

Sub-10-nm nanolithography with a scanning helium beam

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(Received 13 April 2009; accepted 23 June 2009; published 24 July 2009)

Scanning helium ion beam lithography is presented as a promising pattern definition technique for dense sub-10-nm structures. The powerful performance in terms of high resolution, high sensitivity, and a low proximity effect is demonstrated in a hydrogen silsesquioxane resist. © 2009 American Vacuum Society. [DOI: 10.1116/1.3182742]

I. INTRODUCTION

With the ongoing trend toward miniaturization and ever higher device performance, the need for controlled realization of sub-10-nm dimensions is becoming increasingly important. For several decades, the mainstream lithography of choice for defining nanostructures has been electron beam lithography. In a long sequence of developments (e.g., the field emission gun, 100 kV acceleration voltage, $\lambda/4096$ laser interferometry, and 16-bit or higher digital-to-analog converter technology), the electron beam technique has reached the nanometer precision needed for sub-10-nm performance in resist. Rather than the control and accuracy of the beam writing process, the limiting factors are turning out to be the electron-resist interactions in conjunction with the resist development. We propose that scanning helium ion beam lithography (SHIBL) is the next step. In particular, low proximity effects, in combination with the demonstrated high sensitivity and resolution, make SHIBL a promising technology for nanopatterning in the sub-10-nm regime, although He ion impact on semiconductor nanodevice structures may not always be harmless.¹

In the past, focused ion beam lithography (FIBL) has been a somewhat less important player in the field of nanolithography. Most work in the field of FIBL has been performed using either a scanning beam or an ion projection approach, which is based on Ga⁺ ion sources. Even fewer beam studies deal with light ions such as Be⁺, H⁺, H₂⁺, and He⁺.²⁻⁴ An early overview was given by Melngailis.⁵ Besides the advantages of higher sensitivity and lower proximity effects⁶ compared to the electron beam approach, major disadvantages of the earlier FIBL work are a lower resolution due to a larger beam diameter and (certainly with respect to Ga⁺ ion exposure) potential damage or contamination from the ion impact (Ga implantation⁷). High-resolution FIB tools have been

available for about 2 decades. Features including a 12–15 nm linewidth in poly(methyl methacrylate) (PMMA) resist⁸ and a 30 nm dot size in polyphenylsilsesquioxane resist⁹ have been reported. Today, the smallest probe size of Ga⁺ ion beams is typically around 5 nm, and direct engraving in a 20 nm thick membrane material yields nanopores as small as 3 nm in diameter.¹⁰

Recently, scanning helium ion microscopy¹¹ with a helium probe size of 0.75 nm in diameter was launched in the market. In the work described here, we used the scanning He⁺ ion beam setup as a beam writing tool for nanolithography and compare its performance with electron beam exposure behavior. The expected advantages of SHIBL over electron beam lithography (EBL) comprise of reduced proximity effects and the ability to write smaller features given the subnanometer probe size. The former is due to a more directional scattering profile of He⁺ ions and the different secondary electron (SE) generation mechanisms with ions in resist, which causes a high yield of slow SEs.¹¹⁻¹⁴ Altogether, these characteristics greatly suppress the blurring background that arises when writing dense patterns with EBL.

II. EXPERIMENT

A Carl Zeiss OrionTM Plus scanning helium ion microscope and an FEI Strata DB 235 scanning electron microscope (SEM), both operating at 30 kV, were used for helium ion beam and electron beam exposures, respectively. The FEI setup and a Hitachi S4800 SEM were used for the inspection of the developed structures. Especially, the latter imaging setup is superior in contrast and resolution due to its sophisticated system to handle the secondary and backscattered electrons. A concise discussion of high-resolution scanning electron and helium ion microscopy was published recently.¹⁵ Dose variations were achieved by means of the beam current and by controlling the dwell time per pixel, which varied from 20 to 2000 μ s.

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This work deals with exposures in hydrogen silsesquioxane (HSQ) resist from Dow Corning (FOX-12 product) on silicon substrates. Resist thickness was achieved by tuning the spin speed (Karl Suss RC 5/8) and by appropriate dilution of HSQ with methyl isobutyl ketone. Typically, 1:10 and 1:1 dilutions are used for thicknesses of 5 and 55–70 nm, respectively. All resist films were dried for 30 min in vacuum at room temperature in order to minimize film roughness.¹⁶

Two types of experiments were conducted. In the first type, the exposure sensitivity is measured for both EBL and SHIBL in HSQ films of ~ 70 nm thick using a defocused beam in a dose range from $0.1 \mu\text{C}/\text{cm}^2$ to $1 \text{ mC}/\text{cm}^2$. Patterns used for that purpose were $50 \times 50 \mu\text{m}^2$ squares. After development in MF 322 developer (Rohm & Haas) for 1 min, the height of the structures was measured by means of profilometry (*Tencor*). Sensitivity values were obtained as the dose D_s necessary to achieve 50% of its original thickness after development. Contrast values were determined according to the procedure of Thompson.¹⁷

In the second type of experiments, high-resolution He^+ beam spot exposures were performed in 5 and 55 nm thick layers of HSQ. Exposures were performed with a fine probe size, a 1 pA beam current, and a dwell time of $100 \mu\text{s}$ at $20 \mu\text{m}$ aperture and 7 mm working distance. A single imaging raster scan was used, resulting in the formation of arrays of dots with a variable pitch. The field of view (FOV) was $25 \times 25 \mu\text{m}^2$ and the numbers of pixels were 256×256 , 512×512 , and 1024×1024 , resulting in pitches of 98, 47, and 24 nm, respectively. In one exposure, the FOV was $15 \times 15 \mu\text{m}^2$ with 1024×1024 pixels, resulting in a pitch of 14 nm. A droplet of Au nanoparticles (~ 20 nm in diameter) was placed directly on the resist surface, which allowed the ion beam to be focused accurately *in situ* prior to the actual writing in resist. After development for 5 min (MF351 from Rohm & Haas) and immersion in a “stopper” solution (MF351:H₂O=1:9), samples were rinsed with de-ionized water and blown dry in nitrogen.

III. RESULTS AND DISCUSSION

A. Contrast and sensitivity

Residual HSQ film thickness dependencies after development on helium ion beam and e-beam exposure dose are shown in Fig. 1. The results demonstrate that HSQ resist is 4.4 times more sensitive for helium ions ($D_s = 31 \pm 3 \mu\text{C}/\text{cm}^2$) than for electrons ($D_s = 137 \pm 5 \mu\text{C}/\text{cm}^2$), whereas the contrast values are practically the same ($\gamma = 2.1 \pm 0.1$) for both types of exposure. The difference in sensitivity between SHIBL and EBL can be partly due to the higher yield of SE for helium ions compared to electrons of the same energy.¹¹ Additionally, the resist sensitivity is further enhanced because in He^+ ion exposure the fraction of low-energy SE is higher. The same contrast for both EBL and SHIBL indicates similar molecular weight distributions of cross-linked resist monomers. It is solely the yield and energy distribution of the SE that differ. The higher sensitivity of HSQ for He^+ than for electron ex-

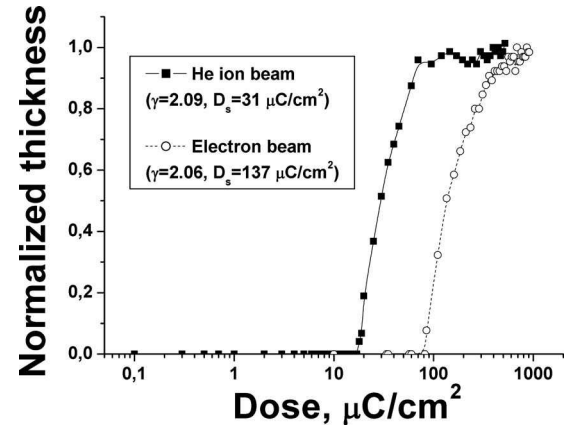


FIG. 1. Normalized thickness of HSQ resist film as a function of dose for helium ion beam and electron beam exposures at 30 keV. The thickness was normalized prior to the development. The sensitivity D_s and contrast values γ_s are shown in the inset.

posure is relatively moderate compared to the sensitivity enhancement by factors of 100–300 in PMMA under Ga^+ , He^+ , H^+ , and Ar^+ exposure¹⁸ and 16–50-fold enhancement in a range of other resist products under He^+ ion projection lithography.¹⁹ Recent work on H^+ beam writing of HSQ at MeV ion energy exposure shows a sensitivity of $3.2 \mu\text{C}/\text{cm}^2$,²⁰ which drops to about $20 \mu\text{C}/\text{cm}^2$ sensitivity upon HSQ resist aging. The difference between the values from these studies and those obtained in our experiment with helium ions could be due in part to differences in experimental conditions. Beam type and energy, initial thickness,²¹ development time, developer type, concentration, and temperature²² have been known to have a substantial impact on the sensitivity of the resist. However, additional impact from a different exposure mechanism cannot be ruled out.

B. Minimal feature size and proximity effect

SEM images of an array of isolated dots at a pitch of 98 nm in 5 and 55 nm thick HSQ films are shown in Figs. 2(a) and 2(b), respectively. The average dot diameters are 6 ± 1 and 14 ± 1 nm, respectively. These results prove the He^+ ion beam capability for nanostructuring in ultrahigh resolution mode. In both cases the exposure dose ($100 \mu\text{s}$ dwell time per pixel) and the development time (5 min) were the same.

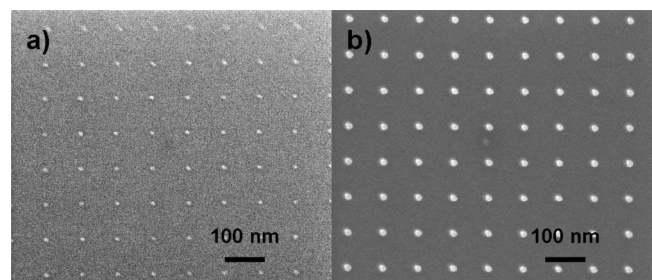


FIG. 2. SEM images of arrays of dots written in (a) 5 nm and (b) 55 nm thick HSQ layers at 98 nm pitch using scanning helium ion beam lithography. Field of view is 900 nm in SE mode at 20 kV. Average dot diameters: (a) 6 ± 1 nm and (b) 14 ± 1 nm.

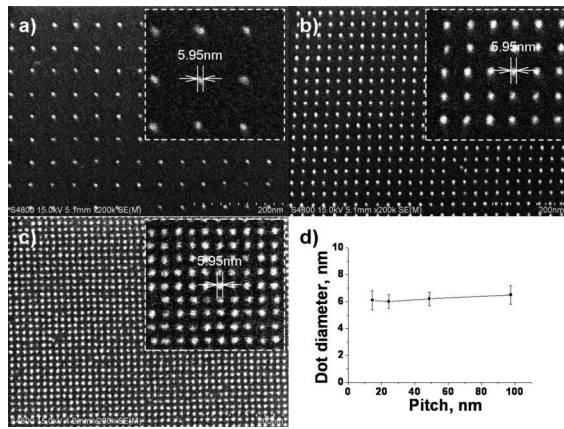


FIG. 3. SEM images of arrays of dots written in a 5 nm thick HSQ layer using SHIBL at pitches of (a) 48 nm, (b) 24 nm, and (c) 14 nm, and (d) the average dot size vs pitch. The insets are SEM images at a higher magnification. The average dot size for all pitches is 6 ± 1 nm.

Nevertheless, the dot size for the thicker layer is about twice as large as the thinner one. This observation corresponds with previous results for electron beam exposure of HSQ films of different thicknesses. Lines 2 pixels wide written with an e-beam in 5 and 55 nm thick HSQ have linewidths of 11 and 24.5 nm, respectively,²¹ when processed under identical exposure and development conditions (but different from the current He ion experiments). To our knowledge, the best results in e-beam exposure of HSQ are currently 6 nm lines on a pitch of 20 nm written at 100 keV.²³

The dot size as a function of pitch is shown in Fig. 3. Remarkably, an identical dot size of 6 ± 1 nm was achieved down to a pitch of 14 nm [see Fig. 3(d)]. This observation indicates an undetectably small proximity effect for helium ion beam exposure. Several factors may be involved in this low proximity effect. First, the scattering profile of helium ions is known to be confined within a narrow cone penetrating relatively deep into the material with very low backscattering.¹¹ Second, helium ions produce mostly low-energy SE (<20 eV, when derived from H⁺ data on CO₂, scaled to the same velocity¹⁴), which do not travel far in the resist.

Finally we need to address two other aspects in this work. First, the exposure dose of 1 pA for 100 μ s corresponds to about 600 ions/dot. The inherent shot noise implies a dose uncertainty of 4%/dot. Although the insets in Figs. 3(a)–3(c) show dot-to-dot size irregularities, a possible relation to shot noise is not likely, given the contrast curve in Fig. 1. The observed nanometer scale irregularities touch on the second aspect related to the minimum achievable feature size in HSQ resist, where several factors play a role. Irrespective of the writing probe size, HSQ studies in literature²³ and this work indicate absolute lower limits of about 5–6 nm for HSQ structures. Adhesion to the substrate becomes too low with decreasing feature size in order to withstand the force interactions during the wet development step. In addition, the number of cross-linked resist monomers decreases with shrinking feature size. Consequently, at some minimum feature size the contrast in the exposed area is too low to “sur-

vive” the development. Future work to test this size limit hypothesis could deploy special patterns, wherein the ultrafine features are attached to larger scale structures that will provide sufficient mechanical stability. With its negligible proximity effect, SHIBL seems to be the best exposure technology by far to realize the ultimate limit in this matter.

IV. CONCLUSION

Scanning He⁺ ion beam lithography on HSQ is demonstrated to have very a high resolution and a superior low proximity effect. Furthermore, He⁺ ion exposure is several times more effective than electron beam exposure at the same acceleration voltage, whereas the contrast is equal. Overall, He⁺ ion beam lithography is a very promising technique for the formation of ultrahigh resolution structures of a high density and having feature sizes in the sub-10-nm range.

ACKNOWLEDGMENTS

This research is part of NanoNed, a national research program on nanotechnology funded by the Ministry of Economic Affairs in The Netherlands. Ping Cheng, Kees Hagen, and Pieter Kruit are acknowledged for their contributions to this work.

- ¹E. van der Drift, R. Cheung, and T. Zijstra, *Microelectron. Eng.* **32**, 241 (1996).
- ²C. W. Slayman, J. L. Bartelt, and C. M. McKenna, *Proc. SPIE* **333**, 168 (1982).
- ³J. N. Randall, D. C. Flanders, N. P. Economou, J. P. Donnelly, and E. I. Bromley, *J. Vac. Sci. Technol. B* **1**, 1152 (1983).
- ⁴I. Adesida, C. Anderson, and E. D. Wolf, *J. Vac. Sci. Technol. B* **1**, 1182 (1983).
- ⁵J. Melngailis, *Nucl. Instrum. Methods Phys. Res. B* **80-81**, 1271 (1993).
- ⁶S. Matsui, Y. Kojima, Y. Ochiai, and T. Honda, *J. Vac. Sci. Technol. B* **9**, 2622 (1991).
- ⁷D. C. Ferranti, J. C. Morgan, W. B. Thompson, and W. C. Joyce, *Proc. SPIE* **2194**, 394 (1994).
- ⁸R. L. Kubena, J. W. Ward, F. P. Stratton, R. J. Joyce, and G. M. Atkinson, *J. Vac. Sci. Technol. B* **9**, 3079 (1991).
- ⁹R. L. Kubena, R. J. Joyce, J. W. Ward, H. L. Garvin, F. P. Stratton, and R. G. Brault, *J. Vac. Sci. Technol. B* **6**, 353 (1988).
- ¹⁰J. Gierak *et al.*, *Microelectron. Eng.* **84**, 779 (2007).
- ¹¹J. Morgan, J. Notte, R. Hill, and B. Ward, *Microscopy Today* **14**, 24 (2006).
- ¹²R. Ramachandra, B. Griffin, and D. Joy, *Ultramicroscopy* **109**, 748 (2009).
- ¹³S. Matsui, Y. Kojima, and Y. Ochiai, *Appl. Phys. Lett.* **53**, 868 (1988).
- ¹⁴W. Q. Cheng, M. E. Rudd, and Y. Y. Hsu, *Phys. Rev. A* **40**, 3599 (1989).
- ¹⁵A. Vladar, M. Postek, and B. Ming, *Microscopy Today* **17**, 6 (2009).
- ¹⁶V. Sidorkin, A. Grigorescu, H. Salemink, and E. van der Drift, *Microelectron. Eng.* **86**, 749 (2009).
- ¹⁷L. F. Thompson, *Solid State Technol.* **17**, 27 (1974).
- ¹⁸H. Ryssel, K. Habeger, and H. Kranz, *J. Vac. Sci. Technol.* **19**, 1358 (1981).
- ¹⁹S. Hirscher, R. Kaesmaier, W.-D. Domke, A. Wolter, H. Loschner, E. Cekan, C. Horner, M. Zeininger, and J. Ochsenhirt, *Microelectron. Eng.* **57-58**, 517 (2001).
- ²⁰J. A. van Kan, F. Zhang, C. Zhang, A. A. Bettiol, and F. Watt, *Nucl. Instrum. Methods Phys. Res. B* **266**, 1676 (2008).
- ²¹V. A. Sidorkin, A. van Run, A. van Langen-Suurling, A. Grigorescu, and E. van der Drift, *Microelectron. Eng.* **85**, 805 (2008).
- ²²J. Yang and K. K. Berggren, *J. Vac. Sci. Technol. B* **25**, 2025 (2007).
- ²³A. E. Grigorescu, M. C. van der Krogt, C. W. Hagen, and P. Kruit, *J. Vac. Sci. Technol. B* **25**, 1998 (2007).