An improved phase mask for electron vortex beams

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The use of electron vortex beams for SEM and (S)TEM imaging and analytical electron microscopy is expected to have a revolutionary impact on scientific discoveries and technical developments. It will enable researchers to image in new ways, make new types of measurements, and manipulate the electrons and atoms in materials using its associated orbital angular momentum (OAM) in a manner never before possible [1-5]. In order to obtain the spiral phase shift to produce the electron vortex beam, the primary electron beam must pass through a structure responsible for creating the wavefront phase shift by n2□.

Electron vortex beams have been created in the TEM by passing the primary beam through a dislocated hologram [3, 4] and in the STEM using a spiral zone plate. A focused ion beam (FIB) can produce a nicely shaped grating in a controlled manner although fine gratings for creating high angle diffracted beams are not possible. Creating phase shifted beams from a crystal with a dislocation has been shown possible [1] with the advantage of producing high angles of diffraction useful for separating the diffracted beams. However, using a dislocated crystal has many experimental difficulties including the production of diffuse scattering of the electrons, radiation damage effects that may change the nature of the dislocation, the possible build-up of surface contamination, as well as the difficulty of finding a pure edge dislocation without a screw component where the screw component will give some wavefront distortion due to the surface relief associated with its core. Thus creating a dislocated phase mask from a thin film of material with known properties using the FIB has the most advantages, although making a good dislocated phase mask with a fine grating and complex dislocation core shape had many challenges. The subject of this abstract is a new method of producing a consistently reproducible, highly effective, and highly precise diffraction grating as a phase mask.

There were several issues with the previous methods tried: (1) the FIB cut pattern left a long cantilever section that formed the dislocation, which deflected slightly after fabrication, Fig. 1a, (2) the ratio of grating width to film thickness made it difficult to ensure the cut pattern was clean with straight sides, Fig. 1b and Fig. 1c, (3) the thickness of the metal coating on each side of the film was not very precisely measured, (4) the windows were prone to breakage during transport and handling. Reducing the number of FIB passes of the Ga ion beam resulted in a cleaner cut to produce the best results Fig. 1d, e.

A new FIB cutting pattern was used, which is the negative image of the previous pattern. This means that the center section of the diffraction grating is not an unsupported cantilever. Instead it is shaped like a tuning fork, completely supported. The new gratings were fabricated in 50 nm thick SiN film on TEM grids with 3x3 arrays of 0.1 mm x 0.1 mm windows. The smaller windows reduce breakage, and the thinner film reduces the aspect ratio of grating width to material thickness, which allows the sides of the cut to be cleaner and more perpendicular. The grids were coated on both sides with 2 nm Ti and 20 nm Au with an Angstrom Engineering electron beam deposition system (+/- 0.5 nm thickness resolution) before fabrication with a Hitachi FB-2100 FIB system.

Two diffraction gratings were made; a 5 µm diameter, and a 10 µm diameter as shown in figure 2. The grating spacing was 140 nm in both cases. The fabrication used two passes with a 0.01 nA Gallium beam accelerated at 30 kV (30-0-30 beam in Hitachi parlance). It was discovered that a large number of passes resulted in grating collapse of varying severity, but that a short number of passes resulted in a clean cut. The optimal number of passes was found to be two, with the second cut being in the reverse raster direction from the first. When the FIB is used with high dwell time per pixel (more than 50 µs per pixel), the depth of the cut increases as the beam rasters across the pattern. By exploiting this behavior, it is possible to select a dwell time per pixel so that the depth ramps such that it breaks through the film thickness approximately halfway across the pattern, so the return pass in the opposite direction finishes the cut cleanly. The ideal dwell time for the 5 µm diameter grating cut at 25k times magnification was found to be 450 µs per pixel for both passes, and 115 µs per pixel for both passes for the 10 µm diameter grating cut at 12k times magnification.

References

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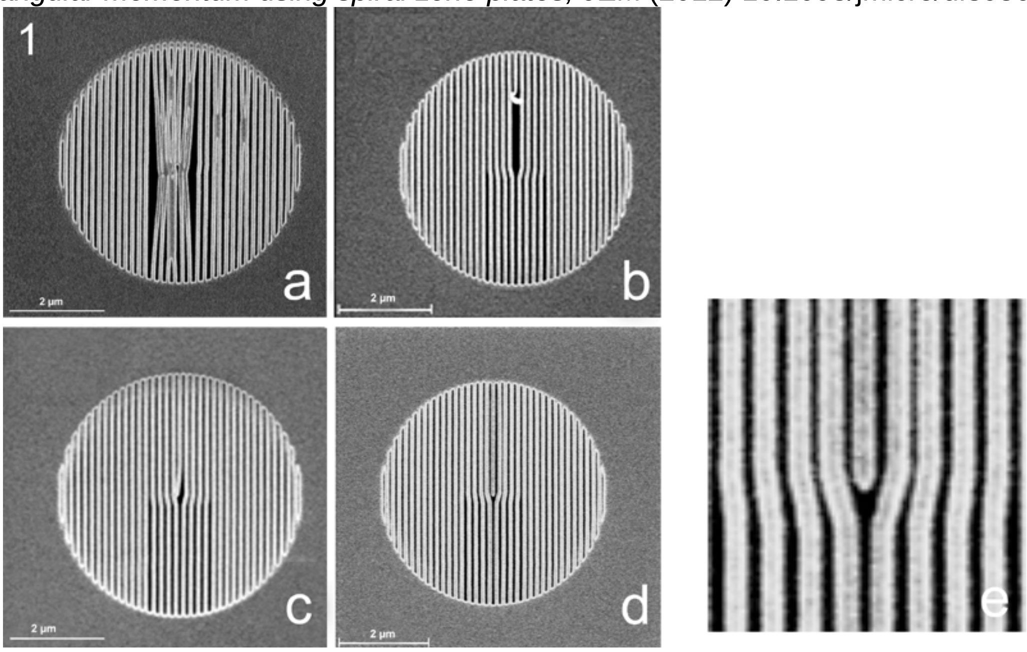

Figure 1. *Dislocated holograms produced by FIB processing of Au-Pd epilayers on Silicon Nitride showing in a) grating collapse, b) and c) half grating curling d) a perfect phase mask having 139 nm periodicity sufficient for separating the electron vortex beams for aperturing and e) a higher magnification of Fig. 1d showing the perfect dislocation core region that can be used to produce a $\pm$ 2* □ *phase shift o beams.*

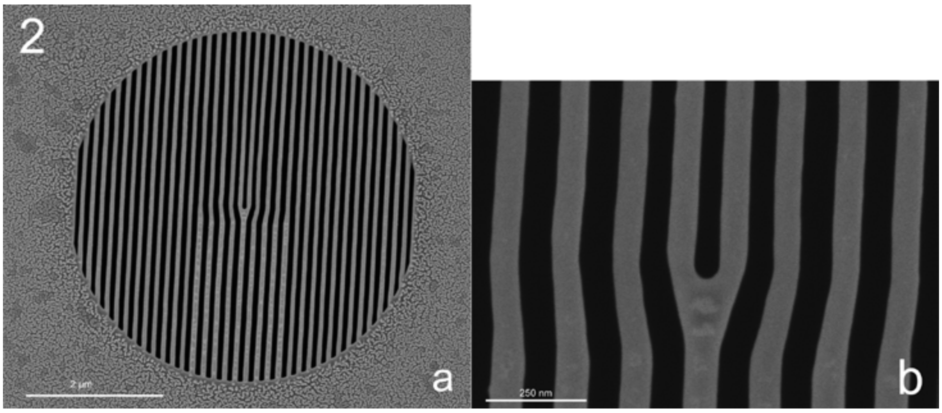

Figure 2. *a) successful inverted phase mask and b) dislocation core of inverted phase mask showing supported tuning fork shape rather than cantilever.*