Fabrication of a Boersch Phase Plate for Phase Contrast Imaging in a Transmission Electron Microscope

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Introduction

- Weak-phase objects like biological specimens show very low contrast in conventional TEM images.
- Splitting the critical maximum electron dose by taking defocus-series leads to a low signal-to-noise ratio of the images.
- Additional phase plate creates phase shift of 90° between scattered and unscattered electrons [1].

Phase plates

- Zernike phase plate in TEM [2]: Thin carbon film with small hole in the center placed in the back focal plane (BFP) of objective lens.
- Boersch phase plate [3]: phase shift of unscattered electrons by an electrostatic potential in a microscaled electrostatic lens.
- First experimental realization of Boersch phase plate [4].

Experimental Techniques

Fabrication of the Boersch phase plate

- Electron-beam lithography in a scanning electron microscope (SEM) Leo SUPRA 55VP with a Raith Elphy Plus pattern generator
- Electron-beam evaporation of Au and Al_2O_3
- Focused ion-beam (FIB) lithography with a Zeiss FIB EsB 1540 with a Raith Elphy Plus pattern generator

Experimental verification of the function

- Zeiss SESAM II Cryo 200 keV energy-filtering TEM (EFTEM)
- Positioning with piezodriven Kleindiek MM3A micromanipulator

Proposed Technological Realization

Weak lens as a constant phase-shifting device

Matsumoto & Tonomura [5]: uniform phase shift at low voltages.

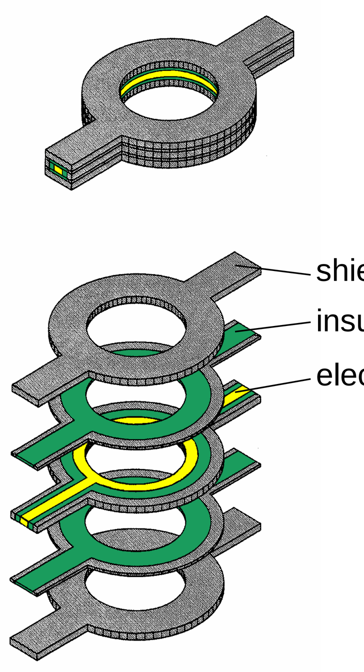

Figure 1

Phase-shift is proportional to the integrated voltage.

Modified design with three-fold symmetry (Fig. 3): Allows recovery of obstructed information by single sideband imaging according to [6].

References

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Fabrication of a Boersch Phase Plate

- Realization of an electrostatic microlens by a five-layered electrode structure in the center of the phase plate (Fig. 3 right-hand side).
- Confinement of the electrical field to the central lens opening by a surrounding Au layer.

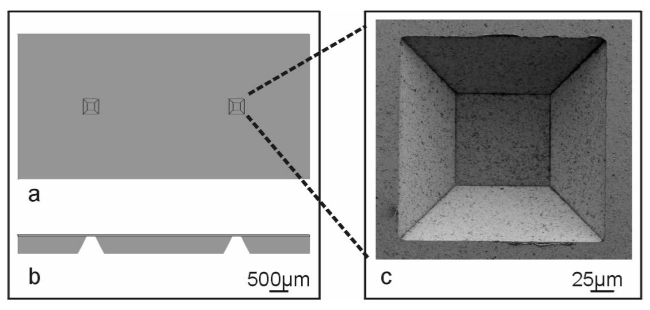


Figure 2

Basic substrate

- Commercially available low-stress $Si_{3+x}N_{4-x}$ membranes on Si chips (Fig. 2 and layer No. 4 in Fig. 3 right-hand side).

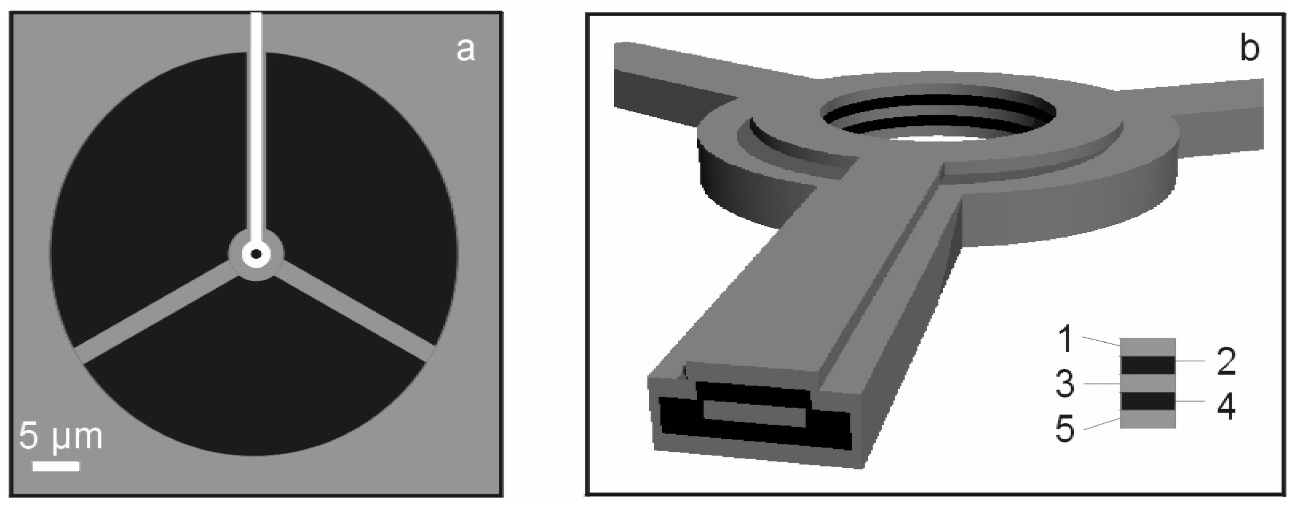


Figure 3

Step 1

Patterning of the electrode

- Electron-beam evaporation of the lower shielding Au layer on the $Si_{3+x}N_{4-x}$ membrane (layer No. 5, Fig.3 right-hand side).
- Patterning of the electrode layer by electron-beam lithography: Chip is coated with PMMA resist. Shape of the electrode, connecting leads and contact pad are defined with SEM. Fig. 4 a,b: light microscope image of the structure after development of the PMMA resist.

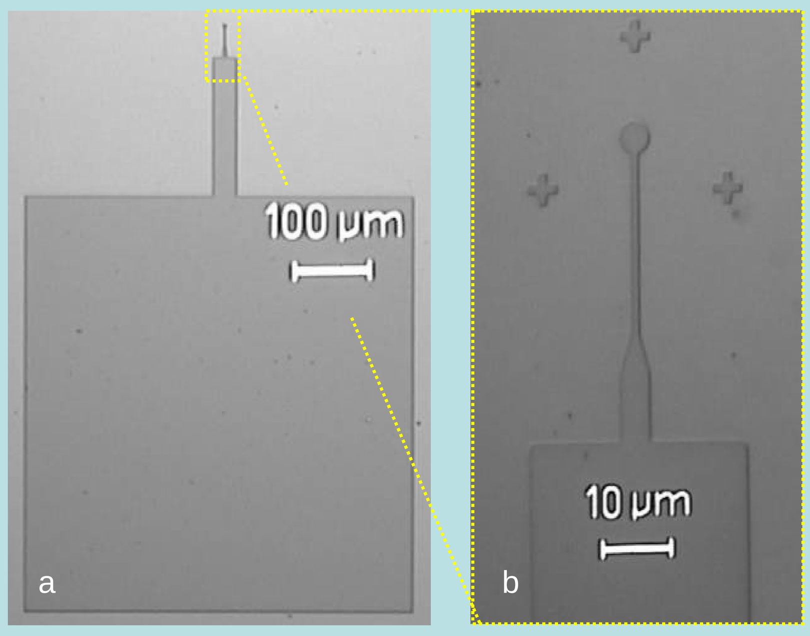


Figure 4

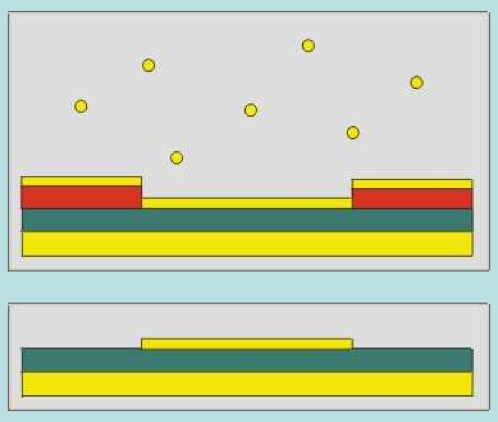


Figure 5

- Evaporation of Au layer (Fig. 3, No. 3) on PMMA pattern and lift-off process (only structure remains, see Fig. 5).

Step 2

Shaping of the phase plate

- Ion-beam lithography with Zeiss two-beam system (Fig. 8).
- Exact positioning by cross-markers (see Fig. 4b).
- Milling of the 3 sectors.
- First tested design: outer diameter d_o = 45 µm, bar width b = 3 µm, outer diameter of microlens d_m = 7 µm (Fig. 6).
- Improved design: d_o = 60 µm, b = 2 µm, d_m = 3 µm (Fig. 7).

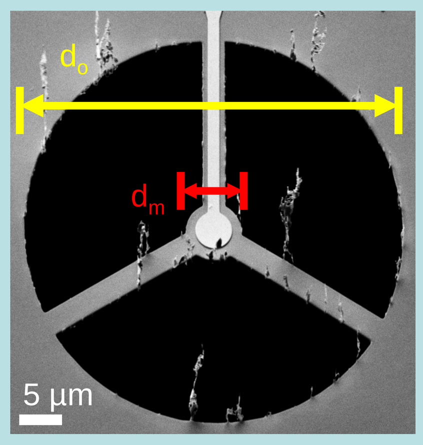


Figure 6

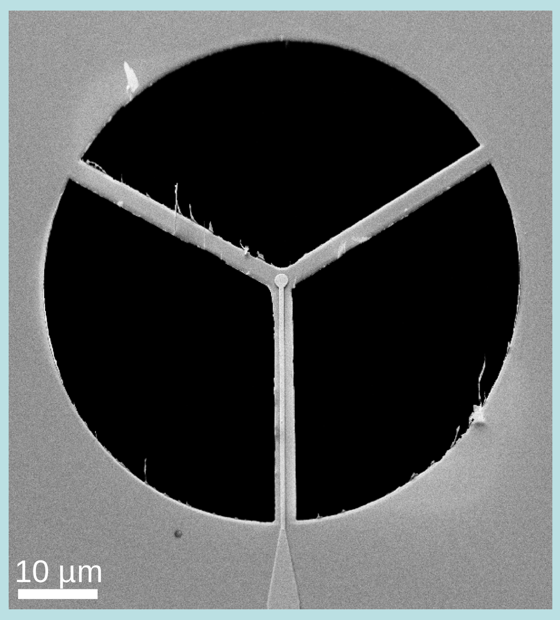


Figure 7

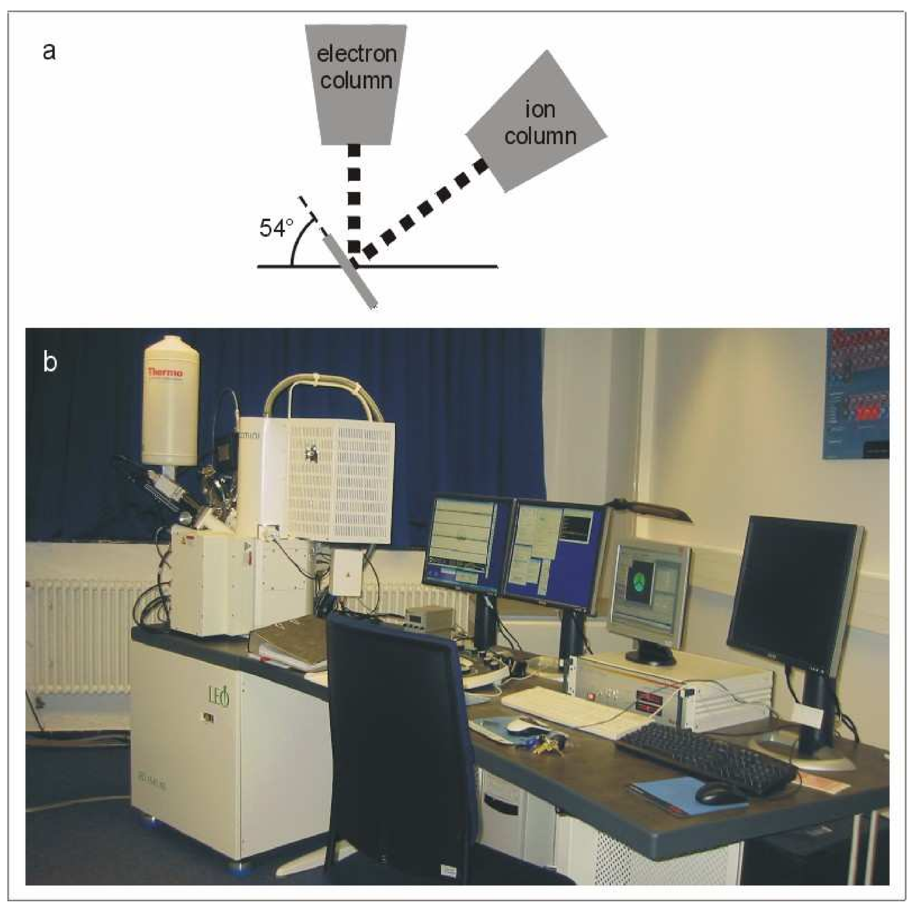


Figure 8

Zeiss FIB/SEM two-beam system with Raith Elphy Plus pattern generator (Fig. 8)

- Electron and ion column, oriented at an angle of 54° to each other.
- Working distance must be adjusted carefully: Chip in the crossing point of both beams

Step 3

Covering layers

- Evaporation of a 2nd insulating layer (Al_2O_3).
- Complete coverage of the structure by an Au layer: Special rotating holder that is tilted at 45°(Fig. 9): ⇒ side surfaces of the supporting bars are completely covered with shielding layer.

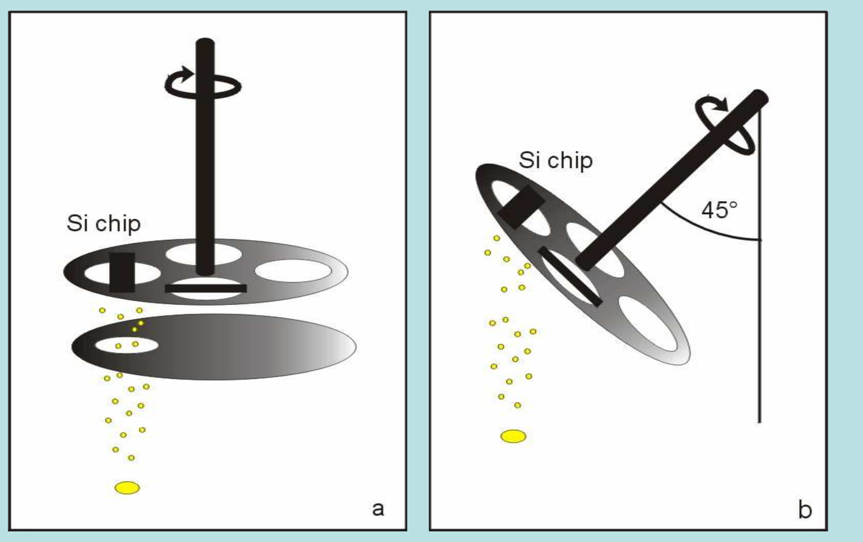


Figure 9

Step 4

FIB milling of the central lens opening

- The last step is the milling of the central lens opening with the FIB (Fig. 10).

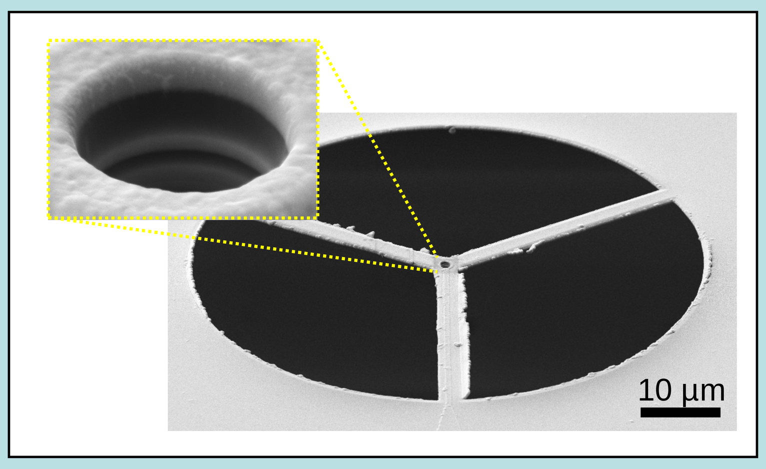
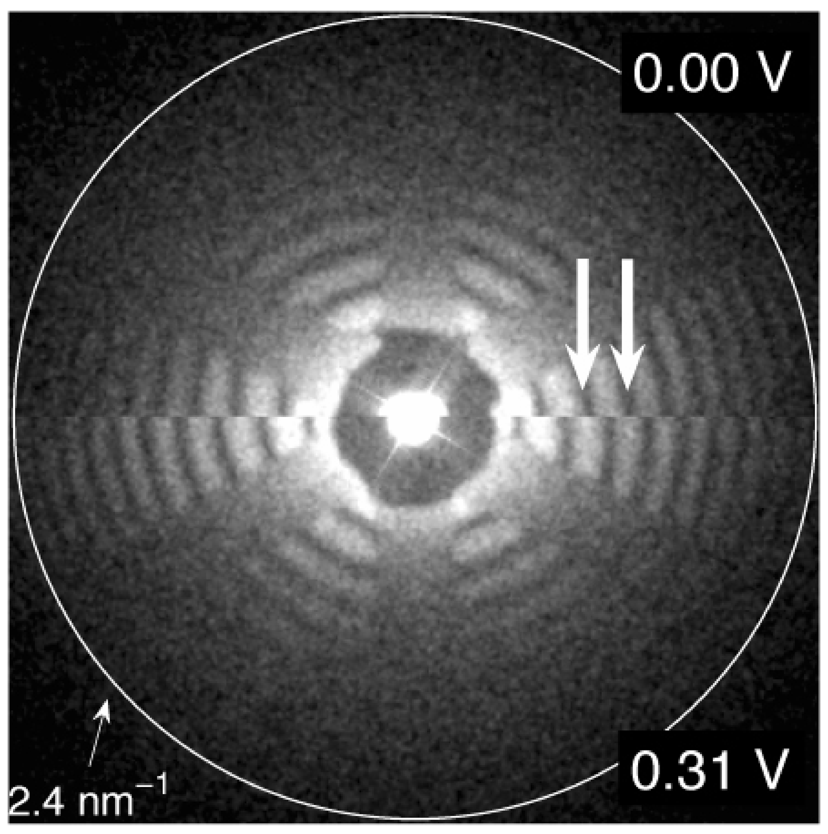


Figure 10

Proof of Phase Plate Concept



- Images of amorphous carbon films taken with different applied voltages.
- CTF maxima (Thon rings) in calculated frequency spectra compared.
- Constant phase shift of 90° achieved (Fig. 12) [4].

Figure 12

Implementation and Test of Phase Plate

- Si chip with 2 phase plates fixed on a special aluminum holder (Fig. 11).
- Contact pads connected with bondable terminals on the left by isolated wires fixed with conductive silver.
- Attachment of aluminium holder to a piezodriven micromanipulator with 3 motors for exact positioning in the BFP.
- Electrical bushing through a flange.

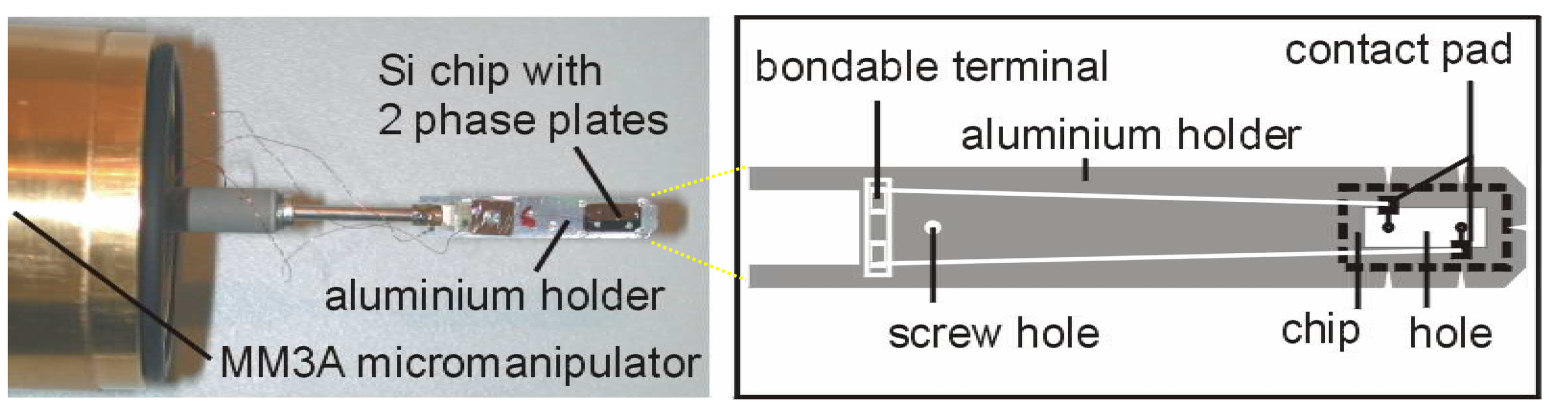


Figure 11

Conclusions

- Fabrication process suitable for the fabrication of microscaled electrostatic lenses.
- Realization of first Boersch phase plate and its implementation in a transmission electron microscope.
- Phase shift up to 90° of unscattered electrons achieved with respect to scattered electrons.
- Further reduction of phase plate dimensions or magnification of the diffraction plane of the objective lens advantageous for future applications.
- In combination with aberration correction: tuning to adjust spherical aberration, defocus and relative phase shift in such a way as to obtain perfect phase contrast transfer for a large range of spatial frequencies up to 1 / 0.1 nm^{-1}.