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Fabricating Arrays of Si(110)/Si(100) Microstructures by Atom Lithography using Organosilane Self-assembled Monolayers

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Abstract. We reported a monolayer-derived fabrication of Si(110)/Si(100) microstructures arrays over an area of about 2 cm² by atom lithography, in which self-assembled monolayer (SAM) of organosilane (octadecyltrichlorosilane, ODTS; dodecyltrichlorosilane, DDTS) and metastable helium beam (He-MAB) were used to pattern the surface of silicon substrates without coating intermediate layer. ODTS and DDTS SAMs were formed on Si(110)/Si(100) surfaces as resists for exposure to MAB. The hydrophobicity and the durability of the outermost surface of the SAM in the regions exposed to MAB were altered by varying MAB dose of exposure and the length of alkyl chains of SAM molecule. The negative/positive patterns of Si(110)/Si(100) surfaces with depths of 10~220nm and edge step resolutions of 40~500nm were obtained successfully. The effect of a wet etching process on the resolutions was discussed in brief. Our results suggested that the top-down atom lithography combined with bottom-up chemical assembly techniques could contribute significantly to the development of more monolayer-derived micro- and nanofabrication.

1. Introduction

The arrays of micro- and nanostructures of silicon show interesting characteristics in applications such as photonics, electronics, optoelectronics, and sensing, etc. [1, 2]. The array properties can be tuned further by varying the geometry, the doping, the periodicity, and the size of the micro- and nanostructures [2]. These controllable and elaborate arrays are commonly fabricated by X-ray, electron-beam or ion-beam lithography [3-5]. However, there are inherent limitations to pattern over large areas at nanoscale using these lithographical techniques due to the light diffraction, the long-range inter-particle interactions or the proximity effects. The conventional photolithographic techniques currently used in IC industry and micro-/nano- devices fabrication have also reached their ultimate spatial resolution because of both the light diffraction and the photoresist restrictions [5]. To overcome these limitations, alternative techniques with a shorter wavelength and a thinner resist for traditional optical lithography, including extreme ultraviolet lithography, atom lithography, microcontact printing and soft lithography, etc., have been proposed and developed to meet the requirements of a high spatial resolution of sub-100nm as well as the capability to fabricate an arbitrary structure and achieve mass production [5-9].
The atom lithography using metastable atoms beam (MAB) and self-assembled monolayer (SAM) has shown great potential to fabricate arrays of micro- and nanostructures, which is a major goal in nanoscience and technology [9-14]. This new fabrication technique, which bridges the gap between the bottom-up chemical self-assembly techniques and the modern top-down lithography, can overcome the intrinsic resolution limitations of traditional photolithographic techniques [9, 10]. In principle, the atom lithography based on neutral MAB and SAM ideally meets the required conditions for sub-100nm fabrication [9]. MAB with a de Broglie wavelength of less than 0.1nm eliminates diffractive resolution limitations, and an ultrathin organic SAM resist with a thickness of 1~3nm ensures the sharpness of the edge profile within the resist. Moreover, the neutral metastable atoms are insensitive to the electric and magnetic fields, and the long-range inter-particle interactions are weak. Therefore, this novel method permits the direct and large area manufacturing of micro- and nanostructures on a silicon wafer, avoiding some inherent complications of electron-beam, ion-beam or photolithography. With these unique advantages, the atom lithography with neutral MAB and high-resolution SAM resists makes it possible to achieve nanolithography and provides a potential way in manufacturing structures at a large-scale based on micro- and nanoscale features.

Previous reports have investigated the mechanism of the metastable atom lithography and the microfabrication with different resolutions on various substrates, such as gold, silicon, silicon oxide, and mica [9-14]. The typical micropatterning with sub-100 nm resolution on a silicon substrate covered with a SiO2 or Au layer has been achieved successfully [9, 11, 14]. Most of these studies were conducted through multistep etching processes using the cover layers as intermediate masks. For example, in the He-MAB lithography with dodecanethiol-SAM [9, 11], a Au/Ti coating layer was used to create an intermediate mask on silicon substrate by the first etching process, and then, the intermediate mask pattern is transferred to silicon substrate by the second etching process. It is very interesting to directly transfer negative and positive patterns of SAM induced by metastable helium atoms beam (He-MAB) into a silicon substrate to fabricate arrays of micro- and nanostructures, which did not use metal/oxide coating layer. Recently, the He-MAB lithography of Si with SAM, which formed directly on silicon substrate by direct covalent linkage instead of metal/oxide coating layers, has been accomplished [10]. The latent image formed in SAM by He-MAB passing through a stencil was directly transferred into the underlaying Si substrate by KOH etching, which was a single step etching process. Undoubtedly, to realize and understand this direct transfer process will open a novel way in the practical application of the atom lithography in micro- and nanofabrication of silicon, especially in arrays of micro- and nanostructures of silicon.

In this paper, we reported a micropatterning of Si(110)/Si(100) wafer surfaces by atom lithography using He-MAB, SAMs of organosilanes (ODTS, DDTS) and a single step wet etching process, which indicated a potential application in fabricating arrays of silicon micro- and nanostructures. The internal energy (~20eV) of the metastable helium atom was used to selectively damage the SAM bonded directly on the Si(110)/Si(100) surfaces through a stencil masks to generate a latent pattern in SAM, and then a wet etching process was employed to transfer and develop the latent pattern into the underlying silicon substrate. Negative/positive patterns of Si(110)/Si(100) surfaces with a depth of 10~220nm and an edge resolution of 40~500nm were obtained, and arrays of silicon microstructures were fabricated successfully on Si(110) and Si(100) substrates. The mechanism of positive-tone and negative-tone patterning of silicon substrates were investigated and discussed briefly. Our results suggested that this versatile process, combining bottom-up chemical assembly techniques with top-down atom lithography, could contribute significantly to the development of more monolayer-derived micro- and nanofabrication.

2. Experimental procedure

2.1 Preparation of Si(110)/Si(100) substrates with SAM
The p-type Si(110) /Si(100) wafers with a native oxide layer (Φ=100mm, thickness=300µm, ρ=1~3 Ω·cm) were used and cut into pieces of 10mm×20mm as Si substrates. The Si substrates were first
rinsed using toluene, ethanol, and deionized water sequentially, then immersed into a 2vol.% aqueous HF solution for 5 min to remove the native oxide layer and hydrogenate the silicon substrates, and finally dried. Protected from air by a flowing argon gas, the Si substrates were immersed into a 10mM organosilanes (ODTS and DDTS) toluene solution at room temperature for 24 hours, and then rinsed in toluene and dried at 100°C for 30 min. The mechanism of forming SAM on silicon substrate is illustrated in details in Scheme 1. Thus, through direct covalent linkage, i.e., silicon-carbon, fine organosilanes (ODTS and DDTS) SAMs were formed as a resist on silicon surface by introducing a hydrogenation process into the pretreatment of silicon wafer and a controlled argon atmosphere at elevated temperature.

2.2 Exposure to MAB

The experimental setup for the sample exposure to He-MAB was depicted in Scheme 2. The Si substrate with organosilane SAM was covered by a mask with TEM Cu grids. As shown in the inset of Scheme 2, six TEM Cu grids with a pitch size of 12.5µm (5.5µm for wires and 7.0µm for spaces) were well distributed on a nickel sheet (with a thickness of 0.1mm and a square area of 1cm×2cm) and fixed over the opening holes with a diameter of 2mm using Ag conductive paste. The assembly was immediately inserted into the sample chamber of the MAB source (as shown in Scheme 2) and exposed to He-MAB for 5~120 min, producing latent patterns in organosilanes SAM on Si substrates. The experiments were performed with the existing apparatus, as described in detail elsewhere [10, 13]. Briefly, the He* source was operated in a dc discharge mode, and a stable pressure (~5×10⁻⁷ Pa for the ultimate pressure and ~3×10⁻² Pa for the working pressure) of chambers was retained by turbo-molecule pump (T.M.P) vacuum systems. A transverse electrical field with a deflection voltage of ~800 V was applied to an atom beam for removing ions or electrons that can cause damage to the SAM resist during the whole exposure. The helium beam through a skimmer opening with a diameter of 0.7 mm was directed toward a rotatable thin tantalum (Ta) sheet sample holder at a 26.5 cm distance. This Ta holder had a triangle arrangement along the rotation axis, on which two faces were used for installing samples and the rest for monitoring the intensity of the primary beam (shown in the inset of Scheme 2). The thickness of the sheet defines the gap between masks and sample surfaces (~100µm), and avoids the mechanical contact that may cause possible artifacts. The metastable flux was estimated by measuring metastable-atominduced electron emission on both the sample and Ta plate. The typical values of the measured current on both the Ta plate and sample were 100 and 50 nA, which corresponded to an electron emission rate of ~1×10¹² and ~2×10¹² s⁻¹·cm⁻², respectively.
2.3 Etching processing and pattern transferring
After exposure, the samples with latent patterns in SAM were taken out from the vacuum chamber and rapidly dipped into an etching solution in which a magnetic bar was used for stirring, and were etched for 5–60 min at room temperature in an aqueous solution of 0.1 M KOH, rinsed by deionized water, and then dried in air.

2.4 Characterization
AFM images were taken with a multimode Nanoscope IV atomic force microscope (Veeco Metrology Group, Santa Barbara, CA), operated at ambient conditions. For all images we recorded the retrace direction of the tip using a scan angle of 0° or 90°. Substrates decorated with organosilanes SAMs were imaged in tapping mode using silicon cantilevers (NanoWorld, Neuchâtel, Switzerland) with a spring constant of 42 N m⁻¹. All images were recorded at a rate of 1.0 Hz, and with a pixel resolution of 512. SEM inspection of patterned Si substrates and the masks was carried out with a JSM-6500/SG scanning electron microscope operated at 30 keV.

3. Results and discussion
The process used to create the arrays of silicon micro- and nanostructures on a Si substrate was illustrated in Scheme 3. By introducing a hydrogenation process into the pretreatment of silicon substrates (as described in (I) and (II) in Scheme 3), we successfully formed fine organosilanes SAMs as a resist on silicon surface under a controlled argon gas atmosphere at elevated temperature. The chemical modification of non-oxidized silicon surfaces utilizing monolayers could be achieved by neutralizing the silicon surfaces with alkyl chains through direct covalent linkage, i.e., silicon-carbon [15]. This provided us an opportunity to directly use an ultrathin hydrophobic SAM with a thickness of 1–3 nm as a super resist on silicon surfaces. The samples with the stencil masks were exposed to the irradiation of He-MAB at thermal energy, and the SAM was damaged through the interaction between the outermost surface of the SAM and the metastable atoms (as described in (III) in Scheme 3). When the metastable atoms hit the outermost surface of the SAM on Si substrate, they transferred their internal energy (~20 eV) to the chemisorbed hydrocarbons of SAM molecules and caused chemical changes that might result in either the formation of a durable crossing-linking polymerization material or the loss of hydrophobicity of the SAM (as indicated in (IV) and (V) in Scheme 3). After exposure, the pattern was transferred into a latent pattern in SAM. The SAM exhibited the positive-tone and negative-tone sensitivity due to largely different dosage of MAB and the length of the alkyl chains of SAM molecule. The negative patterning indicated more He-MAB irradiation dose than the positive.
patterning, corresponding to the polymerization induced by He-MAB. Meanwhile, the positive patterning corresponded to the hydrophilic region induced by He-MAB. Furthermore, the longer alkyl chains are easier to undergo cross-linking polymerization than the shorter alkyl chains under the MAB irradiation. Consequently, these could affect the etching process in the fabrication of the micro- or nanostructure (i.e., the hydrophilic exposure region should be easier to etch using KOH solution than the hydrophobic unexposed region, whereas the cross-linking polymerization region should be more durable to KOH etching than the hydrophobic unexposed region), resulting in the positive and negative pattern transfers (as described in (VI) and (VII) in Scheme 3), respectively. Our previous investigations of the pattern transfer process have confirmed this mechanism for the contrast inversion, i.e., the competition between the loss of hydrophobicity and the cross-linking polymerization of SAM molecules determined the polarity [10]. In our experiments, the lithographic patterns were obtained in samples with a relatively large area of about 2 cm². In these areas, the mask pattern was reproduced with high fidelity, and the repetition of lithographic patterns was consistent in different runs. To obtain better contrast patterns, the experimental parameters were carefully optimized. After etching, the resulting arrays fabricated on the silicon substrate were clearly observed under the optical microscope. Furthermore, the samples were investigated under the atomic force microscope (AFM) and the scanning electron microscope (SEM).

Scheme 3. Schematic illustration of the procedure used for the fabrication of periodic arrays of Si(110)/Si(100) by He-MAB lithography (excited helium atom: He*; ground-state helium atom: He).

Figure 1. (a) SEM image of TEM Cu grid used as a mask (Note: the pitch size of patterns is 12.5 um, 5.5 um for wires, and 7.0 um for spaces) and (b) SEM image of negative patterning on Si(100) substrate with the ODT SAM (exposure time: 60min; etching time: 5min).
Figure 1 showed the SEM images of the mask and one sample. The pattern from the stencil mask (as shown in Figure 1(a)) was transferred into the silicon substrate well, and the resulting micromesas array of silicon fabricated on the substrate were very intact and clear (as shown in Figure 1(b)). The preliminary SEM observations revealed that the direct fabrication of the array on the surfaces of silicon substrates by He-MAB and SAM was viable, and our results indicated that the patterning also appeared on either kind of surface of the Si(110) or Si(100) substrate with various organosilane SAM mentioned above.

![Figure 1](image1.png)

**Figure 2.** Tapping-mode AFM images of periodic arrays of square silicon micromesa fabricated on Si(110) substrate with ODTS SAM (exposure time: 20 min, etching time: 5 min): (a) AFM image with sectional analysis of the arrays of silicon micromesas with a height of about 26 nm; (b) 3D views of the arrays of silicon micromesa over a 80 µm×80 um area and with a periodicity of 12.5 um.

The AFM images of the square micromesa arrays formed on the Si(110) substrate with ODTS SAM was shown in Figure 2, which corresponded to negative patterning with an exposure time of 20 min and an etching time of 5 min. A square silicon micromesa arrays with a micromesa size of ~7×7 µm² (e.g., the size of one mesh of TEM stencil mask) was fabricated successfully on the substrate. The average height of the step was 27 nm, indicating an etching rate at 5 nm/min. The sharpest step edge had a width of 1 pixel (41 nm) and a height of 26 nm, indicating a steep sidewall with an aspect ratio of 1:1.7. The sectional analysis shown in Figure 2(a) indicates that the rms roughness of the top area of the micromesa and the pedestal base area were 2 nm and 15 nm, respectively. The flat top area of the micromesas could be observed clearly, suggesting the strong durability of the exposed SAM resist against chemical etching. The flaw pedestal base area of the micromesa was also very conspicuous, and this rough characteristic could be attributed to the imperfect SAM formation on the silicon substrate, which induced a local fluctuation in the etching rate of transferring the latent image in SAM resist onto the underlying silicon substrate and, consequently, produced a rough surface. As described in scheme 3, the negative-tone sensitivity of the SAM might be mainly due to the cross-linking polymerization of ODTS molecules induced by the irradiation of the metastable helium beam [10]. In addition, it was noteworthy that the etching time had a notable influence on the production of high-quality patterning. In our practical runs, the following etching times were chosen: 5, 10, and 20 min with identical exposure times. The sectional analysis of AFM images of these samples indicated that the rms roughness of top area of the micromesa in the case of overetching was bigger than that in the case of underetching.
Compared with the negative patterning discussed above, a positive patterning could be obtained by adjusting the dosage of He-MAB irradiation and the length of alkyl chains of SAM molecule. Figure 3 showed the AFM image of the square microwell arrays fabricated on the Si(100) substrate with DDTS SAM, corresponding to the positive patterning (exposure time: 20 min; etching time: 10 min). A square microwell arrays with a microwell size of ~7×7 µm² was fabricated on the Si(100) substrate. The depth of the microwells was about 220 nm. The sharpest wall edge had a width of 1 pixel (41 nm) and a depth of 100 nm, indicating a steep sidewall with an aspect ratio of 2.5:1. The sectional analysis shown in Figure 3(a) indicated that the rms roughness of the bottom area of the microwell and the top area of the wall were 31 nm and 22 nm, respectively. The rough characteristic could also be attributed to the imperfect SAM formation on the silicon substrate, as mentioned above. The positive-tone sensitivity of the SAM should be mainly due to the loss of hydrophobicity of DDTS SAM (with shorter alkyl chains) induced by the irradiation of the metastable helium atoms. The topography and line profile of the microwells array indicated a much rougher top surface of the sidewall with an rms roughness of 22 nm. This large rms value suggested that the hydrophobic area might be much weaker in resisting the KOH etchant, and the DDTS SAM would not be a good resist for atom lithography.

For the wet etching of Si in KOH solution, the anisotropy of etching rate of silicon have been studied in details in the case of the photolithography. The etching rates for the silicon wafer in the KOH solution are known to depend strongly on its orientation (etching ratio: {110}>{100}>>{111}) [16]. The anisotropy can induce obvious difference of the spatial resolution of patterning of Si along different crystal orientation. In our experiments, the anisotropic etching rate seemed to improve the edge resolution, and the anisotropic etching process of silicon strongly affected the patterning results including edge step resolution. It was easier to obtain a large aspect ratio and a better resolution for Si(110) rather than for Si(100). The sharpest step edge resolution obtained presently in Si(110) samples was only 41nm with 1 pixel and the step edge resolution of about 120 nm, indicating an aspect ratio of 3:1. In principle, the estimated value of geometrical blurring is ~240 nm with the geometry described in Scheme 2, and the spatial resolution could be improved by reducing the distance between the source and the sample, which was related to the divergence of the metastable

**Figure 3.** Tapping-mode AFM images with sectional analysis of periodic arrays of square silicon microwell fabricated on Si(100) substrate with DDTS SAM (exposure time: 20min, etching time: 10min): (a) AFM images with sectional analysis of arrays of square microwell with a depth of about 220 nm; (b) 3D views of the arrays of silicon microwell over a 80 um×80 um area and with a periodicity of 12.5 um.
helium beam. The achievable width in our experiments was much smaller than the calculated one because of the hardening effect in the process of chemical etching. If the experimental parameters in both the exposure and etching processes (e.g., the exposure time, etching time, etc.) were optimized further, higher resolution of the patterning onto the silicon substrates could be obtained.

The interaction between the outermost surface of the SAM and the irradiation of metastable atoms has been investigated for about ten years [17-20]. However, the detailed change in surface chemistry of the outermost of the SAM molecules under the irradiation of helium atom beam is still difficult to predict and remains to be explored. What we demonstrated in the present study was that both the positive-tone and the negative-tone sensitivity of the SAM were related to largely different dosage of MAB and the length of the alkyl chains of SAM molecule, as illustrated in Scheme 3. When we optimized the experimental parameters we accidentally obtained a novel patterning with both negative and positive pattern on one Si(100) substrate with DDTS SAM, which was very different from previous reports. In this case, a novel array of square micromesas with microholes in it was fabricated successfully. The mechanism to induce this dual patterning has remained to be explored at present. This dual array indicated some interesting flexibility of atom lithography in micro- and nanofabrication.

4. Conclusions
We reported a monolayer-derived fabrication of Si(110)/Si(100) microstructures arrays over a large area (greater than 1cm×2cm) by atom lithography, in which SAM and MAB were used to pattern the surface of silicon substrates without coating intermediate layer. ODTS and DDTS SAMs can not only be formed on Si(110)/Si(100) surfaces but also be used as resists for exposure to metastable-atom beams. Negative/positive patterns of Si(110)/Si(100) surfaces with a depth of 10~220nm and an edge step resolution of 40~500nm were obtained. We were able to alter the hydrophobicity and the durability of the outermost surface of the SAM in the regions exposed to MAB by varying MAB dose of exposure and the length of alkyl chains of SAM molecule, subsequently realize positive-tone or negative-tone patterning of silicon, and finally fabricate arrays of silicon microstructures. Our strategy, which was essentially a top-down integrating with bottom-up approach, involved the formation of SAM on the surface of silicon substrate and pattern transferring from physical mask to silicon substrate. The improved approach presented here could be commonly used and extended to the micropatterning of a variety of other metal oxides or materials, in which SAM could be formed directly onto the substrate surfaces without an oxide or metal intermediate layer. Our results suggested that this versatile process, combining chemical assembly schemes with top-down atom lithography, could contribute significantly to the development of more monolayer-derived micro- and nanofabrication.

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