Karlsruhe Institute of Technology

Laboratory for Electron Microscopy

Optimized Fabrication and Application of Electrostatic Phase Plates for Transmission Electron Microscopy

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Introduction

- Electrostatic physical phase plates (PP) enhance the contrast of weak-phase objects in transmission electron microscopy (TEM)
- An electrode, surrounded by insulating and metallic shielding layers, generates an electrostatic field close to the zero-order beam. (Fig.1) Depending on the applied voltage, a relative phase shift between scattered and unscattered electrons is induced.
  - **Boersch-PP**[1]: Three supporting rods and an inner ring lens guarantee a homogeneous electrostatic field but obstruct information at low spatial frequencies (Fig. 2a). Cut-on frequency is determined by outer ring diameter.
  - **Zach-PP**[2]: With only one supporting rod, obstruction of spatial frequencies is significantly reduced (Fig. 2b). Cut-on frequency is determined by the shape of the electrostatic field.

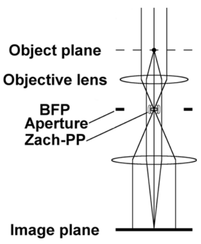

Fig. 1: Schematic illustration of Zach-PP located in the back-focal plane (BFP) of the objective lens.

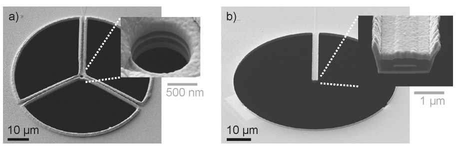

Fig. 2: Scanning electron microscopy (SEM) images reveal layer system with gold cover layer, insulating layers and gold electrode. a) Boersch-PP with three and b) Zach-PP with one supporting rod.

Motivation

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- Electrostatic charging limits application of PPs
- Low spatial frequencies cannot be resolved with present PP design
  - ➡ ratio between size of diffraction pattern and PP dimension has to be enlarged by increasing the focal length and scaling down the PP sizes
  - ➡ need for small tip, large aperture radius and thin layers

Fabrication of Zach-PP

- Seven production steps to produce a Zach-PP (Fig. 3) including electron-beam evaporation (EBE), electron-beam lithography (EBL), focused ion beam (FIB) and reactive ion etching (RIE).

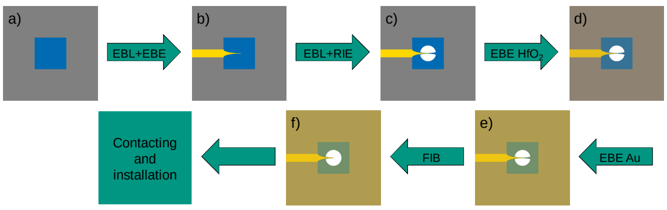
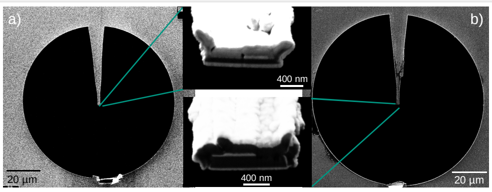


Fig. 3: Production scheme of Zach-PP. a) Si with 100nm $Si_{3+x}N_{4-x}$-membrane b) Gold electrode c) etched aperture hole d) insulating HfO_2 layer e) shielding Au layers f) FIB cutting of tip.

- RIE simplifies process and allows production of PPs with larger aperture radius and narrower tips due to high precision of EBL (Fig. 4).
- The high ε of HfO_2 guarantees good isolation in spite of thinner layers.

Fig. 4: FIB images of Zach-PPs. Tip width < 1 µm and aperture diameter > 160 µm possible. SEM detail images reveal layer system.
a) HfO_2 layer with reduced roughness
b) PP with Al_2O_3 layer



PP heating

- To reduce contamination and thus electrostatic charging, PP is heated by a microstructured heater (Fig. 5)
  - Fabricated with EBL and EBE
  - Heating power of 90 mW at 30 V

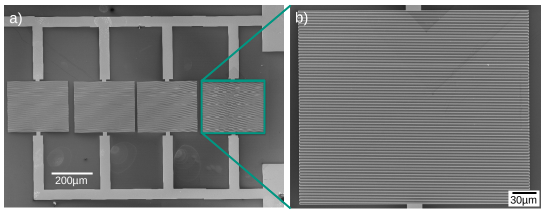


Fig. 5: SEM images of a microstructured gold heater.
a) Four parallel heaters with total resistivity of 4 kΩ
b) Line width and distance of 1 µm result in total length of ~ 4 cm.

- Application of a constant voltage ➡ resistivity and current drop with increasing temperature allowing temperature measurements (Fig. 6)
  - Thermal stability is reached within one hour
  - Temperatures > 50°C possible

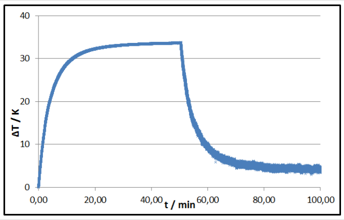
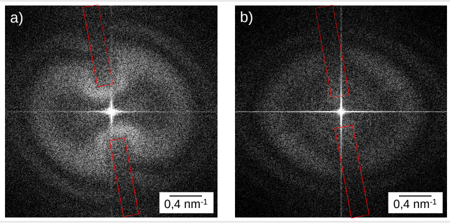


Fig. 6: Plot of temperature change over time with an applied voltage of
30 V (t < 50 min) and
0.1 V (t > 50 min).
PP holder is thermally isolated to the microscope column to minimize heat flow. Thermal expansion of several ten microns has to be corrected after reaching the equilibrium state but then stays constant.

- Higher PP temperature reduces contamination and electrical charging visible in power spectra (Fig. 7)

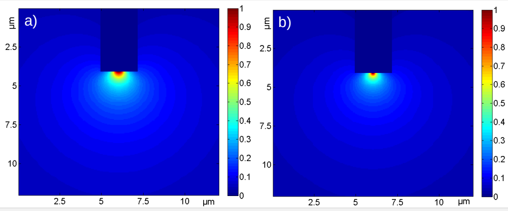
Fig. 7: Power spectra of an amorphous carbon film with Zach-PP indicated by red lines. Defocus and applied voltage of 0.5 V are constant in both images
a) Before heating: Distortion of Thon rings clearly visible.
b) After heating: No distortion visible.



Field simulations

- Simulations of electrostatic potential of Zach-PP provide better knowledge of phase-shifting characteristics (Fig. 8)

Fig. 8: Normalized simulated phase shift in units of π for electrons in TEM at 200 kV in 12x12 µm area around tip for electrode width of
a) 500 nm
b) 100 nm



Summary

- Optimized fabrication with narrower PP design and enlarged aperterure radius
- PP heating and thinner insulating layers significantly reduce electrostatic charging
- Potential simulations provide better knowledge of phase shift

References

[1] K. Schultheiß et al., Rev. Sci. Instrum. **77** (2006) 033701
[2] K. Schultheiß et al., Microsc. Microanal. **16** (2010) 785-794

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