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Application of a Hilbert phase plate in transmission electron microscopy of materials science samples

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Introduction:

- Weak-phase objects like biological samples show low contrast in conventional TEM images.
- Additional phase plate creates phase shift of $\pi/2$ between unscattered and elastically scattered electrons, enhancing the contrast of weak-phase objects[1].
- Goal of present study: Application of phase-contrast TEM in the investigation of materials science samples.
- Hilbert phase plate[2]: Thin amorphous carbon (a-C) film covering half of the back focal plane (BFP) of the objective lens without affecting the beam of transmitted electrons (Fig. 1).

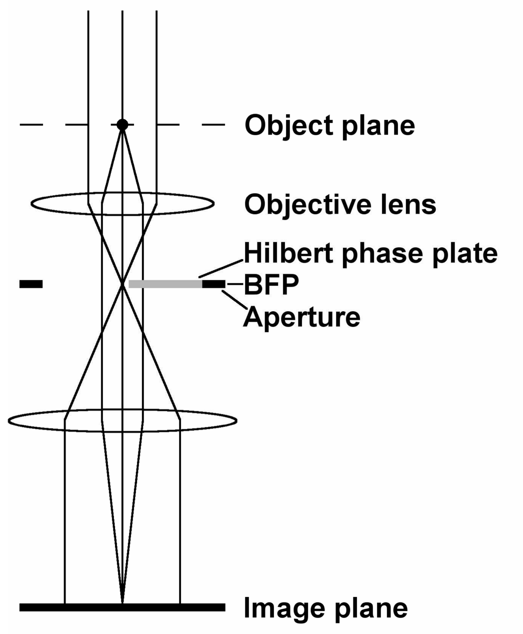

Fig. 1: Hilbert phase plate covering half of the BFP of the objective lens.

Proof of phase plate concept:

- Thon rings outside the narrow stripe (indicated by arrows in Fig. 2) are shifted.
- Nearly cosine-type phase contrast transfer.

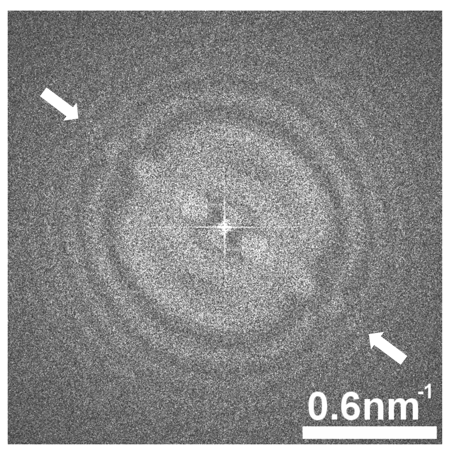

Fig. 2: Fourier-transformed image of an a-C film acquired using Hilbert phase plate.

Experimental techniques:

- Electron-beam evaporation of Al_2O_3 and C.
- Focused ion beam (FIB) milling using a *Zeiss EsB 1540* FIB system.
- High-resolution TEM (HRTEM) and electron holography using a *Philips CM200 FEG/ST* TEM.
- Electron energy loss spectroscopy (EELS) using a *Zeiss Leo 912 OMEGA* TEM.
- TEM samples prepared by standard methods as well as FIB techniques using a *FEI Strata 400 STEM* FIB system.
- Application of the Hilbert phase plate in a *Zeiss Leo 912 OMEGA* TEM.

References:

[1] F. Zernike, *Phys. Z.*, 23, (1935), pp. 848

[2] R. Danev, K. Nagayama, *J. Phys. Soc. Jpn.*, 73, (2004), pp. 2718-2724

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Fabrication of the Hilbert phase plate:

- Adjustment of the a-C film thickness to induce a phase shift of π using the equation $\Delta\varphi = C_E\, V_0\, t$ with the interaction constant C_E, the mean inner potential of a-C V_0 and the film thickness t. At *120 keV*, $t = 36\ nm$ is required assuming $V_0 = 10\ V$.
- Deposition of the a-C film on cleaved mica surfaces using electron-beam evaporation.
- Floating of the a-C film on the surface of distilled water and placement on standard copper grids (Fig. 3(a)).
- Milling of rectangular apertures into the floated a-C film using a FIB system (Fig. 3(b), (c)).

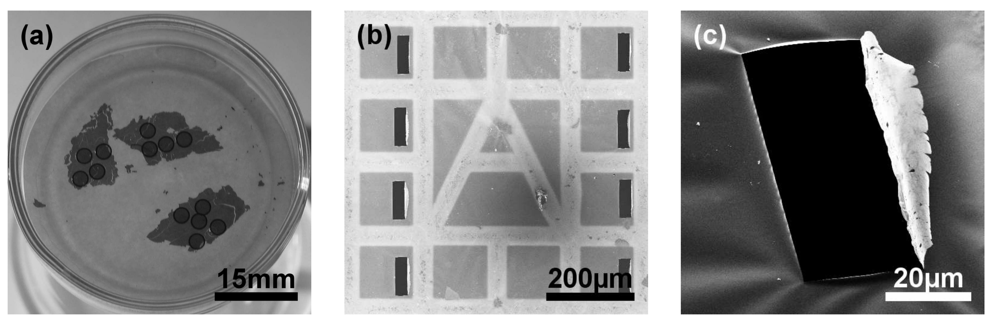

Fig. 3: Fabrication of a Hilbert phase plate.
(a) Floated a-C film on grids.
(b) Hilbert phase plates in several meshes.
(c) Single Hilbert phase plate.

Characterization of the Hilbert phase plate:

- Measurement of the a-C film thickness either using cross-section TEM samples (Fig. 4) or by EELS (Fig. 5) using the equation $t = \lambda_i \ln(I_{tot}/I_0)$ with the inelastic mean-free path λ_i, the total intensity I_{tot} and the intensity contained in the zero-loss peak I_0: Thickness of the Hilbert phase plate was determined to be $t = 32\ nm$.
- Measurement of the induced phase shift by electron holography (Fig. 6): Phase shift induced by the Hilbert phase plate was determined to be $0.8{\cdot}\pi$ at *120 keV*.
- Mean inner potential of a-C deposited by electron-beam evaporation was determined to be $V_0 = 8.9\ V$.

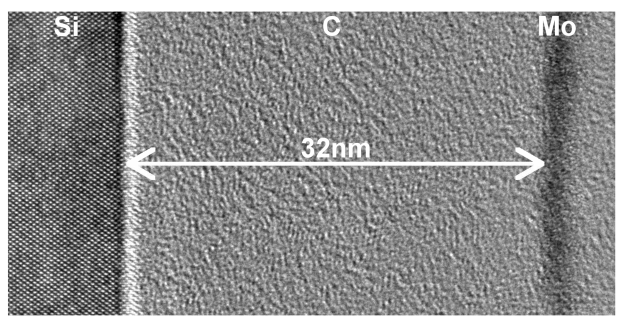

Fig. 4: Cross-section TEM sample. a-C film on Si substrate.

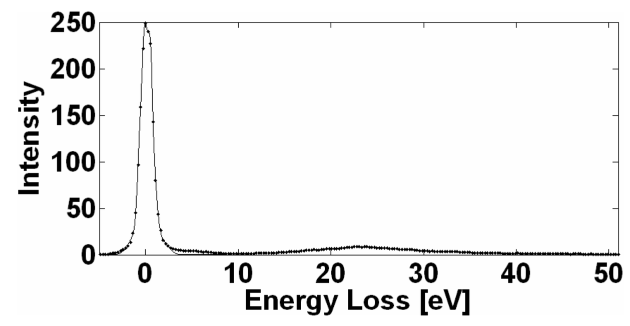

Fig. 5: Determination of a-C film thickness by EELS.

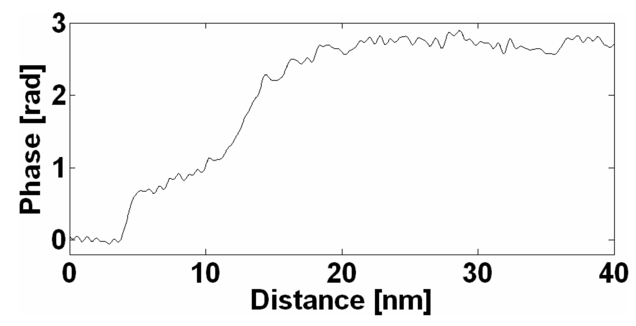

Fig. 6: Determination of phase shift by electron holography.

I. Application of the Hilbert phase plate:

- Cross-section TEM sample of Al_2O_3 and *C* layers deposited on a *Si* substrate using electron-beam evaporation.
- Comparison of conventional in-focus TEM image (Fig. 7(a)), phase-contrast image (Fig. 7(b)), HRTEM image (Fig. 7(c)) and reconstructed phase obtained by electron holography (Fig. 7(d)).

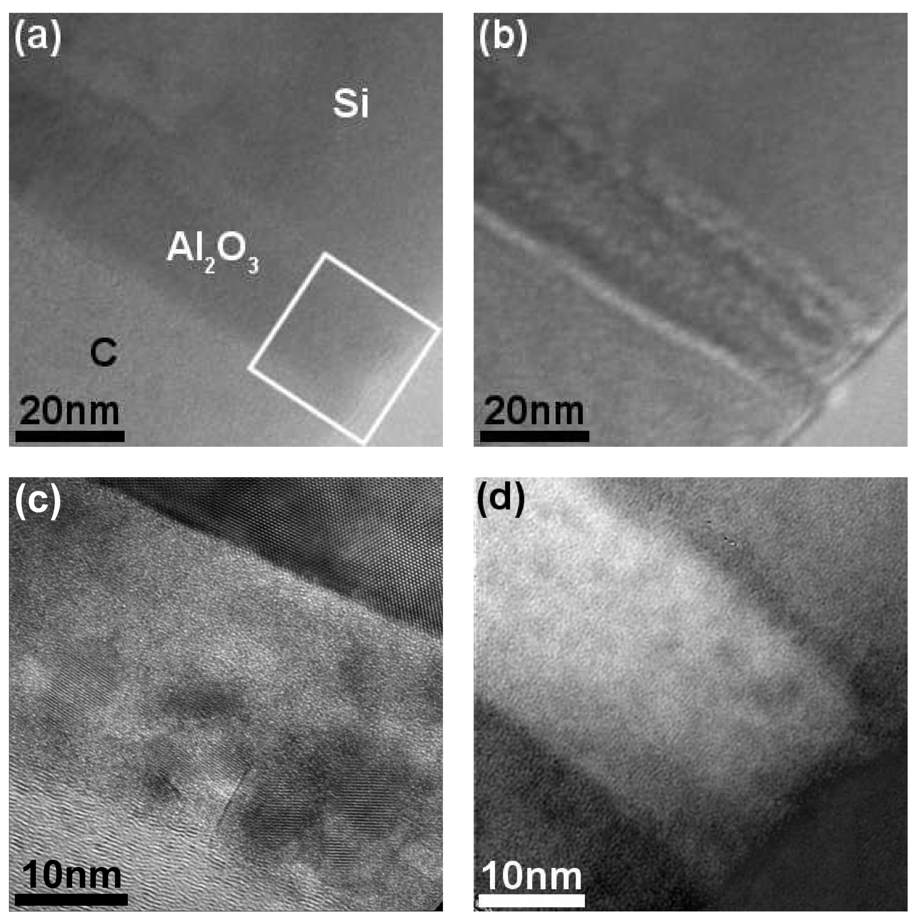

Fig. 7: (a) Conventional bright-field cross-section TEM image.
(b) Phase-contrast image using Hilbert phase plate. Slight astigmatism due to charging of the Hilbert phase plate.
Both images are taken close to Gaussian focus.
(c) HRTEM image and (d) reconstructed phase of a hologram of the region indicated by the frame in Fig. 7(a).

- Additional contrast features in phase-contrast image within the amorphous Al_2O_3 layer not visible in the conventional TEM image possibly related to nanoscaled crystallites (see HRTEM image Fig. 7(c)) and local density fluctuations (see reconstructed phase Fig. 7(d)).

II. Application of the Hilbert phase plate:

- TEM sample of amorphous Nb_2O_5.
- Application as dielectric in electrolyte capacitors.
- Comparison of conventional in-focus TEM (Fig. 8(a)) and phase-contrast image (Fig. 8(b)).

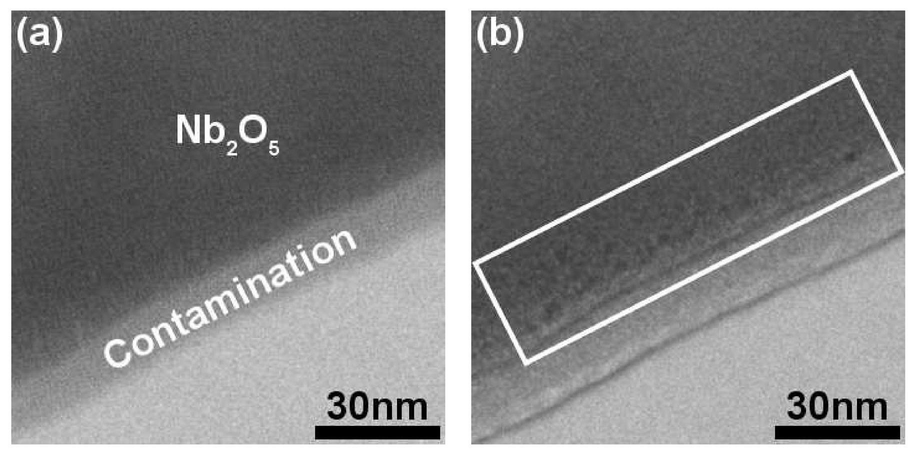

Fig. 8: (a) Conventional bright-field TEM image.
(b) Phase-contrast image using Hilbert phase plate.
Both images are taken close to Gaussian focus.

- Phase-contrast image shows structures (within the frame in Fig. 8(b)) not visible in the conventional TEM image possibly related to artifacts induced by ion milling.

Summary:

- Fabrication of a Hilbert phase plate by deposition of an a-C film using electron-beam evaporation and milling of suitable apertures by a FIB system.
- Characterization of Hilbert phase plates regarding the a-C film thickness and induced phase shift.
- Additional contrast features not present in conventional TEM images of amorphous Al_2O_3 and Nb_2O_5 are visible in in-focus Hilbert phase contrast images.
- Promising applications of phase plates not only for the investigation of biological samples but also in materials science.