

Technique for preparation of precise wafer cross sections and applications to electron beam lithography of poly(methylmethacrylate) resist

Wenchuang Hu,^{a)} Tatyana Orlova, and Gary H. Bernstein

Department of Electrical Engineering, University of Notre Dame, Notre Dame, Indiana 46556

(Received 28 May 2002; accepted 9 September 2002)

We have developed a method of cross-sectioning silicon wafers with high placement precision. It is implemented by using optical lithography and deep plasma high aspect ratio etching of a thin line, which is used as the breaking axis. This technique is then applied to cross-sectional scanning electron microscopy (SEM) studies of sub-50 nm electron beam lithography in (poly)methylmethacrylate resist. We also report the development of a process for protecting the resist during SEM examination. © 2002 American Vacuum Society. [DOI: 10.1116/1.1518020]

I. INTRODUCTION

Cross-sectional scanning electron microscopy (XSEM) is widely used as fast and efficient feedback for diagnosing very large scale integrated circuit (VLSI) failure and process problems.¹ It is also a powerful tool to study materials, films² and nanofabrication processes.³ In particular, XSEM of electron beam lithography (EBL) structures in organic resists is widely used to study and optimize the whole EBL process, which has been intensively used in mask making, Coulomb blockade devices^{4,5} and nanotechnology fabrication.⁶ As the fabrication resolution scales down to the nanometer region, XSEM study of the processes requires higher quality and less contamination to the cross section and top surface of the specimens. How to obtain high quality, site-specific cross sections for XSEM becomes an important issue.

There are several cross-section preparation methods reported and widely used: scribing and cleaving, precision microcleaving, wedge polishing,^{7,8} and focused ion beam (FIB) techniques.⁹ Most of these methods involve direct cleaving or polishing, which will damage the very edge of cross sections and introduce contamination. Also, some of them have precision limitations. FIB techniques are capable of site-specific precision cleaving,^{10,11} but are sophisticated and highly equipment dependent. Also, the incident ions can introduce damage and contamination to the sample. It is reported that the 20 nm top layer of sample surface or sidewalls is damaged by Ga⁺ ions.¹² This is a vital problem for nanoscale, thin, cross-section studies such as the study of nanoscale EBL in organic resists. However, the cross-section preparation method we have developed has overcome these drawbacks. It enables us to break our samples along the desired axis (site specific) with micron precision and gives complete cross sections allowing perfect edge-on viewing with the SEM, at any angle, without introducing chemical contamination or physical damage to the edges. It is especially useful for XSEM studies of EBL structures because of the ability to prepare cross sections through tiny areas. It is also applicable to general wafer precision breaking for prototyping traditional semiconductor devices or investigating associated fabrication processes.

II. METHOD PROCEDURE

The precise breaking of wafers or small samples was accomplished by deep plasma etching of a 5–10- μm -wide line in an inductively coupled plasma etcher using the Bosch process (Alcatel 601E). The wafers used were 200 and 500 μm thick and were broken along the etched scribe lines with proper allowances for nanometer EBL regions. The following process is illustrated in Fig. 1(a): Design a photomask for optical lithography. The line pattern is throughout the whole sample with opened areas of interest in the middle. The line is 10 μm wide and 1 mm to several cm long, depending on sample size. Alignment marks are used there for EBL or device fabrication. In Figs. 1(b) and 1(c), after optical lithography of the line with 100 μm unexposed sections, the pattern is etched to a depth of 90 μm or more using the Bosch high aspect ratio etching technique with 2- μm -thick AZ5214 resist as etch mask. After removing the AZ resist [Fig. 1(d)], 60–200 nm thick, 950 000 D (poly)methylmethacrylate (PMMA) is applied to the surface, and EBL is performed on the unetched area in the line. In Fig. 1(e) a simple fixture consisting of two glass plates is used to break precisely along the line. A mini metal bender can make the breaking easier to operate. We found that a 500- μm -thick wafer can be broken with a 100- μm -deep etched line at 90% success rate, but the 200 μm wafer is easier to break and gives more consistent results. If using a 200- μm -thick Si wafer, the lattice direction will affect the breaking axis significantly. When doing optical lithography, the breaking lines should be aligned roughly parallel to the Si wafer lattice direction [100]. In Fig. 1(f) the broken etched line interface with PMMA pattern was imaged with our Hitachi S-4500 cold cathode field emission SEM (FESEM). The original pattern and broken line interface is shown in Fig. 2. This technique is especially useful for high-resolution lithography over small scan fields where large patterns with long exposure times are not practical. For general wafer or sample breaking, one can replace the EBL step with an associated fabrication process, accordingly.

The deep plasma etching of the silicon substrate is a key process in this technique. The etcher we used is an Alcatel 601E. Its inductively coupled plasma source utilizes the Bosch process and a 2 KW, 13.45 MHz rf source. Clamping of the wafer on the chuck allows backside cooling as well as

^{a)}Electronic mail: whu@nd.edu

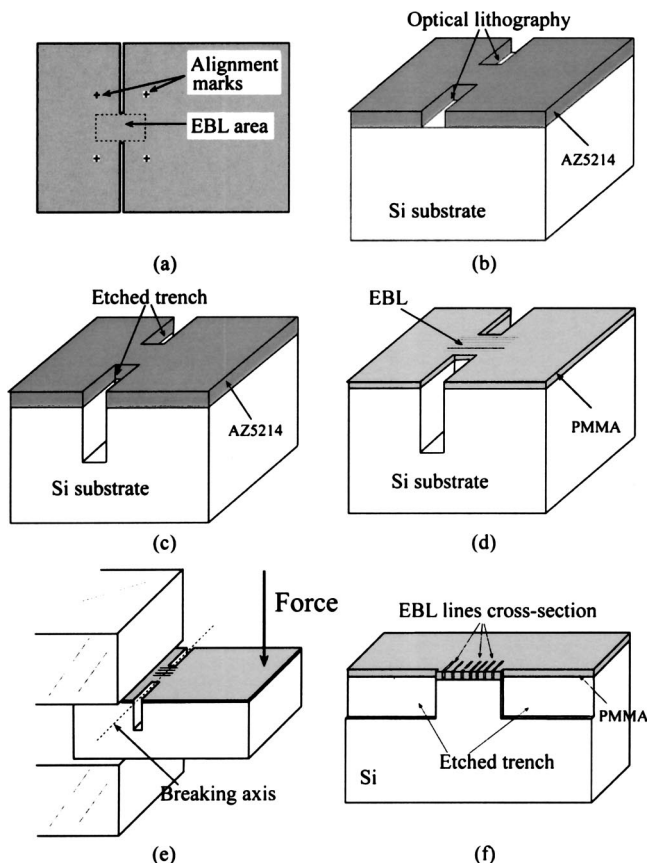


FIG. 1. Schematic illustration of our precise wafer breaking technique (not drawn to scale). (a) Mask design of a 10- μm -wide line with 100 μm broken area for EBL. (b) Optical lithography using 2- μm -thick AZ5214 resist, (c) exposed line was etched down by 100 μm , (d) after removing AZ5214, 60-nm-thick PMMA layer is spanned on and EBL was done in the interest area. (e) Sample was broken along desired axis by pressing one end with the other fixed. (f) Cross sections were imaged by FESEM in any angle.

biasing during etching by a 500 W LF 50-460 KHz rf source. Before we start the standard Bosch process, the chamber is turbo-pumped to 3.5E-5 Pa and a delay of 5 min ensures constant substrate temperature of 20 °C. The standard Bosch process consists of alternating SF₆ for etching and C₄F₈ for passivation in pulse mode, which means in one consecutive etching cycle the SF₆ gas flows at a rate of 300 sccm for 7 s with the C₄F₈ gas valve closed and then the C₄F₈ gas flows at a rate of 130 sccm for 2 s with the SF₆ gas valve closed. The rf source power in this process is set to 1800 W, and the backside biasing during etch is 80 W. We found that 20 min etching gave us 100 μm depth of the 10- μm -wide thin line in silicon. The aspect ratio of this process is at least 10. We can easily achieve 60- μm -deep and 5- μm -wide lines, which results in high precision and a high success rate for our breaking technique. With further improvement of this high aspect ratio etching process, we expect to be able to place the cross-section area to better than 1 μm accuracy.

High-resolution EBL was performed using the same Hitachi S-4500 FESEM controlled by a custom pattern generator. This system is capable of ultrahigh resolution patterning with accelerating voltage at 30 kV. A cold field emitter as the electron source generates a high current density electron

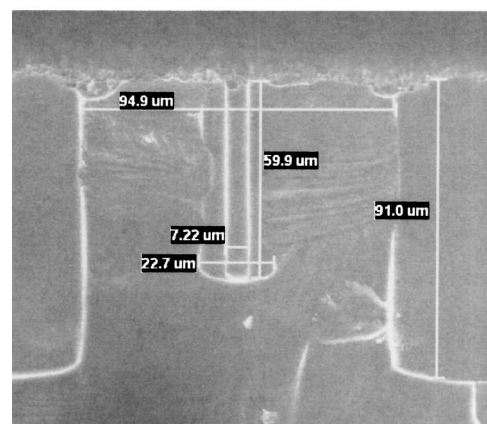
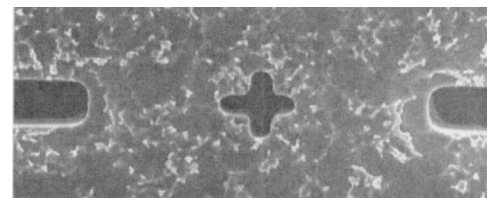


FIG. 2. FESEM of cross-section interface.

beam with diameter less than 2 nm at 30 kV. The resist used is PMMA with molecular weight 950 000 D and 60–200 nm thick on a Si substrate. The developer used was isopropyl alcohol:methyl isobutyl ketone (3:1) with 1.5% methyl ethyl ketone¹³ in an ultrasonic bath¹⁴ for high resolution.

III. CROSS-SECTION STUDY OF EBL IN PMMA

As a sample application of this method, we have performed a XSEM study of EBL in PMMA resist. These cross-section studies of EBL can provide improved information on the nanometer scale about profiles of PMMA resist after development, the attainment of development contrast, and the metal or molecular lift-off process.¹⁵ All this information is very valuable feedback for fabrication process evaluations, monitoring and resist system investigation. For example, our cross-section results of EBL in PMMA tell us directly the EBL depths in different line doses at 30 kV. This information is critical for us to design appropriate PMMA thickness for successful metal or molecular lift-off, which requires clean trench bottoms. Also, the quality of the EBL profiles can be determined for optimizing the lift-off process.

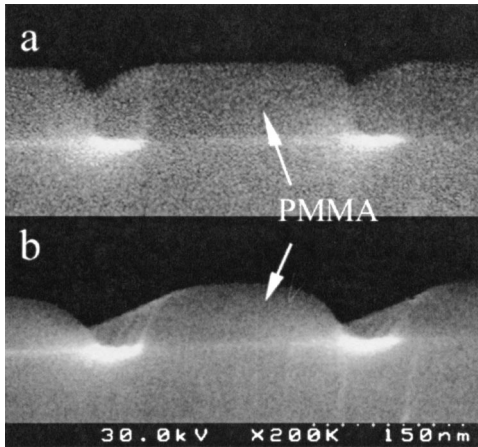


FIG. 3. Electron beam damage to PMMA trenches. (a) Scan-once SEM image of PMMA trenches. Beam current is 15 pA. Scan time is about 1 s. (b) Multiple-scan SEM image of the same area. Scan time is about 5 s.

However, it is difficult to obtain unadulterated PMMA profiles of EBL, especially on the nanometer scale, for several reasons: (1) Cross sections are mostly imaged with SEM, which will quickly and heavily damage the PMMA

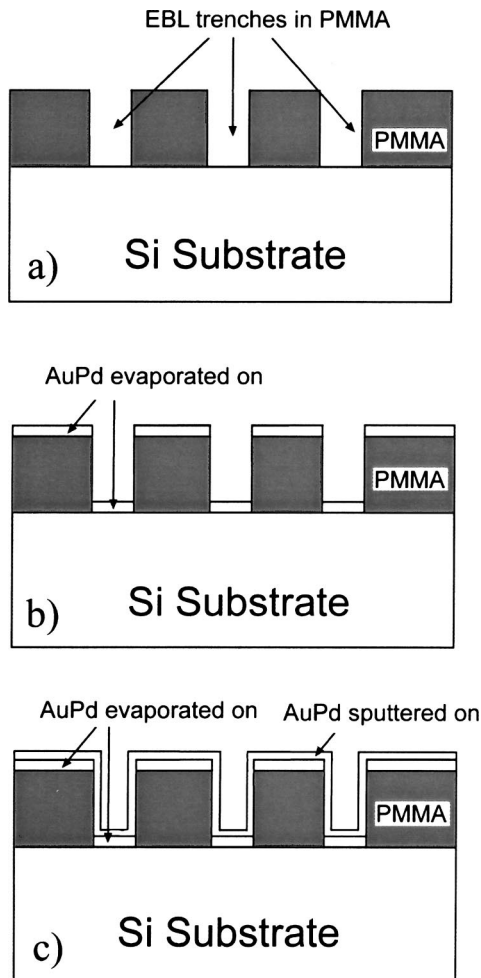


FIG. 4. Optimized process to protect the original PMMA EBL profiles. After (a) EBL and development of PMMA, (b) 5 nm AuPd was deposited using thermal evaporation, then (c) 5 nm AuPd was deposited using sputter coater.

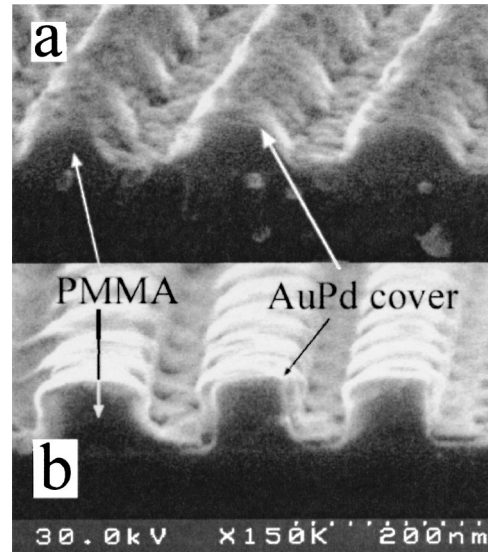


FIG. 5. SEM of PMMA trenches: (a) 10-nm-thick AuPd film was deposited directly using plasma sputter coater, which damaged the trench corners, (b) 10-nm-thick AuPd film was deposited using our optimized process, which protected the trench corner and resulted in original cross sections.

resist and cause significant change to the profiles. Figure 3 illustrates how quickly and significantly the electron beam changed the PMMA trenches. (2) Most cross-section sample preparation techniques will physically damage the very edge or the top surface of PMMA trenches or introduce contamination. (3) Plasma sputter coating, which is used to cover samples with thin metal films (such as AuPd 60:40) for XSEM, will often damage the very corners of sharp PMMA EBL trenches. We developed an optimized process that enables us to see nearly the original PMMA EBL profiles on the nanometer scale. The following process is illustrated in Fig. 4: First, after the EBL process on PMMA, samples are coated with 2–5 nm AuPd (60:40) film using a thermal evaporator. Then, samples are coated with 2–5 nm AuPd (60:40) again using a plasma sputter coater to cover trench sidewalls. The previous AuPd film thermally evaporated protects the corners and top surface of PMMA trenches from heavy damage by the plasma during sputtering. Now, the PMMA trenches are fully covered by a AuPd thin film, and are then broken along the desired axis using our sample breaking technique with the edge and surface perfectly protected. Figure 5 compares the PMMA cross sections made from direct sputter coating and our optimized process.

IV. RESULTS AND DISCUSSION

With this site-specific, precision cross-section preparation technique and PMMA trench-protection process, we have studied the EBL profile of PMMA, particularly the aging effect of PMMA resist.

PMMA resist cross-section profiles are closely related to e-beam exposure condition, development process,¹⁶ storage temperature and humidity. We performed a preliminary investigation of the time evolution of PMMA after lithography and before development, which to our knowledge has not

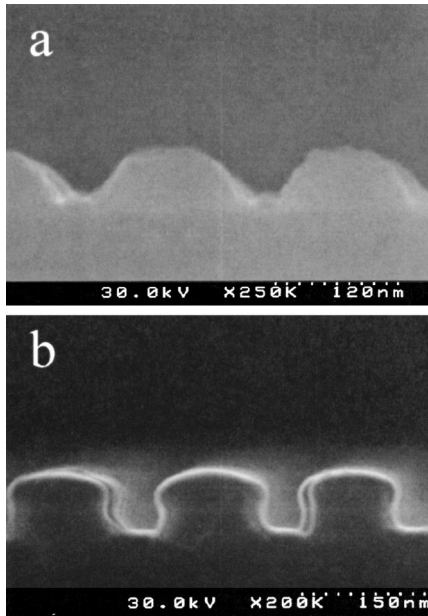


FIG. 6. FESEM images of nanoscale PMMA cross-sectional profiles of EBL. (a) This profile showed that one week aging of PMMA resulted in reduction of contrast compared to (b) profile of PMMA trenches without delay after lithography. In (a) and (b), trenches were covered by 5–10 nm AuPd (60:40) film using our optimized cross-section preparation process for XSEM.

been reported. Our results show that shelf time is capable of affecting resolution and the pattern transfer process. This kind of PMMA aging before development reduces PMMA contrast, widens features, and changes cross-section profiles, and can result in poor lift-off reliability. Figure 6(a) indicates a loss of contrast due to one week aging, and the trench profiles changed significantly compared with Fig. 6(b), which had no delay after lithography. Figures 6(a) and 6(b) were of identical EBL process conditions with the same line dose $2E-9$ C/cm. Future studies will investigate the dependence of time evolution of PMMA after development and before metal evaporation or other pattern transfer process on resolution and fabrication quality.

V. CONCLUSIONS

We have developed a cross-section preparation technique and an optimized cross-section protection process for XSEM imaging. These techniques enabled us to obtain sub-50 nm, clearly resolved cross sections of EBL in PMMA. Also, we have shown some examples of cross-section studies of EBL in PMMA resists. The results for a variety of resists besides PMMA will be studied later. All these studies are believed valuable for a better understanding and improvement of EBL fabrication. The precise breaking technique can be applied to general wafer cross-section preparation, especially useful for SEM cross-sectional studies of EBL related fabrication, thin films, semiconductor devices, and metal or molecular lift-off techniques.

ACKNOWLEDGMENTS

This work was performed with support of ONR/DARPA Grant No. N00014-01-1-0658 and the W. M. Keck Foundation.

- ¹K. Banerjee, D. Y. Kim, A. Amerasekera, C. Hu, S. S. Wong, and K. E. Goodson, 38th IEEE Annual International Reliability Physics Symposium Proceedings, 2000, p. 283.
- ²J. H. Son, M. Y. Park, and S. W. Rhee, *Thin Solid Films* **335**, 229 (1998).
- ³Y. Chen, D. Macintyre, and S. Thoms, *J. Vac. Sci. Technol. B* **17**, 2507 (1999).
- ⁴L. Guo, E. Leobandung, L. Zhuang, and S. Y. Chou, *J. Vac. Sci. Technol. B* **15**, 2840 (1997).
- ⁵W. Chen and H. Ahmed, *J. Vac. Sci. Technol. B* **15**, 1402 (1997).
- ⁶T. D. Happ, A. Markard, M. Kamp, A. Forchel, S. Anand, J.-L. Gentner, and N. Bouadma, *J. Vac. Sci. Technol. B* **19**, 2775 (2001).
- ⁷J. Chen and D. G. Ivey, *Micron* **33**, 489 (2002).
- ⁸H. Li and L. Salamanca-Riba, *Ultramicroscopy* **88**, 171 (2001).
- ⁹E. C. G. Kirk, D. A. Williams, and H. Ahmed, *Inst. Phys. Conf. Ser.* **100**, 501 (1989).
- ¹⁰T. Ishitani and T. Yaguchi, *Microsc. Res. Tech.* **35**, 320 (1996).
- ¹¹L. A. Giannuzzi and F. A. Stevie, *Micron* **30**, 197 (1999).
- ¹²Ni. Kato, K. Tsujimoto, and N. Miura, *Electron Microscopy of Semiconducting Materials and VLSI Devices*, Symposium, 1998, p. 15.
- ¹³G. H. Bernstein, D. A. Hill, and W. P. Liu, *J. Appl. Phys.* **71**, 4066 (1992).
- ¹⁴W. Chen and H. Ahmed, *J. Vac. Sci. Technol. B* **11**, 2519 (1993).
- ¹⁵Q. Hang, Y. Wang, M. Lieberman, and G. H. Bernstein, *Appl. Phys. Lett.* **80**, 4220 (2002).
- ¹⁶K. Murata, E. Nomura, K. Nagami, T. Kato, and H. Nakata, *J. Vac. Sci. Technol.* **16**, 1734 (1979).