Fabrication of nickel diffractive phase elements for X-ray microscopy at 8 keV photon energy.

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The spatial resolution of X-ray microscopes is primarily limited by the characteristics of the employed focusing optical elements. Today, Fresnel Zone Plates (ZPs) are widely used at synchrotron radiation sources, due to their capability of concentrating X-ray beams to spots of diameters in the tens of nanometers range. ZPs can be either phase or amplitude diffractive optical elements. Since phase-shift ZPs can reach in principle higher efficiencies, we propose and demonstrate a fabrication scheme for a Ni phase shift ZP, optimized for a 8 keV X-ray beam. A useful parameter to look at as a guideline for selecting the material for a diffractive phase element is the ratio $\boldsymbol{R}$ between the path length L_π necessary to introduce a π shift on an impinging wave and the attenuation length at the working wavelength. If $\boldsymbol{R} \ll 1$ at the working wavelength, the attenuation is negligible in binary diffractive optics with 0- and π-shift structures. Ni shows favorable ratios ($\boldsymbol{R}<0.3$) in the range 6-8.3 keV, and has a minimum at 8.1 keV, with $\boldsymbol{R}=0.135$ and $L_\pi=3.3$ μm. Since the ultimate resolution of a ZP is determined by the dimension of its outermost zone, aiming reaching a resolution of 100 nm implies aspect ratios exceeding 30 in the final Ni structures.. The strategies to fabricate such structures are usually classified as subtractive or additive, according to whether the absorber is subtracted (by dry etching) from an unstructured X-ray absorbing layer, or added into a patterned resist template (by electroplating). None of these two alternative strategies is exempt from problems. A common additive process often suffers from the collapse of the finest nanostructures of the polymer template, mostly during the drying after wet development, because of capillarity[1,2] effects. On the contrary, subtractive strategies suffers from the low selectivity of the etching of hard metals over resists, thus often a lift-off of metallic layer is required or a tri-layer system is proposed[3]. We recently proposed a variation of structuring strategies to overcome both limitations[4]. Here we show further improvements in our methodology, demonstrating its promising performance by producing a Ni phase shifting ZP optimized for 7-8 keV with a thickness of the absorbing layer of 2.8 μm and with a resolution of 100 nm, resulting in aspect ratio of 28.

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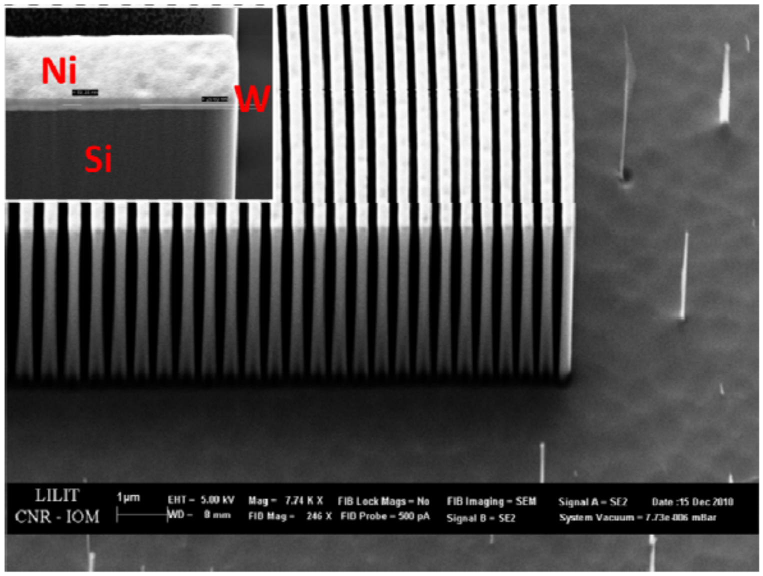

Figure 1: Hard template: SEM micrograph of a 2.8 microns thick hard template of silicon obtained by dry etching (sector of 50 µm diameter ZP, with outermost zones of 200 nm resolution); the inset shows a detail of the etching mask produced: 30 nm of W were deposited by sputtering on the hard template as electroplating seed; the recessed areas of the resist template written by EBL, were filled with Ni by electroplating up to a thickness of 60 nm. Ni acts as etching mask for the thin W layer and for silicon with a fluorine based plasma chemistry in ICP reactive ion etching process.

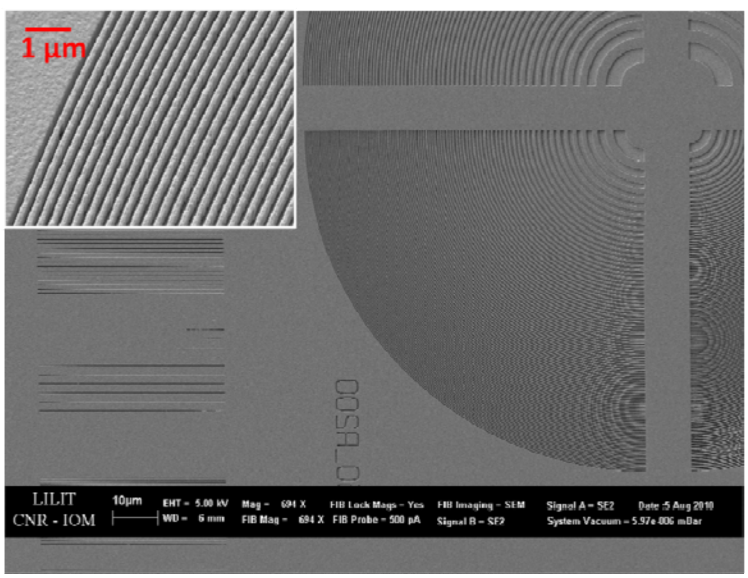

Figure 2: Preliminary results in Ni growing: After the fabrication of the hard template in silicon, Ni was electroplated using a Watts bath, with a current density of 10 mA/cm^2 at 56°C with strong agitation, resulting in a growth rate of 2 nm/s. The Inset shows a detail of the ZP (200 nm resolution) grown to a thickness of 600 nm.