Available online at www.sciencedirect.com
ScienceDirect
Microelectronic Engineering 84 (2007) 1136–1139
MICROELECTRONIC ENGINEERING
www.elsevier.com/locate/mee

SU-8 plating mold for high-aspect-ratio nickel zone plates

M. Lindblom *, H.M. Hertz, A. Holmberg

Biomedical and X-ray Physics, Department of Applied Physics, Royal Institute of Technology/Albanova, SE-10691 Stockholm, Sweden

Available online 2 February 2007

Abstract

Nickel zone plates are fabricated by electrodeposition into a mold with high aspect ratio and narrow line width. This process requires high-mechanical stability of the mold to avoid pattern collapse in the plating bath. In the present paper we demonstrate how SU-8 can be used as plating mold material in a tri-layer resist to fabricate 35-nm half-pitch nickel gratings with an aspect ratio exceeding 11:1. To attain sufficient stability of the mold the SU-8 was cured by e-beam exposure with a dose of 25 mC/cm^2 at 5-keV electron energy.

Keywords: SU-8; Zone plate; Aspect ratio; Electroplating

1. Introduction

Fabrication of nickel zone plates involves electroplating into narrow-line-width high-aspect-ratio molds. This imposes severe demands on the mechanical stability of the mold material as well as the mold-to-substrate adhesion. In the present paper we describe how highly cross-linked SU-8 (MicroChem Corp.) can be used as plating mold material in a tri-level resist for fabrication of high-aspect-ratio nickel structures.

Zone plates are diffractive optical elements commonly used for focusing and imaging in the X-ray spectral range [1]. They are essentially circular gratings in which the period decreases with increasing radius. We have previously reported on the fabrication of nickel zone plates for use as objectives and condensers [2,3] in compact soft-X-ray microscopy [4]. For high-resolution imaging, narrow zone widths are needed [5], typically in the range of 20–50 nm for an objective zone plate. At the same time, high zones are needed for high-diffraction efficiency. For water-window wavelengths ($\lambda = 2.4$–4.2 nm), which are of particular interest for soft-X-ray microscopy, nickel is a suitable optical material. Theoretically, nickel zone plates can have a first order diffraction efficiency of ~23%, at $\lambda = 2.4$ nm, for the optimal zone height of 250 nm [6].

To achieve a high aspect ratio the nickel needs to be electroplated into a mold. An overview of our fabrication process is shown in Fig. 1. The electroforming replicates the mold pattern with high fidelity but it also limits the aspect ratio. If the mold structures are too high and narrow they can be destroyed due to both adhesion failure and deformation when immersed into the plating bath. Based on the nature of the collapse, and work published on a similar phenomenon occurring in the drying process after development of high-aspect-ratio resist patterns [7,8], we assume that it is caused by surface tension forces acting when the liquid fills the mold.

The difficulty of electroplating high-aspect-ratio narrow-line-width structures is well illustrated by the analysis of the effects of surface tension forces on grating lines in resist made by Tanaka et al. [7]. The requirements on resist stability increase strongly with aspect ratio, but also with decreasing line spacing and absolute dimension. So far the best results – in terms of line width, pitch and aspect ratio in plated structures – have been obtained using an X-ray-hardened copolymer of phenylethylene and divinylbenzene (PEDVB) as plating mold material [6]. The PEDVB was synthesized specifically for this application and the radiation dose needed for proper curing was 2×10^9 Gy. To obtain this dose, the samples were exposed

* Corresponding author.
E-mail address: magnus.lindblom@biox.kth.se (M. Lindblom).

0167-9317/$ - see front matter © 2007 Elsevier B.V. All rights reserved.
doi:10.1016/j.mee.2007.01.109

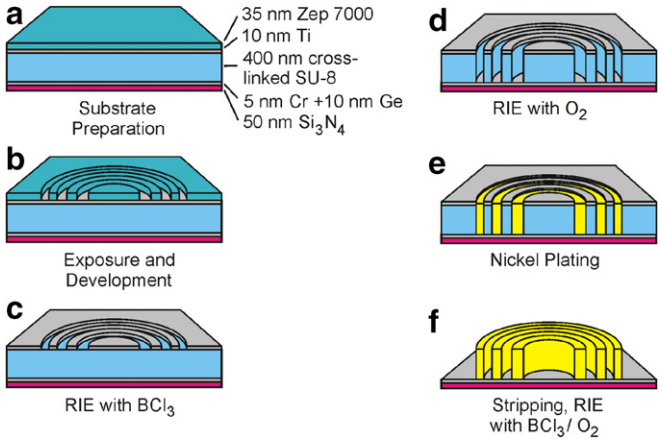

Fig. 1. Process steps for fabrication of high-aspect-ratio nickel zone plates.

at a synchrotron facility (BESSY II). Nickel zone plates with outermost zone width of 20 nm and zone height of 175 nm [9] and nickel gratings with 90-nm half-pitch and a height of 1000 nm [10] were fabricated using this copolymer. This corresponds to aspect ratios of 8.2:1 and 11:1, respectively.

In the following we describe how we have obtained similar results in a simpler way using commercially available SU-8 as plating mold material in a tri-layer resist. SU-8 is an epoxy-based negative-tone photoresist which can also be exposed with electrons or X-rays. It was originally designed for thick-film ultraviolet-lithography for high-aspect-ratio micro-electrical mechanical devices [11,12]. It can be used both as part of a devices and in combination with electroplating [13,14]. The glass-transition temperature is high, about 230 °C [15], which is important in our process due to the heating in the reactive-ion-etching (RIE) process step. Furthermore, SU-8 has good adhesion to germanium, which we use as seed layer in the plating base. Along with the compatibility with electroplating these are key properties for a plating mold material in our process.

2. Experiments and results

2.1. Fabrication process

Fig. 1 shows our process. The SU-8 constitutes the bottom layer of the tri-layer resist and is crosslinked by e-beam flood exposure before the etch mask and the e-beam resist is added. The plating mold is formed by transferring an e-beam written pattern by RIE. In this way the SU-8 can be structured with very high resolution.

Silicon samples coated with 50 nm of silicon nitride were used as substrate onto which a plating base of 5 nm of chromium (adhesion layer) and 10 nm of germanium (seed layer) was deposited. SU-8 2002 was spin-coated and cured to form a crosslinked coating between 350 nm and 500 nm thick. This step is discussed in detail in Section 2.2. A 10-nm titanium etch-mask layer was vapor deposited on top of the SU-8 and finally a 35 nm layer of e-beam resist, Zep7000 (Nippon Zeon), was spin-coated.

The Zep7000 was patterned with e-beam lithography at 25 keV in a Raith150 system and dip developed in hexyl acetate for 30 s. The patterns consisted of gratings with 1:1 line-to-space ratio and half-pitch down to 25 nm. The pattern transfer to the Ti etch mask was performed with RIE (Oxford Instr., Plasmalab 80+) with BCl_3 for 5 min at a pressure of 15 mTorr, an rf-power of 80 W, a BCl_3 flow rate of 10 sccm and with a resulting DC bias between 85 and 90 V. This etch step was followed by a 15 s rinse in deionized water to remove contaminants. The plating mold was then formed by RIE (Oxford Instr., Plasmalab 80+) in an oxygen plasma at an rf-power of 50 W, 3 mTorr pressure and a flow rate of 10 sccm. This resulted in a DC bias of 450 V. The etch rate was approximately 30 nm/min. The electrodeposition of nickel was carried out in a nickel-sulfamate solution (Lectro-nic 10-03s, Enthone Inc.) using pulse plating as for our regular zone plate fabrication [16]. The peak current density was 2 A/cm^2 and the duty-cycle was 1:200 which results in a mean current density of 10 mA/cm^2.

2.2. SU-8 curing process

To evaluate the SU-8 as plating mold, layer thicknesses in the range 350–500 nm were used. This is thicker than the optimal thickness for nickel zone plates (at water-window wavelengths) but enables evaluation in wider lines for which the process yield is higher. SU-8 2002 was diluted with cyclopentanone in a 2:1 ratio and spin-coated on the substrate. The samples were pre-exposure baked at 65 °C for 1 min and then ramped to 95 °C and left for another minute. The ramp time was 2 min. At this stage, a proper curing is needed to attain the rigidity required for high-aspect-ratio structuring. For this purpose we combined e-beam exposure and baking. A wide range of parameters were investigated.

In the initial experiments doses ranging from 1 to 100 μC/cm^2 at 10–25 keV were applied in a Raith150 e-beam lithography system. This range covers commonly reported values for e-beam exposed SU-8 [17–22]. For the post-exposure bake the starting point was the vendor's recommendation: first 60 s at 65 °C, then 60 s at 95 °C after a temperature ramp, followed by hard bake at 200 °C for 60 s [23]. From this the baking parameters were varied and post-bake temperature up to 120 °C and hard-bake temperature as high as 300 °C were applied for as long as 30 min for each step. Within this parameter range no significant difference in the properties of the SU-8 was observed for our application. Typically, an aspect ratio of 4:1 to 5:1 was achieved in electroplated 30 nm half-pitch gratings. Molds with higher aspect ratio always collapsed. This is exemplified by Fig. 2 in which 30-nm half-pitch gratings are shown before (a) and after (b) the electroplating step. The SU-8 thickness was 400 nm and the sample

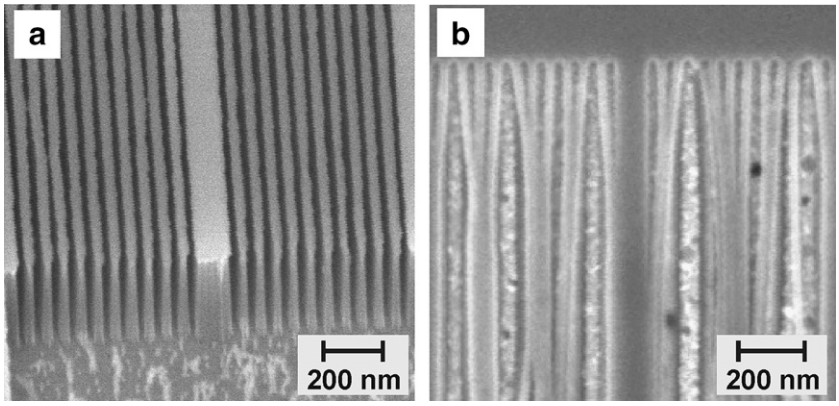

Fig. 2. Scanning electron micrographs of a 400 nm thick SU-8 mold with 30-nm half-pitch gratings. (a) The mold before plating (45° view angle). (b) The tilted mold structures after plating (top view). The SU-8 was cured with a dose of 100 μC/cm^2.

was exposed with 100 μC/cm^2 and post-exposure baked and hard baked according to the vendor's recommendation.

At the electron–radiation doses discussed in the previous paragraph the crosslinking is mainly due to activation of the radiation-sensitive initiator which then acts as catalyst [11]. The crosslink density first increases with dose but eventually reaches a saturation point after which an increased dose does not significantly increase the amount of crosslinking [15]. However, for sufficiently high electron doses a large number of polymer types undergo crosslinking without the presence of an initiator [24]. To exploit this, a 3-kW 5-keV electron gun (Boc Edwards EB3) was used to apply doses in the milli-Coulomb range. The e-beam current was typically 1 mA and the beam was swept over a large area to reduce the current density on the sample. The short penetration depth of 5-keV electrons results in a highly non-uniform dose profile throughout the resist thickness, e.g., the dose at the bottom of a 500 nm thick layer is only a few percent of that at the surface. However, no effect was observed that could be associated with this dose variation. The best results were obtained with the highest applied dose of 25 mC/cm^2. After applying this dose the decrease in film thickness was typically about 25% and the increased hardness could easily be noticed by scratching the surface with a sharp instrument. The improvement of achievable aspect ratio was considerable compared to what was achieved in the low-dose-exposed SU-8 and also to what we have reported previously [2]. Fig. 3 shows 35-nm half-pitch gratings of electroplated nickel after the 400-nm high mold has been removed. The aspect ratio of the nickel lines exceeds 11:1.

In general the SU-8 had good compatibility with our tri-layer-resist process. In particular the adhesion to germanium, which is used as seed layer, was very good. No peeling of the mold in the plating bath was observed for any of the applied doses. The only serious fabrication issue was the poor adhesion of the Ti etch mask to the SU-8. The Ti layer sometimes formed creases and cracks in the rinse step after BCl_3 RIE or in the plating step. This occurred for highly exposed samples only.

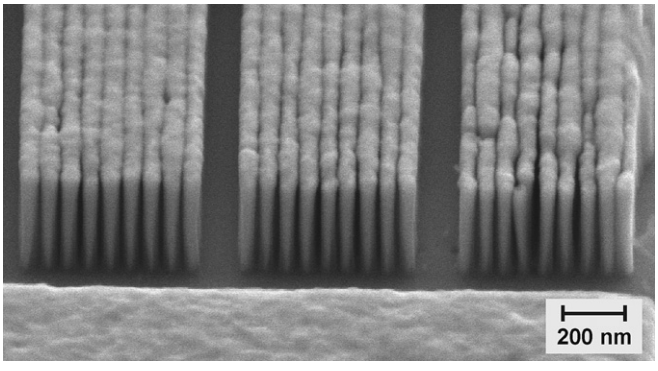

Fig. 3. A scanning electron micrograph of 35-nm half-pitch nickel gratings (60° view angle). The SU-8 was cured with a dose of 25 mC/cm^2.

3. Summary and conclusion

We have investigated SU-8 as a plating mold material for high-aspect-ratio electroforming. We have shown that SU-8 can be used to attain an aspect ratio exceeding 11:1 in 35-nm half-pitch nickel gratings. Despite the much simpler process, the results are comparable to earlier state-of-the-art [9,10].

Acknowledgements

We thank the Göran Gustafsson Foundation and the Swedish Foundation for Strategic Research for financial support.

References

[1] S. Susini, D. Joyeux, F. Polack, (Eds.), in: Proceedings of the 7th International Conference on X-ray Microscopy, J. Phys. IV (France) 104, (2003).
[2] A. Holmberg, S. Rehbein, H.M. Hertz, Microelectron. Eng. 73–74 (2004) 639–643.
[3] S. Rehbein, A. Holmberg, G.A. Johansson, P.A.C. Jansson, H.M. Hertz, J. Vac. Sci. Technol. B 22 (2004) 1118.

[4] M. Berglund, L. Rymell, M. Peuker, T. Wilhein, H.M. Hertz, J. Microsc. 197 (2000) 268–273.
[5] A.G. Michette, C.J. Buckley, Optical Systems for Soft X-rays, Plenum Press, New York, 1986, pp. 217–241.
[6] D. Weiss, M. Peuker, G. Schneider, Appl. Phys. Lett. 72 (1998) 1805.
[7] T. Tanaka, M. Morigami, N. Atoda, Jpn. J. Appl. Phys. 32 (1993) 6059.
[8] T. Kondo, S. Juodkazis, H. Misawa, Appl. Phys. A 81 (2005) 1583.
[9] M. Peuker, Appl. Phys. Lett. 78 (2001) 2208.
[10] D. Hambach, G. Schneider, J. Vac. Sci. Technol. B 17 (1999) 3212.
[11] J.M. Shaw, J.D. Gelorme, N.C. LaBianca, W.E. Conley, S.J. Holmes, IBM J. Res. Dev. 41 (1997) 81.
[12] K.Y. Lee, N. LaBianca, S.A. Rishton, S. Zolgharnain, J.D. Gelorme, J. Shaw, T.H.-P. Chang, Vac. Sci. Technol. B 13 (1995) 3012.
[13] A.L. Bogdanov, S.S. Peredkov, Microelectron. Eng. 53 (2000) 493.
[14] H. Lorenz, M. Despont, N. Fahrni, J. Brugger, P. Vettiger, P. Renaud, Sensor Actuat. A 64 (1998) 33.
[15] R. Feng, R.J. Farris, J. Micromech. Microeng. 13 (2003) 80.
[16] M. Lindblom, H.M. Hertz, A. Holmberg, J. Vac. Sci. Technol. B 24 (2006) 2848.
[17] M. Aktary, M.O. Jensen, K.L. Westra, M.J. Brett, M.R. Freeman, J. Vac. Sci. Technol. B 21 (2003) L5.
[18] W.H. Wong, E.Y.B. Pun, J. Vac. Sci. Technol. B 19 (2001) 732.
[19] E.A. Shields, F. Williamson, J.R. Leger, J. Vac. Sci. Technol. B 21 (2003) 1453.
[20] F. Williamson, A. Shields, in: Proc. of the University/Government/Industry Microel. Symp., 2003, p. 57. <http://ieeexplore.ieee.org/>.
[21] A.K. Nallani, S.W. Park, J.B. Lee, Proc. SPIE, 2003. <http://www.utdallas.edu/~spark/spie2003.pdf>.
[22] V. Kudryashov, X.-C. Yuan, W.-C. Cheong, K. Radhakrishnan, Microelectron. Eng. 67–68 (2003) 306.
[23] MicroChem SU-8 product data sheet, <www.microchem.com>.
[24] V.S. Ivanov, Radiation Chemistry of Polymers, VSP, Utrecht, 1992, p. 123.