High-Resolution Transmission Electron Microscopy with Zach Phase Plate

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Introduction

Physical phase plates (PP) enhance the contrast of weak-phase objects in transmission electron microscopy (TEM) [1]. Different methods exist to impose the required relative phase shift between scattered and unscattered electrons [2]. A major disadvantage of the most common thin-film based PPs is the scattering of electrons in the PP itself leading to a phase-contrast damping and a loss of resolution. The electrostatic Zach-PP (Fig. 2) induces a variable phase shift on the unscattered electrons in the back focal plane (BFP) of the objective lens (Fig. 1) [3]. As a damping or a loss of resolution is not expected, this work aims on the application of the Zach-PP in high-resolution (HR)TEM.

Design and fabrication of a Zach-PP

The Zach-PP consists of a single rod carrying a 5-layer system with Au electrode, insulating layers of Si₃N₄ and Al₂O₃ and Au shielding. Additional evaporation of amorphous carbon minimizes contamination and electrostatic charging. Complex fabrication process needed using electron-beam lithography, physical vapor deposition, reactive ion etching and focused ion beam milling as described in [1]. Implementation in the BFP of a Zeiss 9230 transmission electron microscope operated at 200 kV and equipped with a TVIPS CCD camera.

Sample characterization

Investigation of a Si single-crystal sample in [110] orientation. The (111)-type lattice fringes and reflections are visible in the HRTEM image (Fig. 3a) and the corresponding power spectrum (Fig. 3b).

HRTEM image formation with Zach-PP

The influence of the Zach-PP on the image formation process in HRTEM is best analyzed by the reflection intensity in power spectra. Assuming isotropic conditions, the reflection intensity $I$ shows a cosinusoidal dependence on the induced phase shift $\phi_0$.

$$I(u) \propto 2a_2u^2E(u)\cos(\phi_0 + \chi - \psi_{PP})$$ (Eq. 1)

with the envelope function $E(\cdot)$; the wave aberration function $\chi$; the amplitude and phase of the diffracted/undiffracted beam $a_{111}/\psi_{90}$ and the spatial frequency $u$.

References

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Application of the Zach-PP

- Visibility of lattice fringes and reflections indicate a negligible phase-contrast damping induced by the Zach-PP (compare Figs. 3 and 4).
- (111) reflections are distinguished by their position with respect to the PP-rod (white: Affected (red) and unaffected (green)).

Analysis of the reflection intensity

- Acquisition of a HRTEM image series of the same sample area with different applied voltages and otherwise unchanged conditions.
- A linear dependence of $I_{PP}$ on the applied voltage is verified by Thon-ring analysis.
- Determination of (111) reflection intensities in Wiener-filtered power spectra ($I_{PP}$ in Fig. 5).
- Fit to a function with cosinusoidal behavior (Eq. 1) yields a good agreement with the measured data points (Fig. 5).
- Undesired aberrations, charging effects or a deviation from perfect zone-axis orientation hamper the analysis and lead to the observed lower intensity of the (111) reflections affected by the PP rod (red curve in Fig. 5).

Fit $I(\psi_{PP})$ to a function with cosinusoidal behavior $I_0 \cos(\phi_0 + \chi - \psi_{PP})$ (Fig. 5).

Phase-contrast inversion in HRTEM images

- If one reflection is blocked by the PP rod, the HRTEM pattern is reduced to a line pattern (Fig. 6a), which is formed by the unaffected reflection pair (marked green in Fig. 6c).
- The remaining (111) reflection pair and the corresponding lattice fringe contrast can be influenced by $\psi_{PP}$.
- The contrast of the lattice fringes can be inverted by applying appropriate PP-voltages, which is shown in the line profile in Fig. 6b taken from manually aligned images.

Summary

- Application of the electrostatic Zach-PP for HRTEM is advantageous.
- Oscillation of the reflection intensity with varying $\psi_{PP}$.
- Phase-contrast inversion of lattice fringes induced by the Zach-PP.
- Good agreement between experimental and theoretical calculations.

Outlook

The application of the Zach-PP is limited by the capabilities of the microscope. Further investigation in a state-of-the-art microscope could offer:

- Determination of local information like sample thickness or composition.
- Quantitative HRTEM by object-wave reconstruction with Zach-PP P.
- Improved resolution in single-particle reconstruction.