KOH Polishing of Nanoscale Deep Reactive-Ion Etched Ultra-High Aspect Ratio Gratings

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A fabrication process has been developed to polish 200 nm-pitch gratings via KOH etching following the Bosch deep reactive-ion etching (DRIE) process. Previous KOH polishing experiments focused on micron scale features.¹ We believe this work is the first combined DRIE-KOH etching process on the nanoscale for ultra-high aspect ratio structures. The primary application of the gratings is x-ray spectroscopy which requires polished sidewalls for efficient x-ray reflection.² Polishing is also critical for increasing the open area by narrowing the grating bars, which increases the throughput of x-rays. The increased open area is also valuable for other applications such as ultraviolet filtration, neutron spectroscopy and biofiltration.

Advanced Bosch processes leave approximately 20 nm of roughness on the sidewalls. This roughness needs to be reduced to <1 nm to efficiently reflect soft x-rays with wavelengths between 1-5 nm.³ Furthermore, high aspect ratio DRIE can result in bar width variations of approximately a factor of two from the top to the middle of the channel, commonly referred to as "bowing."⁴ The polishing procedure presented here removes the roughness to below the resolution of the scanning electron microscope (~5 nm), as shown in Figs. 1 and 2. Remarkably, the bowing has also been reduced by at least a factor of 3 (see Figs 3 and 4).

The polishing process takes advantage of the anisotropy of KOH silicon etching. Specifically, the <111> silicon planes etch approximately 100 times slower than other crystal planes.⁵ This anisotropy allows the grating bars to be etched in 50% by weight KOH at room temperature for up to 60 minutes. Long etches have several key requirements, including \pm 0.1 degree alignment of the grating with respect to the <111> planes, mask roughness below 40 nm and minimal defects in the silicon. If these requirements are not met, the grating will quickly be destroyed by the etch, which etches the non <111> planes in excess of 1 µm per hour. The fabrication steps of this work are described in detail including a novel technique to align the 200 nm-pitch interference lithography image grating to the <111> planes of a <110> silicon wafer.

¹ R. Agarwal et al., "Fabrication of vertical mirrors using plasma etch and KOH:IPA polishing," *Journal of Micromechanics and Microengineering*, vol. 17, no. 1, pp.26-35, 2007.

 ² R. K. Heilmann et al., "Development of a critical-angle transmission grating spectrometer for the International X-Ray Observatory," *Optics for EUV, X-Ray, and Gamma-Ray Astronomy IV, Proc. SPIE*, 7437, 74370G, 2009.

⁵ R. K. Heilmann et al., "Diffraction Efficiency of 200 nm Period Critical-Angle Transmission Gratings in the Soft X-Ray and Extreme Ultraviolet Wavelength Bands," *Appl. Opt.* 50, 1364-1373, 2011.

P. Mukherjee et al., "Plasma etch fabrication of 60:1 aspect ratio silicon nanogratings with 200 nm pitch," J. Vac. Sci. Technol. B, vol. 28(6), C6P70, 2010.

⁵ M. Ahn et al., "Fabrication of 200 nm-period blazed transmission gratings on silicon-on-insulator wafers," *J. Vac. Sci. Technol. B* 26, 2179–2182, 2008.





Figure 2.

Figure 1: Scanning electron micrograph of a cleaved 200 nm-pitch grating after DRIE without KOH polishing. Observe sidewall roughness in excess of 20 nm.

Figure 2: Scanning electron micrograph of a cleaved 200 nm-pitch grating after DRIE with 20 minutes KOH polishing. Observe the near perfect reduction in sidewall roughness.



Figure 3.

Figure 4.

Figure 3: Scanning electron micrograph of a cleaved 200 nm-pitch grating after DRIE without KOH polishing. Observe bowed grating bars with narrow waists.

Figure 4: Scanning electron micrograph of a cleaved 200 nm-pitch grating after DRIE with 20 minutes KOH polishing. Observe the nearly uniform straight bars.