Investigation of parameters that influence the performance of a hole-free phase plate and its application on a carbon nanotube sample

Pretzsch, R.[1], Dries, M.[2], Hettler, S.[3], Obermair, M.[4] and Gerthsen, D.[2]

[1] Karlsruhe Institute of Technology - Laboratory for Electron Microscopy, Germany, [2] Laboratory for Electron Microscopy - Karlsruhe Institute of Technology, Germany, [3] KIT - Laboratory for Electron Microscopy, Germany, [4] Laboratory for Electron Microscopy, Karlsruhe Insitute of Technology, Germany

In recent years, efforts were undertaken to improve the contrast of weak-phase objects in transmission electron microscopy (TEM). The implementation of a physical phase plate (PP) in the back focal plane of the objective lens allows to introduce a phase shift between the scattered and unscattered electrons and hence to augment phase contrast transfer of small spatial frequencies. Several PP concepts have been realized [1]. A promising alternative concept is the hole-free PP (HFPP) where the intense zero-order beam (ZOB) forms a charged patch on a thin amorphous carbon film [2,3] which can be explained by surface-related phenomena [4].

The formation of the charged patch depends on experimental conditions, which makes it difficult to control the induced phase shift. We conducted HFPP experiments in a Philips CM200 FEG/ST in dependence of the HFPP temperature and the diameter of the condenser aperture. The diameter of the condenser aperture and thus of the ZOB has a major impact on the induced phase shift. A small ZOB (< 200 nm) generates a positive phase shift due to the build-up of contamination on the unheated HFPP [2]. Heating of the HFPP to 150 °C prevents contamination and leads to a negative phase shift, as expected from [3]. Measurements for larger ZOBs (> 300 nm) show negative phase shifts of several π even for unheated HFPPs. Contamination only builds up at the edge of the ZOB. The observed increase in phase shift with the size of the charged patch is in accordance with the theoretical model in [4].

Bundles of carbon nanotubes (CNTs) on a holey carbon film were investigated with HFPPs using a small condenser aperture. Figure 1 shows TEM images close to focus, close to Scherzer defocus and a HFPP image taken with a heated HFPP. The contrast increase is clearly visible. For measurements without heating of the HFPP, positive and negative phase contrast was observed (Figure 2) since the contamination induces a phase shift of π/2 and 3π/2 depending on its thickness. Simulations and their comparison with measurements suggest that the visible "halo" around the CNT bundles is induced by the spatially extended phase shift distribution from the phase-shift generating patch.

References:
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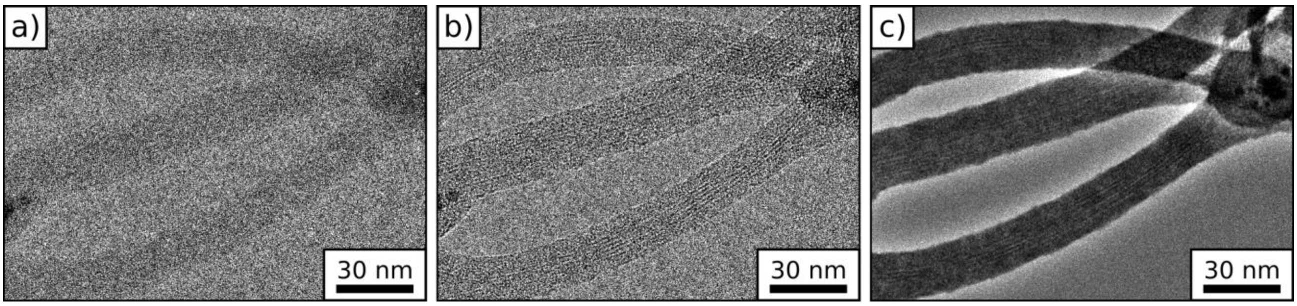

Figure 1. TEM images of CNT bundles with a heated HFPP. a) Without PP and close to focus. b) Without PP and close to Scherzer defocus. c) Phase-contrast image taken with a heated HFPP (150 °C) close to focus shows contrast increase resulting from the formation of a negative charge.

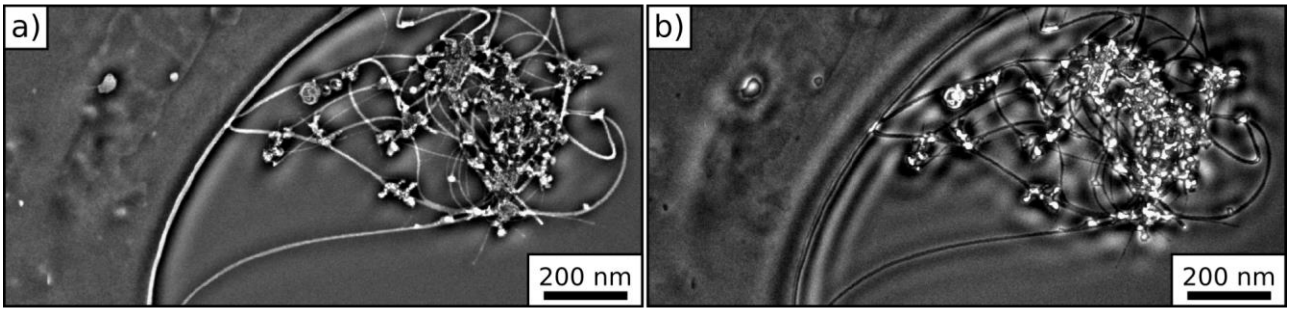

Figure 2. TEM images of CNT bundles acquired with an unheated HFPP. a) Directly after insertion of the HFPP, a positive phase shift of π/2 arises due to contamination growth. b) After approximately 20 seconds, increasing contamination leads to contrast inversion.