ELSEVIER
Microelectronic Engineering 61–62 (2002) 449–454
MICROELECTRONIC ENGINEERING
www.elsevier.com/locate/mee

Fabrication of Si-based nanoimprint stamps with sub-20 nm features

I. Maximov*, E.-L. Sarwe, M. Beck, K. Deppert, M. Graczyk, M.H. Magnusson, L. Montelius

Solid State Physics, Lund University, Box 118, S-22100, Lund, Sweden

Abstract

We present two alternative methods for fabrication of nanoimprint lithography stamps in SiO_2 with sub-20 nm features: (a) optimized electron beam lithography (EBL) and lift-off patterning of a 15-nm thick Cr mask, and (b) aerosol deposition of W particles in the 20-nm size range. In both cases, the pattern transfer into SiO_2 was performed using reactive ion etching (RIE) with CHF_3 as etch gas. In the first approach, we used a double layer resist system (PMMA/ZEP 520A7 positive resists) for the EBL exposure. Resist thickness, exposure dose and development time were optimized to obtain 15–20 nm features after Cr lift-off. In the second approach, we used size selected W aerosol particles as etch masks during etching of SiO_2. Both methods of stamp fabrication are compared and discussed. © 2002 Published by Elsevier Science B.V.

Keywords: Electron beam lithography; Aerosols; Etching; Nanoimprint

1. Introduction

Nanoimprint lithography (NIL) is an inexpensive parallel lithography method with capability to produce sub-10 nm features [1]. Nanoimprint lithography can also be used for high resolution patterning of wafers up to 6 inches in diameter [2]. The NIL process is based on a stamp being pressed into a polymer layer heated above its glass transition temperature followed by cooling and removal of the stamp. The direct result of NIL is a relief pattern in the polymer layer which can be further processed to transfer the pattern to the substrate, e.g via lift-off, wet or dry etching.

The resolution of NIL is determined by the feature sizes on the stamp together with the control of the imprinting process. The quality and resolution of the imprinted pattern can never be better than the corresponding characteristics on the stamp. In this work, emphasis will be on stamp fabrication and how to reduce feature sizes on it.

*Corresponding author.
E-mail address: ivan.maximov@ftf.lth.se (I. Maximov).

0167-9317/02/$ – see front matter © 2002 Published by Elsevier Science B.V.
PII: S0167-9317(02)00488-4

A widespread choice of stamp material is Si with an oxide layer on top. This material system is well known and the technologies used for processing are mature. Therefore, it is possible to concentrate on finding ways to optimize the fabrication of small structures. We have chosen to produce the relief in the oxide layer by reactive ion etching (RIE), so the primary concern is to find a stable etch mask with high resolution. Because of the etch chemistry, the obvious choice is metal. The issue is then to achieve small features in the sub-100 nm range. Electron beam lithography (EBL) is a well-known technique for patterning in that regime and combined with metal lift-off it is a powerful tool in NIL stamp production. To achieve really small (below 20 nm) structures optimization of the process is needed and results are presented below. We have also investigated the use of aerosol particles as etch masks and it seems to be a competitive alternative since the deposition process is capable of producing very small particles covering large areas.

2. Experimental

Two alternative methods were used for creation of etching masks in the stamp fabrication process. The first method used high-resolution electron beam lithography, thermal evaporation of Cr and lift-off. The second method relied on gas phase generation of W-aerosol particles and their deposition on the SiO_2 surface.

For the EBL patterning we used a converted JSM 6400 scanning electron microscope (SEM) with LaB_6 cathode. The ELPHY III pattern generator (Raith GmbH) was used to control the electron beam during exposure. The EBL system was operated at 35 kV with an electron probe current of 10 or 20 pA. Single pixel dots and single pixel lines were exposed in a writing field of 100×100 μm^2 with 16-bit resolution.

As substrate we used one inch Si wafers with a 300-nm thick thermally grown oxide. After cleaning the wafer in trichloroethylene, acetone and isopropanol, a double layer resist system was deposited by spinning. The bottom layer consisted of a 100-nm thick positive resist ZEP-520A7 (Nippon Zeon). The second layer was 60 nm of 950 K PMMA. After deposition of ZEP-520A7, the wafer was baked at 160 °C for 10 min using a hot-plate. Additional baking at 160 °C for 15 min was performed after deposition of PMMA. Following the EBL exposure, the resists were first developed in a 1:3 mixture of methylisobutylketone/isopropanol (MIBK/IPA) and then in *o*-xylene for 2 min. Immediately after development, 15 nm thick Cr was thermally evaporated and lifted-off in *n*-methylpyrrolidone under ultrasonic agitation.

The well-defined nanometer-sized tungsten aerosol particles of this investigation were synthesized by an aerosol technique. This technique, described in detail elsewhere [3], allows the control of density, shape and size of the particles. After a size selection in a differential mobility analyzer (DMA), particles with a nominal diameter of 20–30 nm were deposited onto the substrate in an electric field. One inch Si wafers with a 300-nm thick thermal oxide were used as substrates. The particle density was about $3–8 \times 10^8$ cm^{-2}. The carrier gas throughout the aerosol process was purified nitrogen.

All samples were further processed in a conventional parallel plate 13.56 MHz reactive ion etching (RIE) reactor where CHF_3 was used as etch gas. The flow of CHF_3 and the pressure in the chamber were kept constant at 65 sccm and 16 mTorr, respectively. The plasma power and etch time were adjusted to achieve suitable pattern profiles and etch depths. The etch rate of SiO_2 was 40–50 nm/min for both W and Cr masks.

3. Results and discussion

The two-layer resist system used in our experiments consists of ZEP-520A7 as a bottom layer and PMMA as a top layer. Because of the difference in sensitivity of those resists (≈ 50 and ≈ 200 $\mu C/cm^2$ for ZEP-520A7 and PMMA, respectively) and the use of a two-step development process, it was possible to form a well defined interface between the resists. The development of ZEP-520A7 in *o*-xylene was found not to affect the geometry of the PMMA layer, so the size of the exposed features was determined by the development time of PMMA. The higher sensitivity of ZEP-520A7 compared to PMMA resulted in a sharp negative resist profile after development, which made it suitable for a reliable lift-off process. SEM investigation of arrays of single pixel dot structures with dot-to-dot distances of 1 μm, 500 and 200 nm were used for optimization of the EBL exposure. Probe current, PMMA development time and exposure dose (single pixel dot dwell time) were used as optimization parameters. The SEM measurements indicate that for 20 pA probe current a decrease of the dwell time from 130 to 110 μs results in a decrease of Cr disc diameter from ≈ 25 to 15–20 nm for 30 s PMMA development. The 60-s development time does not lead to any noticeable decrease of the disc size. A dwell time below 110 μs results in underexposure of the resist. To find out the effects of probe current on single pixel size, 10 pA probe current was used for exposure. SEM data show that discs with diameter below 20 nm can be obtained using PMMA development times of 45 and 30 s. In this case the minimum dwell time was found to be 180 μs, somewhat lower than the value of 220 μs, calculated from exposure results with 20 pA current. From these results, it can be seen that the PMMA development time has a stronger effect on the size of the exposed features than the probe current. The Cr thickness was 15 nm in all cases, which was enough to serve as a mask during the reactive ion etching of SiO_2. Metal masks were also created using deposition of W-aerosol particles with a well-defined size in the range of 10–60 nm. Unlike the EBL and lift-off technique, the aerosol particles are distributed randomly over the surface (Fig. 1), with a surface density of up to 10^9 cm^{-2}, which is determined by the deposition time. Advantages of the aerosol generation of the etching masks are the parallel process of generation of the particles and their narrow size distribution (about 10%). A drawback with the current aerosol set-up is that the particles are distributed in a homogeneous but random fashion on the substrate not allowing the production of particle patterns. However, a new method has been developed that, in a simple way, creates patterns of surface charge on the substrate, which in turn focus, the particles to a desired position [4]. Alternatively, one can use the atomic force microscope to manipulate and position the aerosol particles [5]. With the future use of those methods, we will be able to create patterns of nanoparticles and thus patterns of etched structures.

Both lithographically defined discs and aerosol particles were used as masks in reactive ion etching to form nanoimprint stamps with sub-20 nm features. The 15-nm thick Cr mask was found to be very stable in the CHF_3 chemistry. Etch selectivity between Cr and silicon dioxide was high enough to produce 150 nm high pillars in SiO_2 (Fig. 2). The etching experiments with W-aerosol masks using

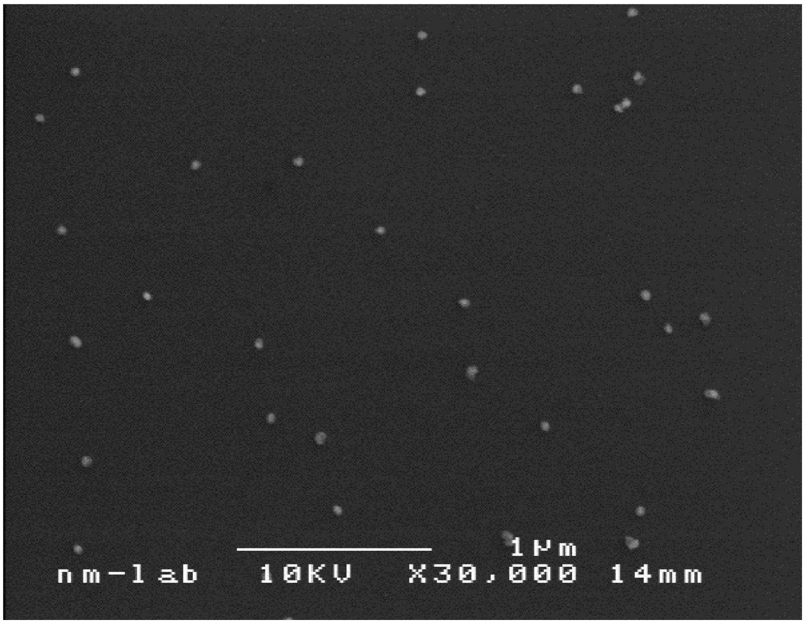

Fig. 1. SEM picture of W-aerosol particles (diameter ≈ 25 nm) deposited onto a SiO_2/Si substrate with surface density of $\approx 4 \times 10^8$ cm^{-2}. Particles are used as etch mask during RIE of silicon dioxide.

the same conditions showed that it is possible to form ≈ 100 nm high SiO_2 pillars with the same surface density as the density of the particles (Fig. 3). The diameter of the pillars is about the same as the diameter of the W-aerosols. The boiling point of WF_6 is much lower (17.6 °C) than CrF_2

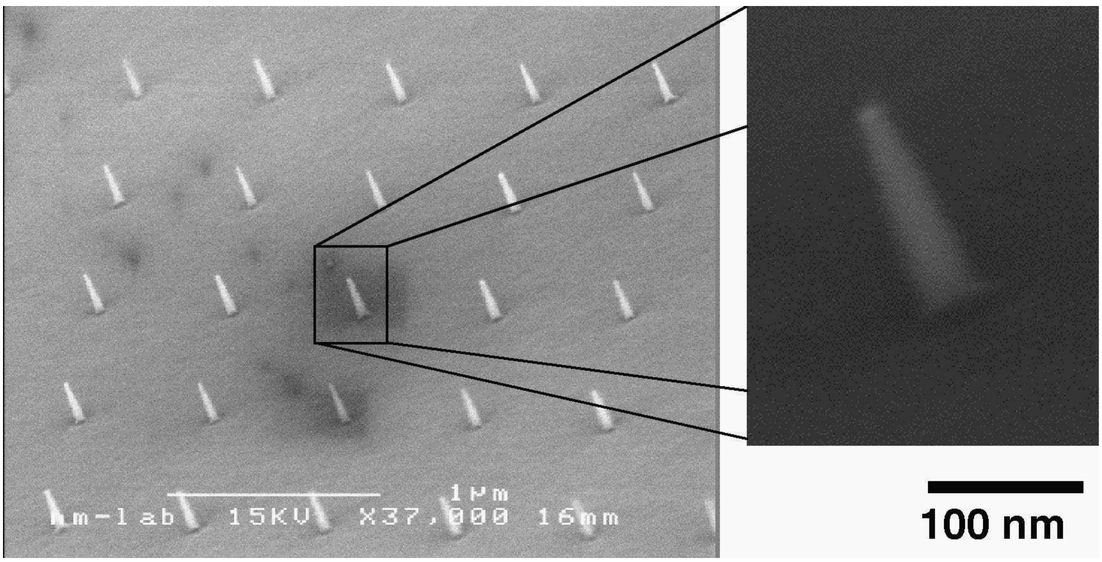

Fig. 2. A feature on SiO_2 nanoimprint stamp showing single pixel dot structures produced by EBL, Cr lift-off and RIE. The upper part of the pillar is less than 20 nm wide.

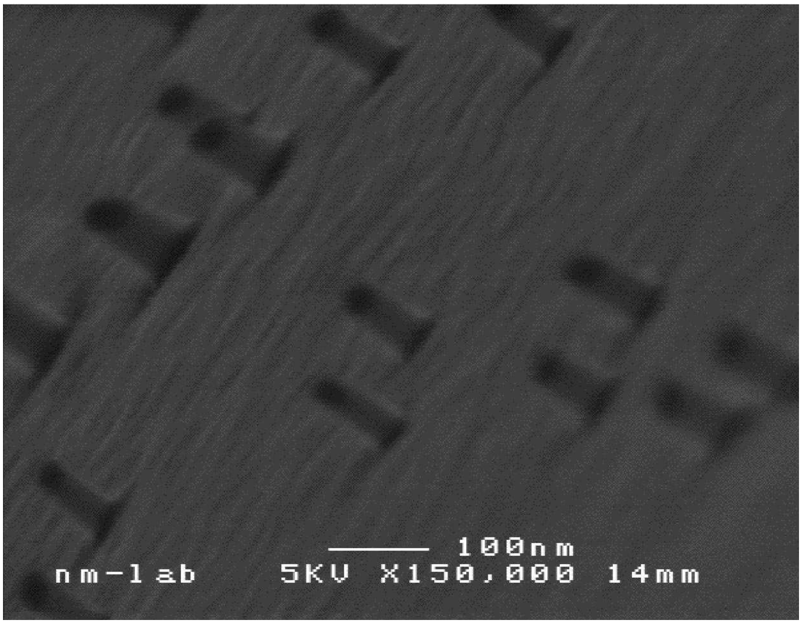

Fig. 3. Nanoimprint stamp in SiO_2 fabricated by deposition of aerosol W-particles and reactive ion etching in CHF_3. The diameter of the columns is 20–30 nm. W-aerosols are visible on top of the pillars (dark parts).

(>1300 °C) [6], so the plasma stability of the W-particles can be expected to be lower than the Cr discs. The SEM investigation of the etched structures indicated the presence of W on top of the pillars, which can be explained by formation of a protective polymeric layer on W-aerosols, similar to what has been observed in CH_4/H_2-based plasma etching of InP with Ag aerosol particles [7].

4. Conclusions

We have demonstrated two alternative approaches for fabrication of NIL stamps with sub-20 nm features using EBL-defined Cr masks or aerosol W-particles. An optimized double-layer resist system allowed a reliable formation of Cr etching masks 15–20 nm in diameter, while gas-phase generation of W-aerosols can produce particles as small as 10 nm. The RIE experiments showed that both Cr and W masks are stable in a CHF_3 plasma and resulted in formation of sub-20 nm silicon oxide pillars after etching.

Acknowledgements

This work is performed within the Nanometer Consortium and funded by the European Community in the CHANIL project (IST-1999-13415).

References

[1] S.Y. Chou, P.R. Krauss, W. Zhang, L. Guo, L. Zhuang, J. Vac. Sci. Technol. B 15 (1997) 2897.
[2] B. Heidari, I. Maximov, L. Montelius, J. Vac. Sci. Techol. B 18 (2000) 3557.
[3] M.H. Magnusson, K. Deppert, J.-O. Malm, J. Mater. Res. 15 (2000) 1564.
[4] T.J. Krinke, H. Fissan, K. Deppert, M.H. Magnusson, L. Samuelson, Appl. Phys. Lett. 78 (2001) 3708.
[5] T. Junno, K. Deppert, L. Montelius, L. Samuelson, Appl. Phys. Lett. 66 (1995) 3627.
[6] N.G. Einspruch, D.M. Brown (Eds.), VLSI Electronics Microstructure Science, Plasma Processing for VLSI, Vol. 8, Academic Press, London, 1984.
[7] I. Maximov, A. Gustafsson, H.-C. Hansson, L. Samuelson, W. Seifert, A. Wiedensohler, J. Vac. Sci. Technol. A 11 (1993) 748.