Characterization of Nanofabricated Electron Transmission Gratings with Electron Diffraction

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Nanofabricated phase-shifting devices for electron optics are of interest for a variety of applications. Periodically structured amplitude and phase gratings can be used as diffractive electron beam splitters in electron interferometers[1]. Intentionally introduced detects and aperiodicity in the gratings can be used to generate non-Gaussian electron beams, such as electron vortex beams, which hold promise for novel imaging and spectroscopy techniques[2]. Zernike phase plates in a transmission electron microscope (TEM) can provide in-focus phase contrast and improve the performance of cryo-electron microscopy of biological specimens[3]. It is crucial to understand the electron beam phase shifts imposed by these phase-shifting devices. Here we report a nanofabricated two-dimensional mesh transmission grating for electron beams and its characterization with electron diffraction in a TEM with various electron energies. Nanofabricated electron transmission gratings can be used as diffractive electron beam splitters, of which the splitting ratio is tunable by material-imposed electron phase-shift.

The electron transmission gratings were fabricated from silicon nitride membrane (5-10 nm) TEM grids (*SiMPore Inc.*). High-resolution electron beam lithography (EBL) was used to define the grating pattern. After resist development, pattern transfer to the silicon nitride membrane was achieved with reactive ion etching (RIE). Finally, the silicon nitride gratings were coated with an approximately 10 nm thick metal (gold or aluminum) layer to prevent charging in the experiment. Nano-gratings were characterized in a FEI Tecnai TEM in selected-area diffraction mode (Figure 1(a)). The electron energy was varied from 120 keV to 40 keV so that the membrane imposed different phase shifts to the electron beam, hence modulating the intensities of diffracted beams. By fitting experimentally measured diffraction beams' intensities to theoretical calculations, the electron beam phase shifts can be estimated. It was found the membrane imposed the same phase shift as if it was a 40 nm thick membrane made of a material with 20 V mean inner potential. The phase shift was found to be 2.2-3.5π for electrons with 120-40 keV energy. We also found relatively high-angle electron diffraction from polycrystalline metals affected the diffraction efficiencies of nano-gratings.

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[1] B. McMorran, J. Perreault, T. Savas, A. Cronin, Ultramicroscopy **106**, 356 (2006).
[2] T. Harvey, J. Pierce, A. Agrawal, P. Ercius, M. Linck, B. McMorran, New J. Phys. **16**, 093039 (2014).
[3] R. Glaeser, Nat. Meth. **13**, 28 (2016).

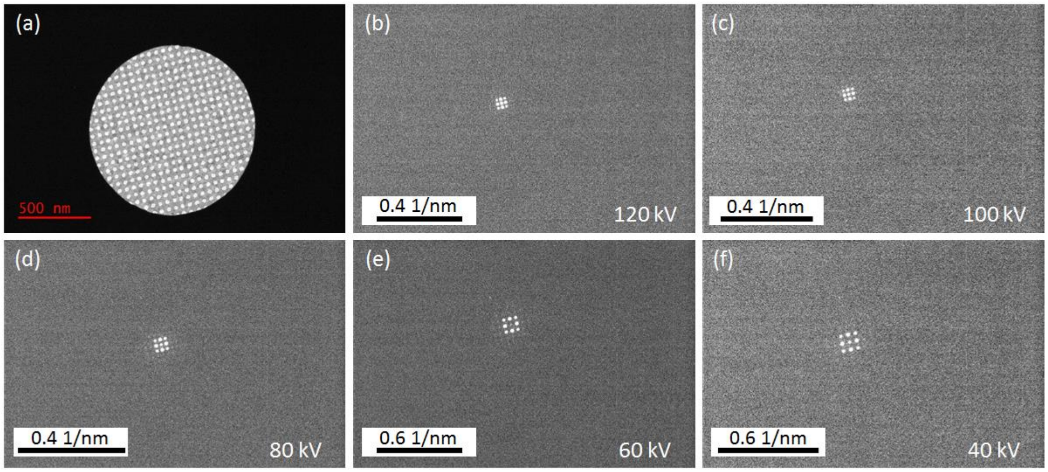

Figure 1: Selected area TEM image and electron diffraction patterns of a nanofabricated transmission grating: (a) A two-dimensional mesh grating patterned from a 10 nm thick silicon nitride membrane by EBL and RIE, followed by 10 nm gold coating to prevent charging during imaging and diffraction. The grating pitch is 50 nm. A selected area aperture is inserted to select a circular area with ~ 1 μm diameter. *(b-f)* Selected-area electron diffraction patterns from the mesh grating shown in *(a)*, with electron energies from 120 keV to 40 keV. The TEM acceleration voltages are labeled. The diffract patterns are taken with a large camera length (4.8 m) to zoom-into the center beam, while relatively high-angle electron diffraction from polycrystalline gold is not shown.

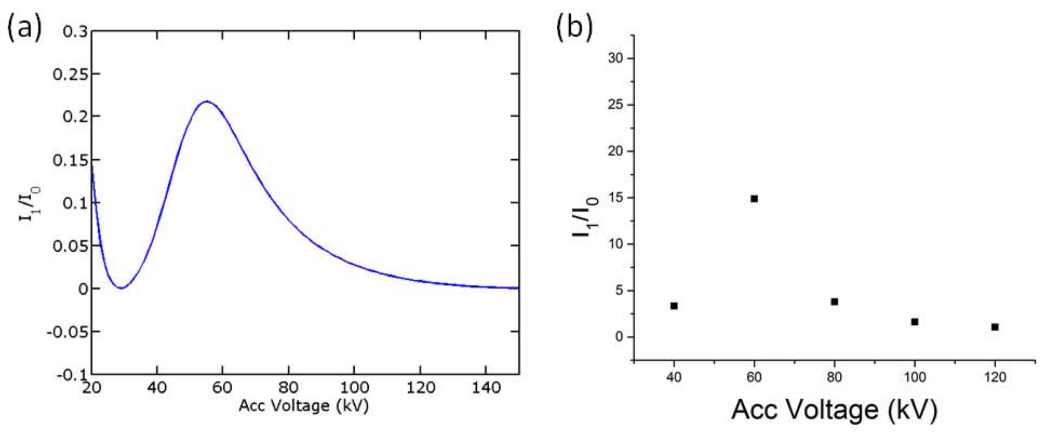

Figure 2: Relative intensity of the first order diffracted beam to the zeroth order direct beam: (a) Theoretical intensity ratio calculated for a mesh grating made from a 40 nm thick membrane with 20 V material mean inner potential, under various acceleration voltages. *(b)* Experimental intensity ratio measured from electron diffraction patterns in *Figure 1*. Difference between theory and experiment in the absolute values of the intensity ratio is due to high-angle electron diffraction from polycrystalline gold.