

Progress toward quantitative hole-free phase plate imaging in a TEM.

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The use of a uniform thin film locally charged by the imaging electron beam itself represents a possible implementation of the phase plate concept [Ultramicroscopy 118 (2012) 77]. Since the microscopic mechanisms behind the functionality of this "hole-free" phase plate (HFPP) are yet to be properly understood [Ultramicroscopy 184 (2018) 252, Micron 96 (2017) 38, Micron 100 (2017) 10, PNAS 111 (2014) 15635], a quantitative interpretation of the resulting image contrast has not been possible so far.

Using a FIB-fabricated phase grating sample, we were able to separate and measure the phase shift added by the HFPP to the direct and diffracted beams, measure the beam current density distribution at the HFPP and observe the image contrast evolution as function of time. The grating was prepared as sinusoidal grooves with 98 nm periodicity in a 13 nm thick amorphous Si membrane. The data were collected with a Hitachi HF-3300S TEM operated at 300 kV. The optics was chosen so that the separation between the direct and each of the primary diffracted beams in the HFPP plane was ~ 1.5 μm .

Computer simulations were performed assuming that the rate of increase of the phase shift at the HFPP is proportional to $\phi(t) = A[1 - \text{Exp}(-t/\tau)]$ for both direct and diffracted beams, although with different A 's and τ 's. The contrast is proportional to the phase shift difference between the direct and diffracted beams and reverses at $\phi = n^*\pi$, with n integer. When n is even, contrast vanishes as the phase relationship between direct and diffracted beams remains unvaried, while if n is odd, frequency doubling of the fringe pattern is expected.

Fig 1a shows an experimental time evolution of the image intensity obtained by combining 400 pixel wide intensity profiles at different times along the horizontal time axis. The dark region on the left of Fig. 1a corresponds to the blanked beam at the beginning of the experiment. Fig. 1b shows the simulated intensity evolution where the magnitude of the diffracted vs. direct beam phase shifts were chosen in proportion 1/3 i.e. A_{diff} tends toward 0.33x the magnitude of the direct beam A_0 phase shift, as consistent with direct measurements by electron holography (not shown). The settling time constants of the diffracted and direct beams were chosen as 300 s and 200 s, respectively. Experimentally, the beam current at the diffracted beams was ~ 200 x lower than at the direct beam. The fact that the settling rate of the diffracted beam appears only ~ 1.5 x slower than the diffracted beam suggests that the phase shift developing at the HFPP is not entirely controlled by the local beam current density and may involve several processes. However, the simulations reproduce the salient features appearing in the experimental data. In particular, the contrast reversal and frequency doubling of the contrast reversal region are seen in both the experiment and the simulations. The settling time constant values, while matching the observed image, deserve further investigation.

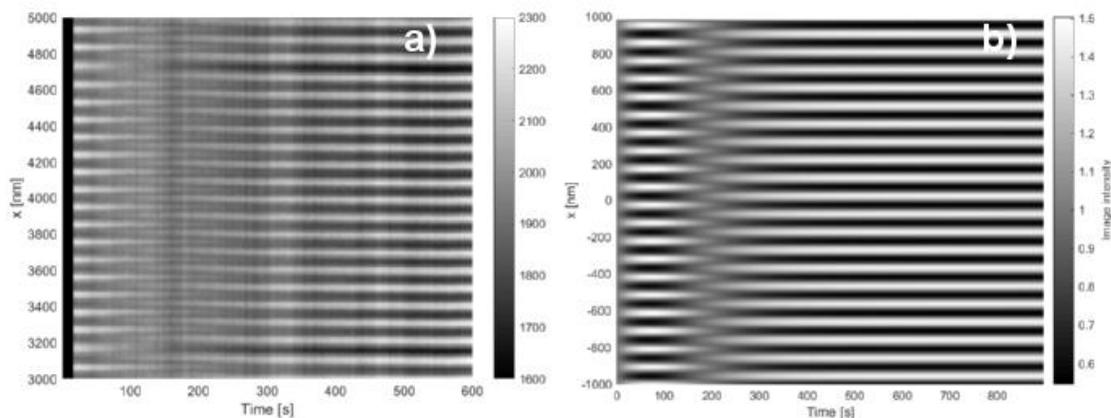


Figure 1 a) Experimental contrast profile evolution of a phase grating b) Simulation with the diffracted beam settling ~ 1.5 x slower than direct beam with final state phase shift at diffracted beam that is 0.33x the phase shift of the direct beam.