Diffractive optics for laboratory sources to free electron lasers

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Diffractive optics for laboratory sources to free electron lasers

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Abstract. In this contribution we present our recent results in the field of diffractive optics for both soft and hard x-ray radiation, and for laboratory sources to x-ray free electron lasers (XFEL). We developed a laboratory soft x-ray microscope that uses in-house produced zone plate optics as high-resolution objectives. We continuously try to improve these optics, both in terms of efficiency and resolution. Our latest development is the manufacturing of tungsten soft x-ray zone plates with outermost zone widths of 12 nm and 90 nm high structures. For hard x-rays, we investigated the possibility to use metal zone plates on a diamond substrate for nano-focusing of the European X-ray Free Electron Laser. The simulations show that the heat conduction is efficient enough to keep a zone plate well below melting temperature. However, metal zone plates will experience large and rapid temperature fluctuations of several hundred Kelvin that might prove fatal. To test this, we manufactured tungsten on diamond prototype zone plates and exposed them to radiation from the LCLS XFEL. Results show that metal zone plates can survive the XFEL beam.

1. Introduction

Zone plates are high-quality diffractive optical elements which can be used for diffraction-limited x-ray focusing and imaging down to the 10 nm spatial resolution range [1]. In the soft x-ray range, zone plates are the method of choice used in all existing high-resolution x-ray microscopes. For focusing of hard x-ray radiation other alternatives in the form of refractive and reflective optics are widely in use, mainly because of the difficulty to produce high-efficiency hard x-ray diffractive optics. However, recent progress in nanofabrication improved the performance of hard x-ray zone plates considerably, so their use becomes more and more common. In this paper we present the progress of the Stockholm x-ray optics group in the field of diffractive optics for both hard and soft x-rays, for applications in the laboratory and at free-electron laser facilities.

Zone plates are circular diffraction gratings with decreasing line widths towards the outer diameter. Two major parameters characterize the performance of these optical devices, namely the height or thickness of the grating lines and the width of the outermost line. The latter defines the numerical aperture of the optic and therefore the achievable spatial resolution, which is approximately equal to the smallest line width. The thickness defines the efficiency of the zone plate, i.e., how much of the incoming radiation can be found in the first diffraction order which is normally used for imaging. With typical values for optimal efficiency ranging from 100 nm to several microns and outer line width below 100 nm, the fabrication of these optics can only be done with state-of-the-art nanofabrication techniques.

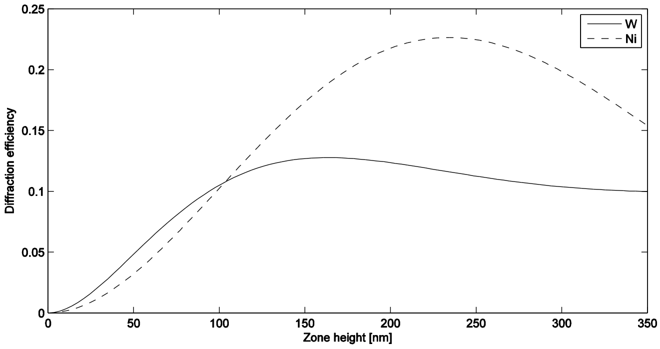

Figure 1. The first order theoretical diffraction efficiency of tungsten (W, solid line) and nickel (Ni, dashed line) as a function of zone height for 2.48 nm wavelength.

In the following we describe our own work towards zone plates with better resolution and higher efficiency for soft x-ray microscopy and hard x-ray zone plates for high-heat-load XFEL applications.

2. Improved soft x-ray zone plates for laboratory x-ray microscopy

The Stockholm group has pioneered the field of laboratory soft x-ray microscopy in the water-window spectral region. Instead of synchrotron radiation, x-rays emitted from laser-produced plasmas are used. Zone plates act as high-resolution objectives in the microscope, and the need for these optics was the reason to start our own nanofabrication of diffractive optics.

Nickel is the optimum material for the 2.48 nm operation wavelength of our microscope, as can be seen in figure 1. Our standard zone plates have outermost zone width of Δr = 25-30 nm and a zone height of 150-200 nm, close to the optimum value for highest efficiency. For details about the manufacturing process, see Ref. [2]. In an attempt to improve the resolution of the optics, i.e., to achieve smaller Δr, we could manufacture nickel zone plates with Δr = 13 nm [3] at a zone height of 35 nm, yielding a theoretical diffraction efficiency of below 2%. This height limit is mainly due to collapsing of higher zones in the electroplating step, and therefore we investigated alternative approaches that could lead to better efficiency.

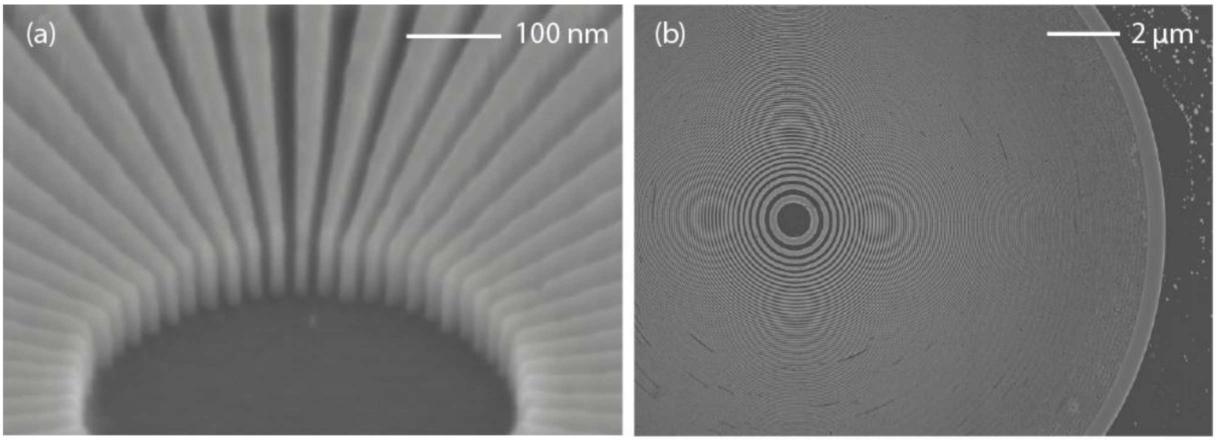

Figure 2. Fabricated high-resolution tungsten structures for soft x-ray microscopy. (a) Inner part of a Siemens star test pattern with innermost half-period of 12.5 nm. (b) Zone plate with Δr = 12 nm. The structure height is 90 nm in both cases.

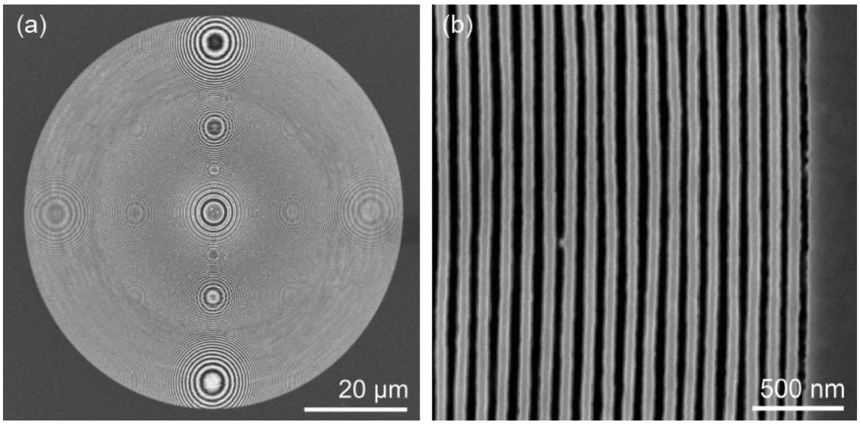

Figure 3. Fabricated prototype tungsten-on-diamond zone plates for FEL applications. (a) Overview of zone plate with 75 µm diameter. (b) Outer part of zone plate with Δr = 50 nm. Tungsten height is 500 nm.

Tungsten is a material used for hard x-ray zone plates [4], and the efficiency curve for 2.48 nm can also be found in figure 1. As it acts as a pure absorbing material for soft x-rays, it cannot achieve the same maximum efficiency as in the case of Ni, but below a thickness of 100 nm both materials have similar performance. However, in contrast to Ni it can be structured directly by reactive ion beam (RIE) etching, avoiding the electroplating step. Accordingly, higher structures and higher efficiencies would be possible.

We successfully developed a novel fabrication process for high-resolution soft x-ray zone plates in tungsten. Details of the nanofabrication process can be found in Ref. [5]. The key step is the pattern transfer into W by RIE in an $SF_6/0_2$ plasma at a cryogenic temperature of -50 °C. Figure 2 shows the scanning electron microscope (SEM) images of a produced zone plate with Δr = 12 nm and a tungsten height of 90 nm, resulting in a theoretical diffraction efficiency of 9.6%, and a star test pattern with the same height and 12.5 nm wide lines.

3. Hard x-ray zone plates for XFEL radiation

Many application experiments using hard x-ray free electron laser radiation at energies around 10 keV would benefit from nanofocused x-ray beams with diameters around 100 nm. However, focusing to these dimensions is far from a standard task and only recently attempts were made using a Kirkpatrick-Baez mirror system and a set of compound refractive lenses. We have investigated the use of zone plates instead. They are easy to handle since the single-element optic can be quickly aligned in the beam. Moreover, they offer the prospect of focal spot sizes well below 100 nm, as described in the previous section.

An important question is if zone plates can withstand the high-intensity XFEL beam, since a considerable amount of the incoming radiation will be absorbed in the relatively thin zone plate material layer. Therefore we developed a simulation tool for the heat transfer in zone plate optics irradiated with XFEL beams, based on the finite element method. Calculations showed that the single-pulse temperature increase in zone plates made out of heavy-metal materials placed in the direct unfocused XFEL beam is well below the melting temperature of the material [6, 7].

Due to our experiences with tungsten, we fabricated prototype zone plates in this material [8], see also figure 3. The optics use a diamond substrate for optimal heat conduction. The zone plates were then used in two types of test experiments [9]. First, we irradiated the optics with focused, pulsed high-intensity visible laser pulses that should give similar temperature profiles as compared with XFEL radiation. No damage to the zone plate nanostructures could be observed after several million laser pulses. Second, we placed the same zone plates in the direct, unfocused beam (6 keV photon energy) of the LCLS XFEL in Stanford, USA. Also here no damage to the optics could be observed after 100000 pulses. The same was true in a similar, independent study [10]. These are very promising results that justify possible future use of zone plates for XFEL nanofocusing.

4. Conclusion

We successfully fabricated soft and hard x-ray zone plates made out of tungsten. In the soft x-ray range, zone plates with outermost zone width as low as 12 nm could be manufactured with a considerable height of 90 nm for high diffraction efficiency. In the hard x-ray range, tungsten-on-diamond zone plates were fabricated for use with XFEL radiation. These optics were successfully tested at the LCLS XFEL, where no radiation damage to the nanostructures could be observed.

Acknowledgements

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