## Demonstration of High Efficiency Diffractive Optics for Electrons Fabricated with Ion Beam Gas-Assisted Etching

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Structured electrons (SEs), electrons electrons with a transverse spatially dependent phase profile, can be holographically produced with off-axis diffraction phase holograms in a transmission electron microscope (TEM). Researchers have found moderate success at producing SEs with holograms to enable the multitude of theoretically predicted uses and improvements in electron microscopy and spectroscopy, while also answering fundamental physics questions [1]. However, low diffraction efficiencies and diffraction artifacts can cause poor signal-to-noise in many SE applications and measurements limiting their predicted scientific and technological potential.

The two most successful techniques for creating holograms have been Ga<sup>+</sup> ion beam milling [2,3] and electron beam lithography [4], but each technique falls short in fabricating arbitrary hologram mill profiles. Introducing XeF<sub>2</sub> gas during an ion beam mill allows for gas-assisted etching (GAE), a process that combines chemical etching with ion beam sputtering [5]. To impart a desired phase profile to an electron wave, an ideal hologram has the smallest possible grating pitch while maintaining a spatial depth profile that achieves the best diffraction efficiency. GAE allows for spatial depth control while reducing ion implantation, improves spatial resolution by decreasing the material redeposition rate, and increases device yield by decreasing process time. The decreased process time also gives the technique scalability, enabling 50 um grating fabrication in 20% the time of normal FIB nanofabrication.

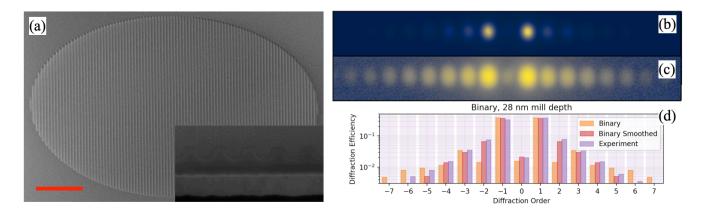
Here we produce binary and blazed grating profiles in thin  $Si_3N_4$  membranes to illustrate the effectiveness of GAE. The maximum first order relative diffraction efficiencies for the binary and blazed grating profiles are 39.6% and 90.3% respectively, when accounting for the loss of electrons due to high angle inelastic scatter in the membrane at 120 kV. We report an average relative diffraction efficiency of 35.5% for the first diffraction orders for binary gratings with nearly complete suppression of the zeroth order Fig. 1 and 70% relative diffraction efficiency into the first diffraction order for blazed gratings Fig. 2, nearly double the largest reported relative diffraction efficiency for blazed gratings (38%), but still 20% from the maximum possible diffraction efficiency. Additionally, we will discuss how low angle inelastic scattering in the  $Si_3N_4$  membrane affects coherence properties of the diffracted probes.

The ability to arbitrarily control the phase of an electron probe with near the maximum possible diffraction efficiency is a significant step in the path to using SEs as nanoscale probes in materials research and will provide an improvement for phase-contrast microscopy techniques [6,7]. GAE could also prove its use in creating holograms for path separated electron interferometry, on-axis aberration correction, and correcting diffraction artifacts of diffracted SE beams.

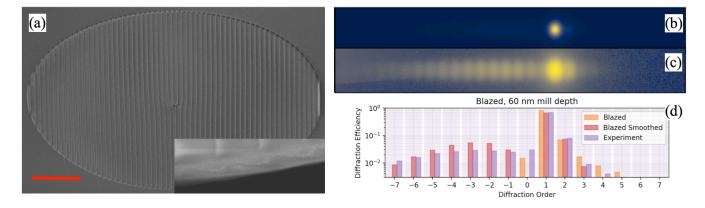
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- [8] Nanofabrication and microscopy were performed at the Center for Advanced Materials Characterization in Oregon (CAMCOR); the authors would like to thank Robert Fischer and Joshua Razink for their help. This material is based upon work supported by the National Science Foundation under No. 1607733, as well as the National Science Foundation Graduate Research Fellowship Program under Grant No. 1309047. Any opinions, findings, and conclusions or recommendations expressed in this material are those of the author(s) and do not necessarily reflect the views of the National Science Foundation.



**Figure 1.** 125 nm pitch binary grating hologram in 100 nm thick Si<sub>3</sub>N<sub>4</sub> membranes. (a) SEM image of grating, scale bar is 2 um, and inset is cross-section with a platinum cap showing grating profile from similar test mills. (b) TEM diffraction pattern, taken in an FEI Tecnai G2 Spirit at 120 kV. (c) Log scale of (b). (d) Theoretical diffraction efficiencies compared to experimental values from (b), orange is from simulated ideal grating profile, red is from simulated ideal grating profile convolved with a Gaussian with the FWHM width of the ion beam, and purple is the experimental data.



**Figure 2.** Same as Fig. 1, but for a 200 nm pitch blazed grating hologram.