Microelectronic Engineering 88 (2011) 3123–3126

Contents lists available at ScienceDirect

Microelectronic Engineering

journal homepage: www.elsevier.com/locate/mee

Platinum zone plates for hard X-ray applications

E. Chubarova [a], D. Nilsson [a], M. Lindblom [a], J. Reinspach [a], J. Birch [b], U. Vogt [a], H.M. Hertz [a], A. Holmberg [a,*]

[a] Biomedical and X-Ray Physics, Department of Applied Physics, Royal Institute of Technology (KTH), Stockholm SE-106 91, Sweden
[b] Thin Film Physics, Department of Physics, Chemistry, and Biology, Linköping University, SE-581 83, Sweden

ARTICLE INFO

Article history:
Received 18 January 2011
Received in revised form 17 May 2011
Accepted 20 June 2011
Available online 26 June 2011

Keywords:
Zone plate
Pt electroplating
X-ray optics
Electron beam lithography

ABSTRACT

We describe the fabrication and evaluation of platinum zone plates for 5–12 kV X-ray imaging and focusing. These nano-scale circular periodic structures are fabricated by filling an e-beam generated mold with Pt in an electroplating process. The plating recipe is described. The resulting zone plates, having outer zone widths of 100 and 50 nm, show good uniformity and high aspect ratio. Their diffraction efficiencies are 50–70% of the theoretical, as measured at the European Synchrotron Radiation Facility. Platinum shows promise to become an attractive alternative to present hard X-ray zone plate materials due to its nano-structuring properties and the potential for zone-plate operation at higher temperatures.

1. Introduction

Nanometer X-ray focusing and imaging is of growing importance, at synchrotrons as well as with laboratory systems [1]. Zone plates are often the preferred high-resolution X-ray optical element in such microscopy systems, both in the water window (approx. 0.3–0.5 keV) and in the hard X-ray regime (up to ~12 keV). The fabrication of such lenses presently relies on a limited number of materials having the necessary combination of appropriate X-ray optical constants and process parameters which allow nano-scale fabrication. In the present paper we introduce a new material, platinum, for zone plates, describe its fabrication and demonstrate its applicability for the hard X-ray range.

Zone plates are circular diffraction gratings with radially decreasing line width. Their imaging properties are determined by two characteristics: the outermost zone width, dr_N, which sets the resolution, and the optical material and its thickness, which determines the diffraction efficiency [2]. Zone plates for the hard X-ray regime typically employ heavy metals as the optical material. Presently zone plates have been demonstrated in Ta [3], W [4], Ir [5,6] and Au [7]. Fig. 1 depicts the theoretically calculated efficiency at 8 kV as a function of material thickness for these materials and Pt. Clearly a high thickness is important for high diffraction efficiency and thus, a high aspect ratio becomes necessary for operation at high-resolution. In brief, three fabrication methods are used for patterning the metal, dry etching via a hard mask (Ta and W), atomic layer deposition on a mold (Ir) and electroplating in a polymer mold (Au). The dry etching processes have typically demonstrated 50–100 nm outer zone width, with aspect ratios up to 12, while the atomic layer deposition of Ir presently allows <15 nm and an aspect ratio of 25 due to the line doubling effect. For the electroplating process, present state-of-the-art hard X-ray gold zone plates have an outer zone width of 24 nm and a thickness of 300 nm [7]. For comparison, electroplated zone plates for the soft X-ray range are best made of nickel, presently allowing 13 nm outer zone width and 35 nm height [8].

The diverse fabrication methods and material choices that have been used so far is a consequence of the quest for optics with higher resolution and improved diffraction efficiency. Pt is a high-Z metal optical material that has not been investigated previously. Although the efficiency for Pt is similar to the other materials (cf. Fig. 1), it has other properties that make it interesting for zone plate fabrication. First, Pt can be electroplated and is therefore suitable for similar processes as Au. Second, when comparing to Au, the melting point of Pt is 2045 K as compared to the melting point of Au at 1338 K. This would potentially allow operation at higher temperatures, something that is of growing importance with the emergence of high-power, high-brilliance 4th generation hard X-ray synchrotron sources [9]. Third, the sputter etch resistance of Pt is 2× that of Au, which can make it suitable for "multi-material" zone plates. We have recently fabricated multi material zone plates for soft X-ray applications [10]. Here an electroplated Ni zone plate was used as mask for extending the pattern into at thick Ge layer by dry etching, thereby doubling the efficiency compared to the original Ni zone plate. Pt has potential to be a very good electroplated mask material for a similar process in the hard X-ray optics. Finally, Pt is also an interesting material for dry etching. It forms

* Corresponding author.
E-mail address: anders.holmberg@biox.kth.se (A. Holmberg).

0167-9317/$ - see front matter © 2011 Elsevier B.V. All rights reserved.
doi:10.1016/j.mee.2011.06.011

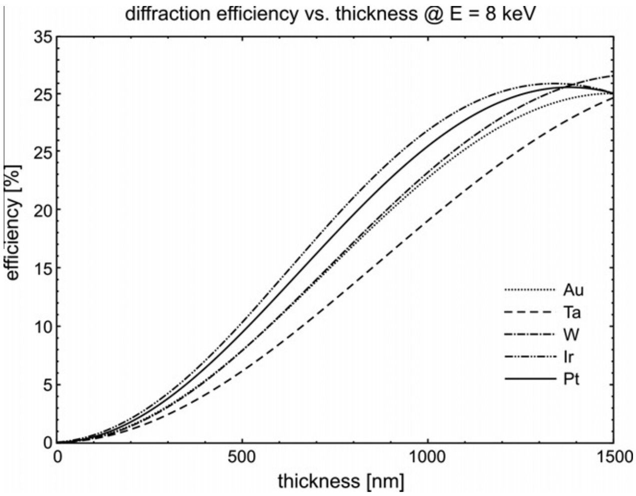

Fig. 1. Theoretical efficiency of hard X-ray zone plates at 8 kV for different heavy-metal elements.

the volatile compound PtF_6 with a boiling point of 69 °C (NTP), which makes it suitable for standard F-based dry etching. It could therefore be interesting for new multi-material approaches as a metal for dry etching. Thus, Pt appears to have a large potential as a future hard X-ray optical material.

In the present paper we demonstrate the fabrication of Pt zone plates down to 50 nm outer zone width and with high efficiency using electroplating. We note that although Pt electroplating is commonly used in a wide range of industrial coating applications [11,12] the Pt plating of repetitive high-aspect-ratio nanostructures with <100 nm dimensions have not been achieved before. Single platinum nanowires have been made using photolithography patterning followed by platinum electrodeposition [13] or by electrochemical fountain pen nanofabrication [14]. Platinum nano-hole-arrayed electrodes have been synthesized by template wetting [15]. Micro structured electro-plated Pt has been used for biomedical and other applications [16,17].

2. Experimental methods

2.1. Nanofabrication process

The fabrication of the platinum zone plate is based on the process depicted in Fig. 2. This process is suitable for high aspect-ratio periodic structures like zone plates, gratings and test structures [18]. In brief, the structures are made by electroplating platinum into a polymer mold made out from a tri-layer resist that is structured by electron beam lithography (EBL) and reactive ion etching (RIE).

Fifty nanometers silicon nitride membranes were used as substrates and coated with a stack of materials (Fig. 2a). First, a plating base was deposited by electron-beam evaporation (Edwards Auto 306 system, 10^{-6} Torr base pressure). It consists of a 20 nm adhesive layer of titanium covered by a 30 nm plating seed layer of gold. Then the trilayer resist consisting of 550 nm thick polyimide as plating-mold (PI-2610, HD Microsystems), a 50 nm SiO_2 hard mask and a 110 nm thick electron-beam resist (Zep 7000, Nippon Zeon Co.) was deposited. The polyimide and the electron-beam resist were spun cast and baked at 350 °C for 2.5 h and at 170 °C for 30 min, respectively. The SiO_2 hard mask was sputter deposited (AJA Orion, 10^{-8} Torr base pressure) at 3 mTorr pressure, 25 sccm Ar flow, and 100 W power.

The samples were patterned by EBL at 25 keV (Raith 150 system), with a typical dose of 150 $\mu C/cm^2$, and developed in hexyl acetate for 30 s (Fig. 2b). The pattern was transferred into SiO_2 hard mask using RIE with CHF_3 (Oxford Instruments, Plasmalab 100) at 10 sccm flow, 3 mTorr pressure and 100 W power (Fig. 2c). The selectivity between SiO_2 hard mask and electron-beam resist upon these conditions is 1:1. 25 nm wide lines are clearly resolved in such a processing. Then the pattern was transferred into the underlying polyimide layer by RIE with O_2 (Oxford Instruments, Plasmalab 80+) at 10 sccm flow, 2 mTorr pressure and 50 W power resulting in the plating mold (Fig. 2d). At the next step of the process the platinum was electroplated into the mold (Fig. 2e). The electroplating of platinum is discussed in Section 2.2. After electroplating the mold was removed by repeating the two RIE steps (Fig. 2f).

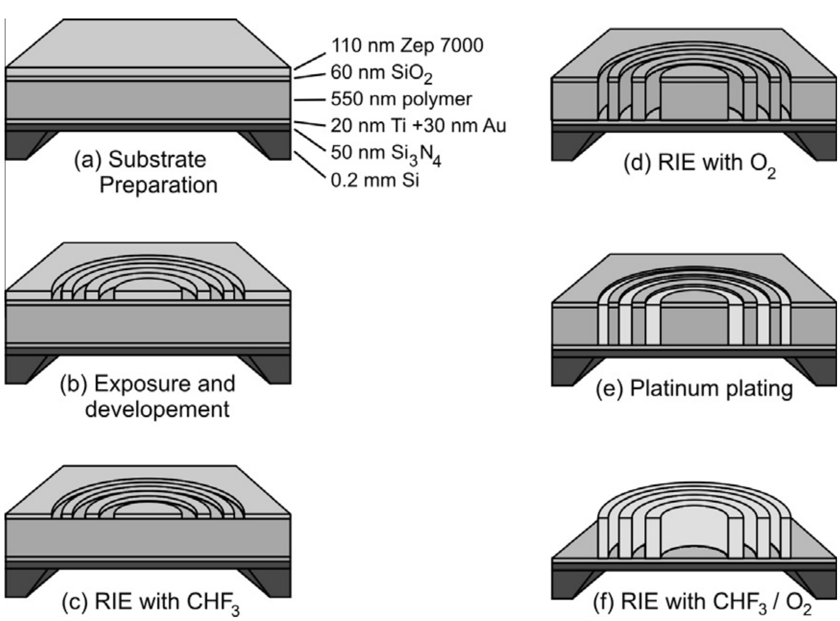

Fig. 2. The electroplating-based platinum zone plate fabrication process.