Vertical Directionality Controlled Metal Assisted Chemical Etching for Ultra-High Aspect Ratio Nanoscale Structures

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A long-standing challenge in x-ray microscopy is the lack of efficient, high resolution x-ray optics, especially important for hard x-rays and for compact x-ray microscopes. While x-ray diffractive optics are playing a major role in nanoscale soft x-ray imaging, they have largely been unavailable for hard x-rays where many scientific, technological, and biomedical applications exist. Desired qualities for x-ray diffractive optics include: ultra-high aspect ratio dense features, high resolution, large area/diameter patterning, flexibility for metallization, and arbitrary shape patterning for multifunctional optics.

We have developed a new method called vertical, directionality controlled metal assisted chemical etching (V-MACE) for fabrication of ultrahigh aspect ratio, dense, nanoscale features that achieve the desired qualities required of x-ray diffractive optics fabrication. Because V-MACE is a wet etch and does not require specialized equipment, it is simple and economical. Additionally, there is great flexibility in the subsequent choice of metallization either using atomic layer deposition or electroplating of these structures, which is important for optimizing efficiencies for specific wavelength ranges.

Using V-MACE, we have fabricated high efficiency x-ray diffractive optics for the hard x-ray region for applications in x-ray microscopy, x-ray diffraction, and astronomy. Structures include x-ray Fresnel zone plates and spiral zone plates with aspect ratios ranging from 20:1 to greater than 100:1 aspect ratio with feature sizes currently ranging from 30 - 100 nm. After e-beam lithography of the zone plate pattern, gold lift-off and V-MACE of the Si < 100 > substrate, a conformal Pt layer was applied using atomic layer deposition. X-ray diffraction efficiency measurements encompassing a 30 μm area of the critical outermost zones with 8.995 keV(0.14 nm) synchrotron radiation resulted in 20.1% efficiency.

We note that the work presented here represents a general nanofabrication method that can also be applied to other fields that would benefit from ultra-high aspect ratio structures, including thermoelectric materials, battery anode patterning, sensors, electron accelerator structures, and others.

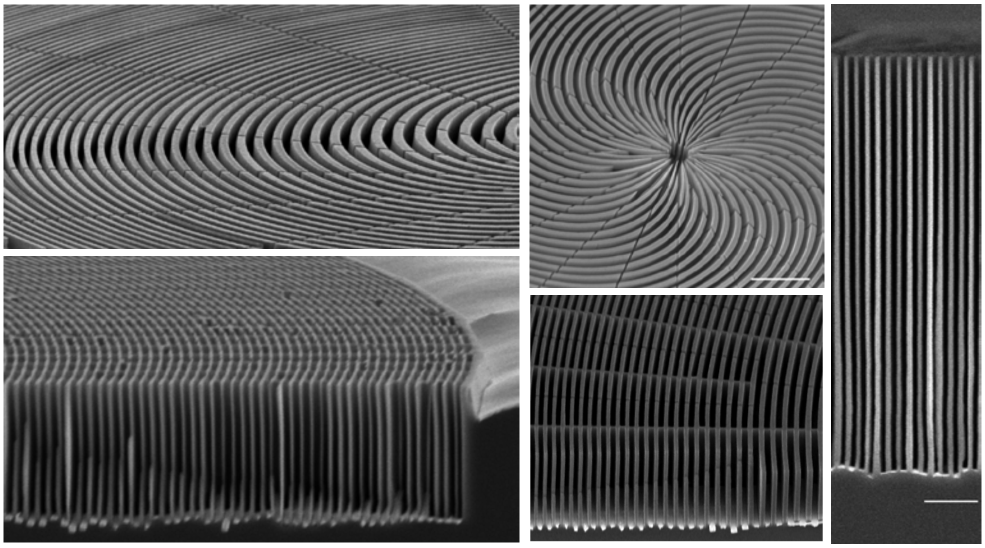

Figure 1: Examples of silicon x-ray diffractive optics fabricated using V-MACE. Circular, spiral, and linear structures are demonstrated here. The rightmost SEM image shows structures with 100 nm lines 6.6 µm tall.

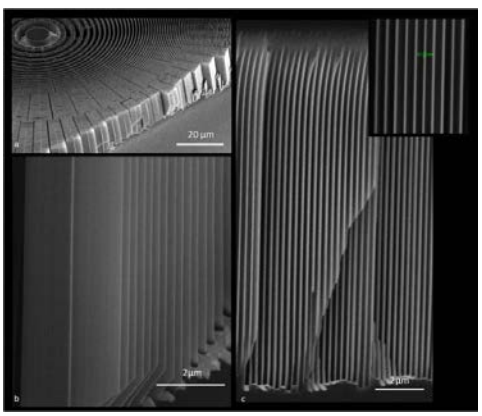

Figure 2: Etched zone plate in Si < 100 > ~14 µm deep, 200 nm period with 51 nm feature size. Smooth sidwalls are also observed.

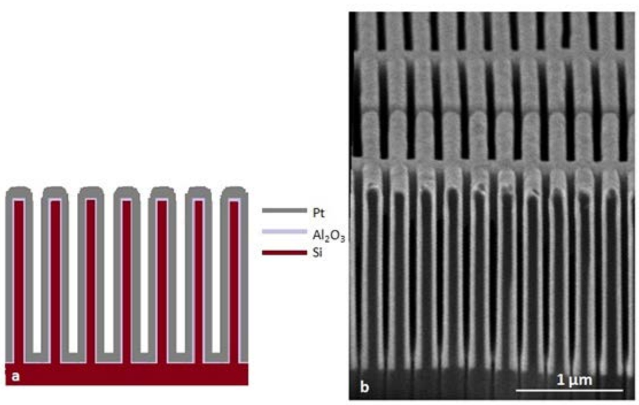

Figure 3: Pt atomic layer deposition used for zone doubling of zone plate structures. SEM of cross section taken at 52 degrees tilt angle.