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

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In the past few years, physical phase-plates (PP) have emerged as an interesting tool to achieve phase-contrast in transmission electron microscopy (TEM). Contrast enhancement of weak-phase objects has been demonstrated for various electrostatic and thin film-based PP concepts. This work considers Hilbert PPs, which consist of a microstructured amorphous C (aC)-film positioned in the back focal-plane (BFP) of the objective lens [1]. Depending on the film thickness, a phase shift is imposed on the electrons in one half of the diffraction pattern, while the zero-order beam and the second half of the diffraction pattern remain unaffected. The induced phase shift affects the phase-contrast transfer function and yields contrast enhancement at low and intermediate spatial frequencies.

The lifetime of a Hilbert PP is limited by a steady irreversible degeneration of the aC-film. Subsequent electrostatic charging induces artifacts and aberrations, which are difficult to correct. However, up to now, the application of metal films with a higher electrical conductivity and material stability was avoided due to their nanocrystalline structure. This work, for the first time, considers the effect of crystallinity on the PP properties and the image formation process.

The effect of a crystalline Hilbert PP is schematically illustrated in Figure 1a. The object plane is separated into a central patch (■) centered on the optical axis and distant patches (▲) off the optical axis. If the image intensity of the central patch is considered, the "primary" image intensity is given by the beams leaving the central patch and passing the BFP without being diffracted (green optical path). Moreover, electrons incident on distant patches, which are diffracted by the PP microstructure, cause a shift of image information in the image plane (red optical path). Hence, shifted "shadow" images of the distant patches are superimposed on the "primary" image intensity of the central patch. Assuming that most of the electrons pass the BFP without being diffracted, the "primary" image intensity becomes predominant, while only a negligible contribution is added by the "shadow" images. Hence, the phase shift is given by the phase imposed on electrons, which are not diffracted by the PP microstructure. In Figure 1b, the phase shift induced by a Au-film is derived by Bloch-wave calculations [2]. Due to dynamic electron diffraction, the phase shift depends on both, the film thickness and the crystalline orientation of the Au-film.

Experimental verification is obtained by the application of a Hilbert PP fabricated from a textured nanocrystalline Au-film. The Au-film is deposited by electron-beam evaporation on cleaved mica surfaces and floated on Cu-grids. Plan-view TEM samples of the Au-film reveal a porous arrangement of grains as illustrated in Figure 2a. Cross-section TEM samples prepared from a simultaneously coated Si-substrate yield a film thickness of 21 (± 2) nm as shown in Figure 2b. Electron backscatter diffraction (EBSD) measurements are performed in a scanning electron microscope to determine the crystalline orientation of individual grains. Most of the grains are aligned close to the [111] zone-axis orientation. Hence, a strong [111] texture is indicated by the [001] pole-figure as illustrated in Figure 2c. Using a focused ion-beam system, rectangular windows are structured into the Au-film providing a Hilbert PP in several meshes. Power spectra of phase-contrast images of an aC-film positioned in the object plane reveal the PP properties as shown in Figure 2d. The distance between the edge of the Au-film and the zero-order beam is marked by vertical white lines. A vanishing phase shift induced by the Au-film is indicated by the Thon-rings, which are almost continuously connected across the vertical white lines. This is in good agreement with the Bloch-wave calculations in Figure 1b, where the dotted curve oscillates close to a phase shift of zero in a wide range of the film thickness [3].

References:

[1] R Danev and K Nagayama, J. Phys. Soc. Jpn. **73** (2004), p. 2718. [2] K Muller *etal*, Ultramicroscopy **109** (2009), p. 802.
[3] Acknowledgement: Financial support by Deutsche Forschungsgemeinschaft (DFG) under contract Ge 841/16.

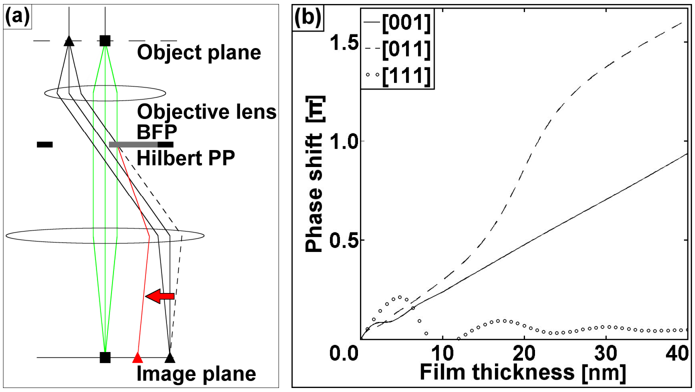

Figure 1. Effect of a crystalline Hilbert PP in phase-contrast TEM. (a) Diffraction of the outer right beam of the distant patch causes a shift of image information (red arrow) and "shadow" images (▲) in the image plane. (b) Phase shift induced by a Au-film in dependence of the film thickness for three representative crystalline orientations along the [001], [011] and [111] direction.

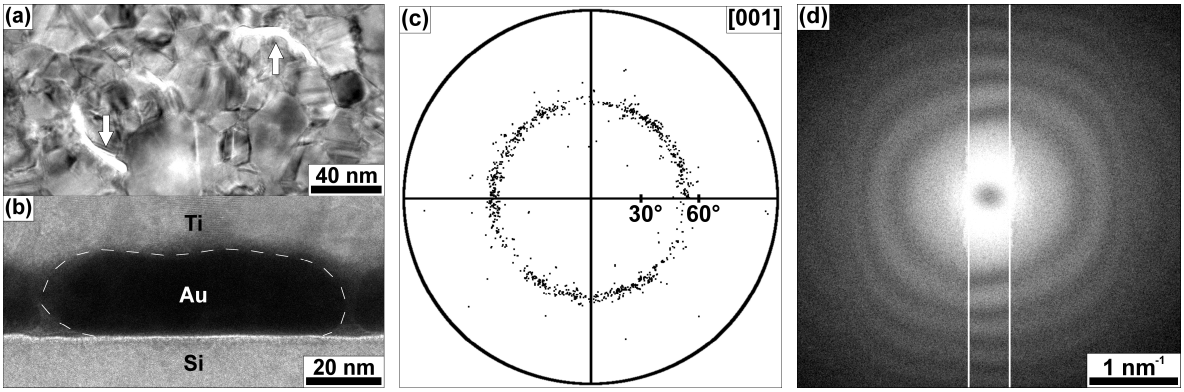

Figure 2. Characterization of the textured nanocrystalline Au-film. (a) Plan-view TEM image of grains between 20 nm and 50 nm in size and pores marked by white arrows. (b) Cross-section TEM image with a grain surrounded by the dashed white line. (c) [001] pole-figure obtained by EBSD measurements indicating a strong [111] texture given by a ring at an angle of 55°. (d) Power spectrum of a phase-contrast image of an aC-film.