conclusions

It was shown that CBED provides a possibility to determine absolute high-tension values (electrolysm at the sample location) of the microscopes with an accuracy in volt-range. For the microscopes examined a relaxation period was found directly after HT switch-on (CM-20) and after 80kV/300kV HT switch-over, during which HT oscillates and drifts in the range of hundreds volts. After this relaxation period the value of HT remains constant within the measurement error (see formula 5).

In particular such stability of the instruments allows to neglect the systematic error due to HT drift during lattice parameter measurements by means of CBED because HT drifts introduce an error which is much smaller than the one determined by the image noise.

REFERENCES


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