Fabrication and Characterization of Membrane Nano-gratings for Electron Diffraction

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Nanofabricated transmission gratings are of interest for a variety of applications including electron interferometry and holography as well as vortex beam generation. Compared with natural crystals, nano-gratings provide more flexibility on the control of diffraction angle and orbital angular momentum, and can be used with various electron energies. Recently reported nano-gratings were fabricated with focused ion beam (FIB) milling[1]. Here we report ~10-nm-thick membrane silicon nitride nano-gratings fabricated with electron beam lithography (EBL) and characterized with electron diffraction in a transmission electron microscope (TEM). EBL provides higher resolution, higher throughput, and is applicable to a wider range of materials compared to FIB. Membrane nano-gratings can be used as weak phase gratings for low loss, asymmetric electron beam splitting, enabling the implementation of some specialized electron interferometry and holography techniques[2].

The fabrication process started with silicon nitride membrane (5-20 nm) TEM grids (*SiMPore Inc.*). Oxygen plasma ashing was applied to clean the membrane and to promote resist adhesion. Then, 50 nm poly-methyl-methacrylate (PMMA) resist was spin-coated on top of the membrane, and the nano-grating patterns were defined by an Elionix F-125 EBL system. After resist development, pattern transfer to the silicon nitride membrane was achieved with CF_4 reactive ion etching (RIE). Both 1D line gratings (Figure 1(a)) and 2D mesh gratings (Figure 1(b)) were fabricated, with the latter more suitable for large area patterning, whereas the former had delamination issues. The grating pitch varied from 40 nm to 100 nm, and the patterned area ranged from 2 μm to 6 μm. Nano-gratings were characterized in both a FEI Tecnai TEM operating at 80 kV (Figure 2(a)) and a JEOL 2010F TEM operating in high-dispersion diffraction (HDD) mode (Figure 2(b)). Low electron energy led to larger diffraction angles, and HDD mode offered a higher magnification. Both techniques helped to better visualize the diffraction spots located close to the central spot on the diffraction plane, as the nano-grating pitch is relatively large compared to a crystal. In both diffraction patterns (Figure 2), the diffraction spot spacing was commensurate with the nano-grating pitch, verifying that these spots were indeed generated by electron diffraction from the nano-gratings.

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[1] J. Verbeeck, H. Tian, and P. Schattschneider, Nature **467**, 301 (2010).
[2] W. Putnam and M. Yanik, Phys. Rev. A **80**, 040902 (2009).

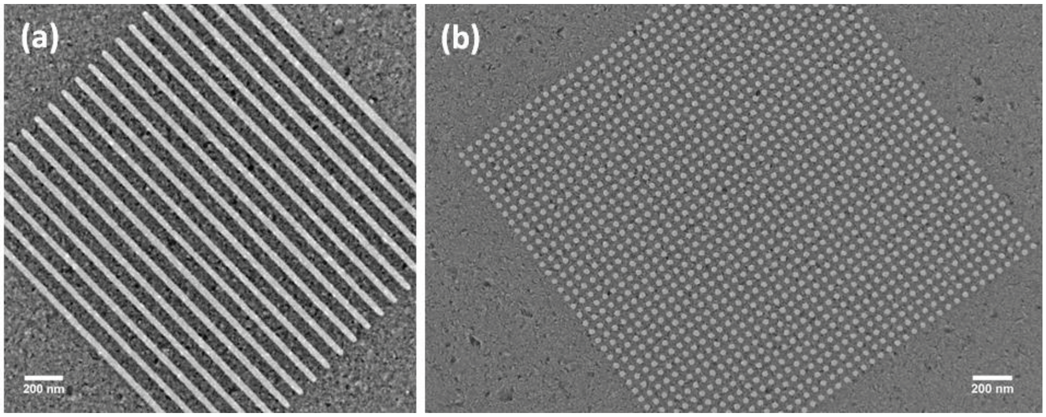

Figure 1: TEM images of fabricated nano-gratings: (a) An 1D line grating patterned on a 10 nm thick silicon nitride membrane with 10 nm gold coating layer. The grating pitch is 50 nm, and the patterned area is a 2 μm × 2 μm square; *(b)* A 2D mesh grating patterned on a 10 nm thick silicon nitride membrane with 10 nm gold coating layer. The grating pitch is 50 nm, and the patterned area is a 2 μm × 2 μm square. The purpose of the gold layer is to prevent charging during imaging and diffraction.

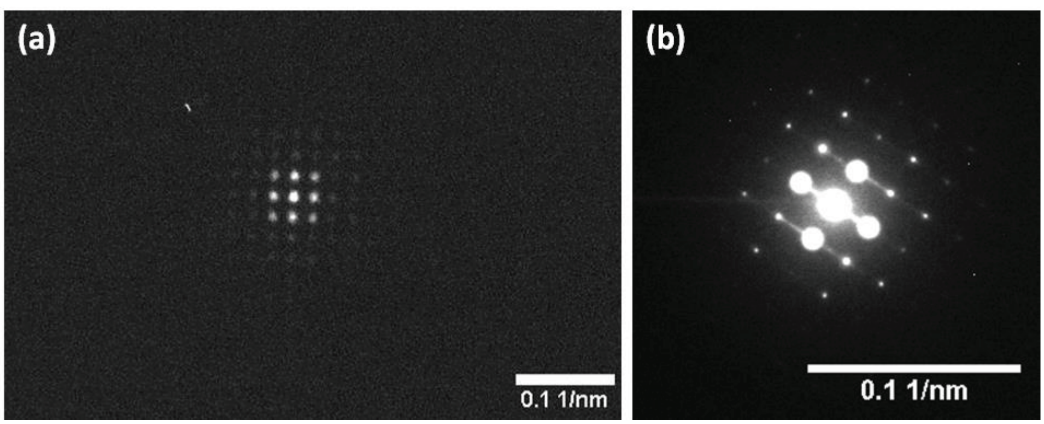

Figure 2: Diffraction patterns of 50 nm pitch 2D mesh nano-gratings: (a) A selected-area diffraction pattern recorded by FEI Tecnai TEM operating at 80 kV with 4.8 m camera length; *(b)* A high-dispersion diffraction pattern recorded by JEOL 2010F TEM operating at 200 kV with 80 m camera length.