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Laboratory for Electron Microscopy

A Nanocrystalline Hilbert Phase-Plate for Phase-Contrast Transmission Electron Microscopy

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Introduction

- Thin film-based Hilbert phase-plates (PP) enhance the contrast of weak-phase objects in transmission electron microscopy (TEM)[1]:

  Half of the diffraction pattern is covered by a thin film excluding the zero-order beam (Fig. 1). Depending on the film thickness, a phase shift is imposed on electrons passing the Hilbert PP.
- Application limited by electrostatic charging of amorphous-C film-based Hilbert PPs[2] (Fig. 2)
- Enhanced electrical conductivity of crystalline metal film-based Hilbert PPs

➡ **Investigation of the effect of crystalline PP-structures on the PP-properties and the image formation process**

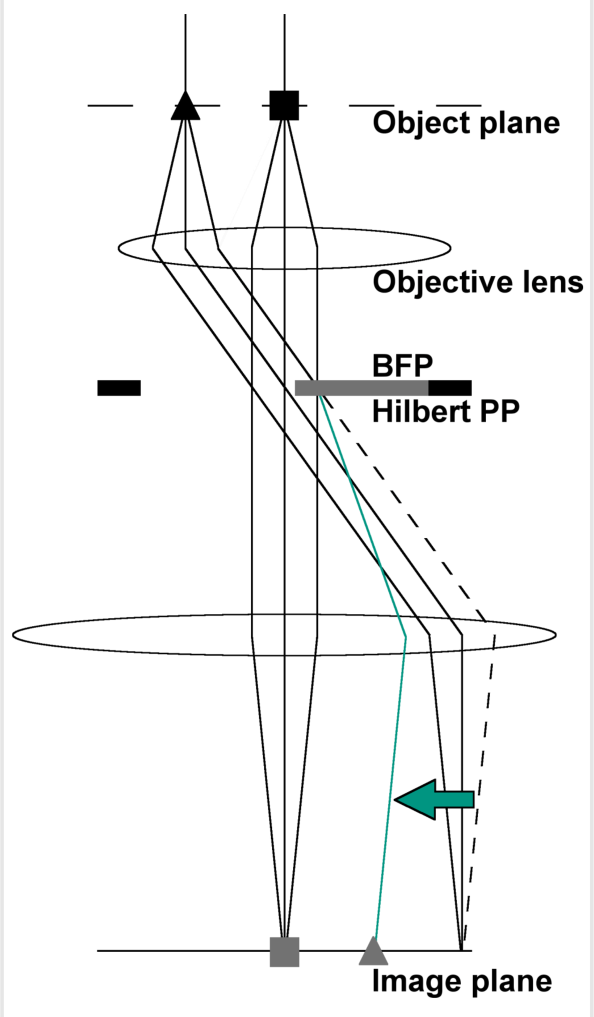

Fig. 1: Schematic illustration of a crystalline Hilbert PP located in the back focal plane (BFP) of the objective lens. Shift of image information in the image plane caused by the beams diffracted by the crystalline PP-structure (green optical path).

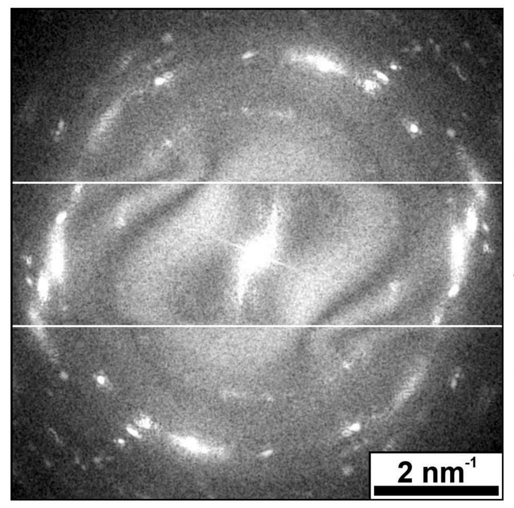

Fig. 2: Power spectrum of a Hilbert phase-contrast image. Distortion by electrostatic charging of the amorphous-C film-based Hilbert PP. Edge of the PP-structure indicated by white lines.

Fundamental considerations on image simulation

- Sample area separated into a central patch (■, cf. Fig. 1) and distant patches (▲)
- Image intensity of the central patch given by a superposition:
  - *Primary* image intensity: Beams leaving the central patch, not diffracted by the PP-structure and not shifted in space (■)
  - *Shadow* image intensity: Beams originating from distant patches, diffracted by the PP-structure and shifted in space (▲)

Image simulation for a monocrystalline Hilbert PP

- Pt-nanoparticle sample on amorphous-C support film
- Monocrystalline Au film-based Hilbert PP of 40 nm thickness in [001] orientation
  - Bragg reflections ⬌ Shift of image information along discrete directions/ Limited number of contributing distant patches

  ➡ Superposition of the *primary* image of the central patch (Fig. 3b) and the *shadow* images of nanoparticles located in the distant patches (Fig. 3c)
  - Homogeneous orientation ⬌ Homogeneous phase shift of π (Fig. 3a, inset)

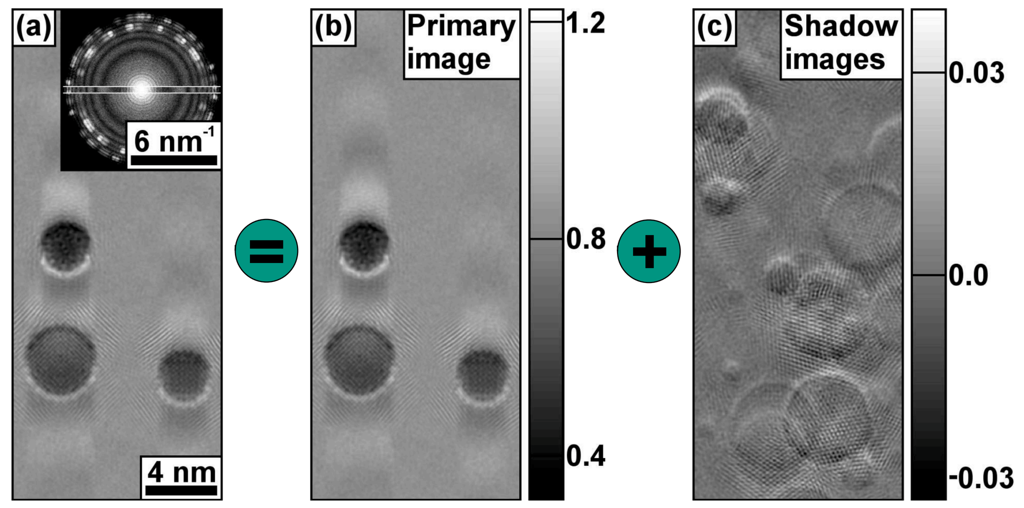

Fig. 3: Simulation of a monocrystalline Hilbert PP. Calculations performed for a Philips CM200 FEG/ST assuming a cut-on frequency of 0.2 nm^{-1}.
(a) In-focus phase-contrast image and corresponding power spectrum (inset).
(b) Primary image intensity.
(c) Shadow image intensity. Note the different grayscale bars.

Image simulation for a nanocrystalline Hilbert PP

- Textured PP-structure: 50% [001]-Au grains, 50% random orientation
  - Debye reflection rings ⬌ Radially isotropic shift of image information/ Numerous contributing distant patches

  ➡ Diffuse background formation (Fig. 4c)

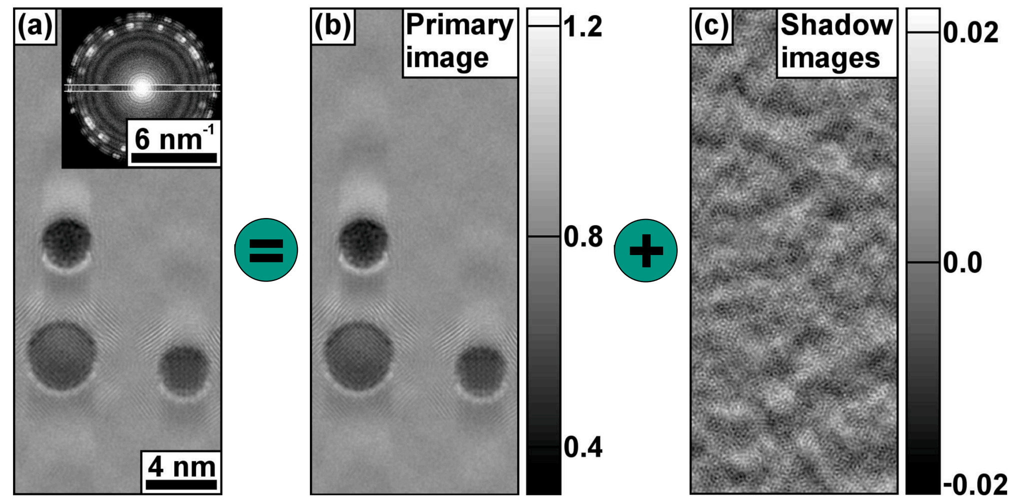

Fig. 4: Simulation of a textured nanocrystalline Hilbert PP.
(a) In-focus phase-contrast image and corresponding power spectrum.
(b) Primary image intensity.
(c) Shadow image intensity which forms a diffuse background.

- Inhomogeneous orientation ⬌ *Phase noise* affecting the coherence (Fig. 5a)

➡ Attenuation of the contrast transfer function (CTF) for weak textures (Fig. 5b)

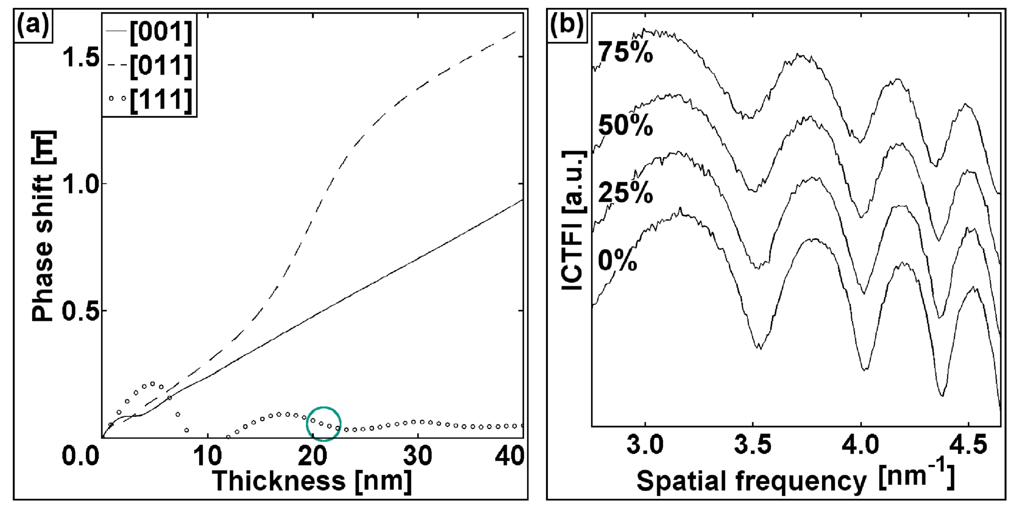

Fig. 5: Effect of Au grains in random orientation.
(a) Phase shift imposed on electrons passing Au grains in [001], [011] and [111] directions without being diffracted.
(b) Attenuation of the CTF by the *phase noise* induced by an increasing number of Au grains (0% – 75%) in random orientation.

Fabrication of nanocrystalline Hilbert PPs

- Electron-beam evaporation of Au on tenside-treated mica substrates
- Floating process on Cu-grids
- Cleaning by air plasma
- Focused ion-beam structuring of rectangular windows (Fig. 6)
- Mounting on customized objective aperture stripes
- Implementation in the BFP of a Philips CM200 FEG/ST

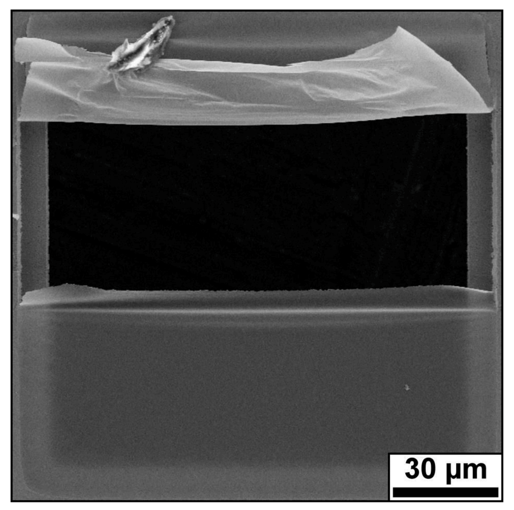

Fig. 6: Scanning electron microscopy image of a Hilbert PP in one mesh of the Cu-grid.

Characterization of the nanocrystalline PP-structure

- Au grains arranged in a porous film of 21 ± 2 nm thickness (Fig. 7a, b)
- [111]-textured PP-structure indicated by strong {022}-Debye ring (Fig. 7c)

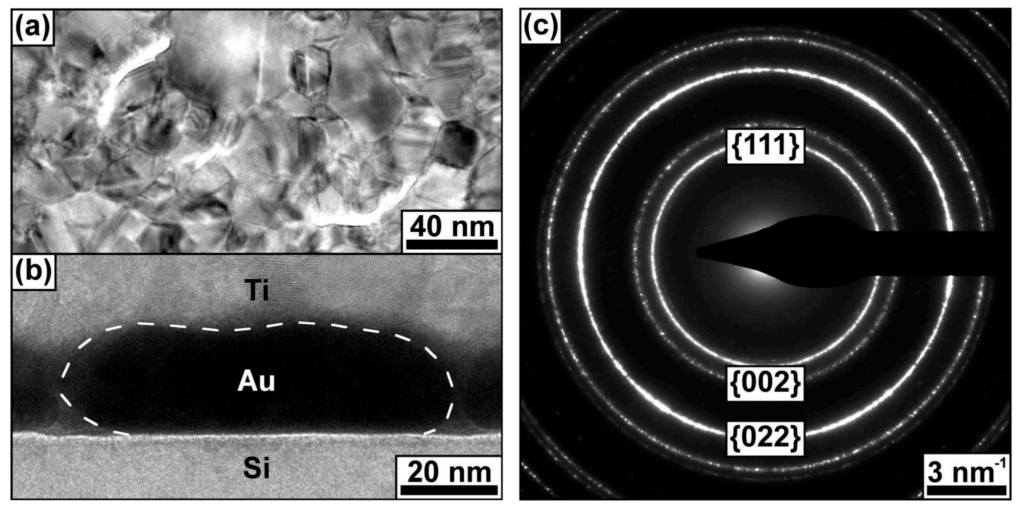

Fig. 7: Characterization of the nanocrystalline PP-structure.
(a) Plan-view TEM image. Au grains of 20 nm to 50 nm size.
(b) Cross-section TEM image of the Au film simultaneously deposited on Si substrates.
(c) Plan-view Debye-Scherrer diffraction pattern of the Au film floated from mica substrates.

Application on Pt-nanoparticle sample

- Behavior expected for a [111]-textured nanocrystalline Au film-based Hilbert PP:

➡ Vanishing phase shift (Fig. 8c, cf. Fig. 5a, green circle)

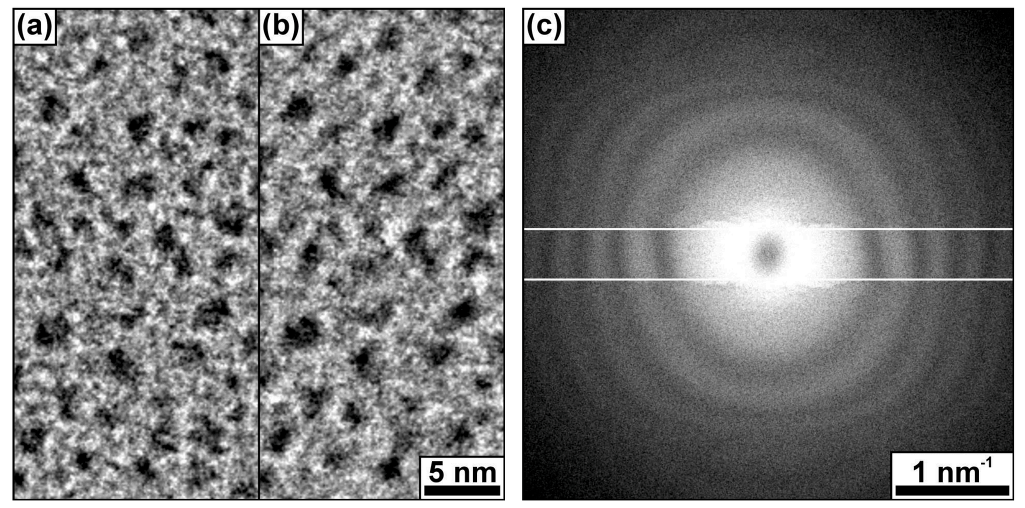

Fig. 8: Application of the textured nanocrystalline Hilbert PP. Pt-nanoparticle sample on amorphous-C support film.
(a) Conventional TEM image.
(b) Phase-contrast image of identical sample area.
(c) Power spectrum of the phase-contrast image.

Acknowledgement

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References

[1] R. Danev et al., J. Phys. Soc. Jpn. 73 (2004) 2718-2724
[2] R. Danev et al., Ultramicroscopy 109 (2009) 312-325

Summary

- Monocrystalline/textured nanocrystalline PP-structures of appropriate thickness and orientation are suitable for phase-contrast imaging
- Tradeoff required between *shadow* images for monocrystalline PP-structures and reduced coherence for textured nanocrystalline PP-structures

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