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Light Stamping Lithography: Microcontact Printing without Inks

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Abstract: We report a new patterning method, called light-stamping lithography (LSL), that uses UV-induced adhesion of poly(dimethylsiloxane) (PDMS). LSL is based on the direct transfer of the contact surface of the PDMS stamp to a substrate via a UV (254 nm)-induced surface bonding between the stamp and the substrate. This procedure can be adopted in automated printing machines that generate patterns with a wide range of feature sizes on diverse substrates. To demonstrate its usefulness, the LSL method was applied to prepare several PDMS patterns on a variety of substrates. The PDMS patterns were then used as templates for selective deposition of TiO_2 thin film using atomic layer deposition as well as resists for selective wet etching.

I. Introduction

Patterning is essential to modern science and technology. Typical applications include microelectronics,[1] microelectromechanical systems (MEMS),[2−4] biological and chemical sensors,[5−8] microfluidics,[9−12] display units, and optoelectronic devices.[13] Among existing patterning methods, soft lithography is the most efficient method to fabricate new types of structures and devices on planar, curved, or flexible substrates at low cost.[14] Soft lithography includes a number of nonphotolithographic techniques that use a patterned elastomer (primarily poly(dimethylsiloxane) (PDMS)) as a stamp, mold, or mask to transfer the pattern to substrates.[15] Among these techniques, microcontact printing is the most versatile and cost-effective method that can routinely form patterned self-assembled monolayers (SAMs).[16−18] The SAMs (∼2−3 nm thick) are thin organic films which form spontaneously on solid surfaces. They can be used either as resists in selective etching or as templates in selective deposition to form patterned thin films.[19,20] The microcontact printing is low in capital cost and can generate patterns on nonplanar surfaces with a wide variety of materials. Even if the microcontact printing shows great promises in micro/nanofabrication, critical issues remain to be solved, for example, poor edge resolution due to the lateral diffusion of the SAM molecules, low thermal stability and density of defects of the patterned SAMs, and difficulty of multiple printing.[14−18]

Recently, several soft-lithographic techniques, based on the process of transferring a thin solid film from a stamp to a substrate, have been developed.[21−28] Especially, Childs and Nuzzo have developed a new powerful method for generating a patterned PDMS film on a variety of solid substrates called decal-transfer lithography (DTL).[25−27] DTL is based on the adhesive transfer of a patterned PDMS film from the PDMS stamp to a substrate. DTL has two different versions: cohesive mechanical failure (CMF) and selective pattern release (SPaR). The CMF patterning of DTL consists of four key steps, as shown in Figure 1. First, a patterned PDMS stamp is fabricated by casting the PDMS prepolymer on a master that has a patterned

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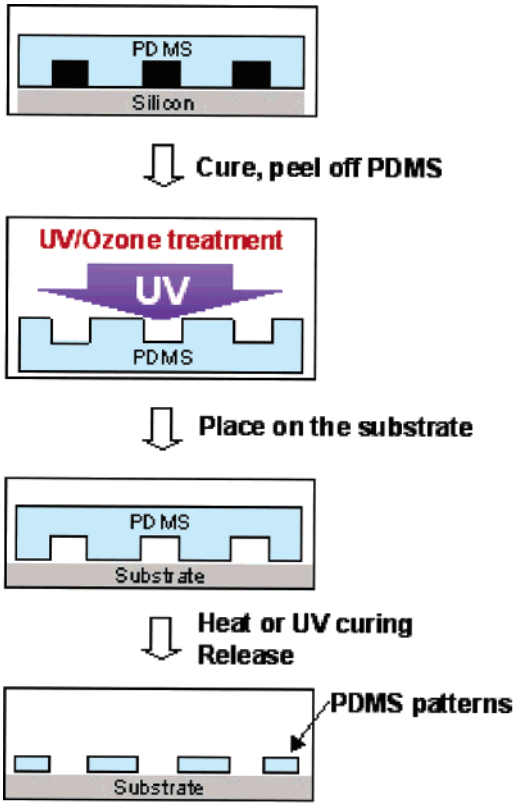

Figure 1. Schematic outline of the procedure to fabricate patterned PDMS by using DTL.

relief structure in its surface. After curing, the PDMS stamp is peeled away from the master. Second, the patterned PDMS stamp is activated by exposure to UV/ozone (UVO) and brought immediately into contact with a substrate surface. The UVO pretreatment of the PDMS, which is the most critical step, allows it to be irreversibly bound to the substrate. Third, the PDMS stamp in contact with the substrate is heated to induce an irreversible adhesion reaction to occur between them. Finally, the PDMS stamp is physically torn from the substrate via the so-called cohesive mechanical failure. The CMF-based DTL method is a very powerful technique to generate a patterned film on a variety of solid substrates without inks (organic molecules or materials).

Here we report another version of DTL method called light-stamping lithography (LSL). In the LSL method, the features of a patterned PDMS stamp are physically torn and transferred onto a substrate via a UV (254 nm)-induced surface reaction that results in bonding between the PDMS stamp and the substrate. LSL consists of three key steps, as shown in Figure 2. First, a patterned PDMS stamp is fabricated and brought into contact with a substrate surface. Second, the substrate is exposed through the PDMS stamp to a 254-nm UV lamp for 2 min. The UV light induces the formation of chemical bonds between the PDMS stamp and the substrate underneath. Finally, the PDMS stamp is physically peeled off from the substrate, remaining torn pieces thereon. The most critical step in LSL is the UV irradiation of 254 nm on the PDMS stamp in contact with the substrate, which induces a strong surface bonding between the PDMS and the substrate, whereas in DTL, the PDMS stamp is activated by exposure to UVO prior to bringing into contact with the substrate.

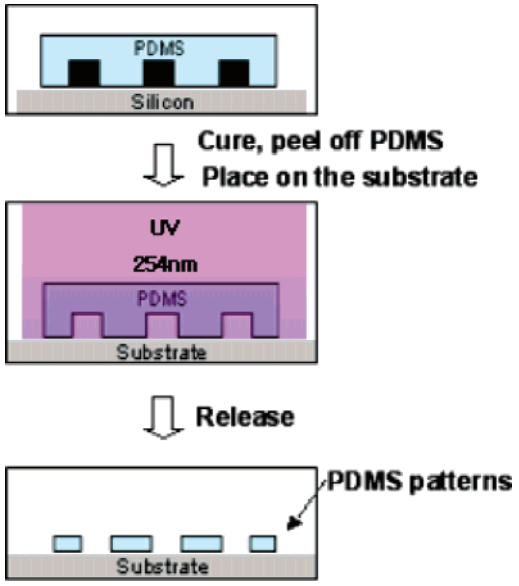

Figure 2. Schematic outline of the procedure to fabricate patterned PDMS by using LSL.

II. Experimental Section

Materials. Octadecyltrichlorosilane [$CH_3(CH_2)_{17}SiCl_3$] (Aldrich; 97%), hexane (Aldrich; anhydrous, 99%), hexadecane (Aldrich; anhydrous, 99%), chloroform (Aldrich; anhydrous, 99%), carbon tetrachloride (Aldrich; anhydrous, 99.5%), and [$Ti(OCH(CH_3)_2)_4$] (STREM Chemicals; 99.999%) were used as received. PDMS (Sylgard 184) was ordered from Dow Corning. Deionized water was purified with a Millipore Milli Q plus system, distilled over $KMnO_4$, and then passed through the Millipore Simplicity system.

Preparation of Substrates. The Si substrates used in this research were cut from n-type (100) wafers with resistivity in the range 1−5 Ω cm. The Si substrates were initially treated by a chemical cleaning process, which involves degreasing, HNO_3 boiling, NH_4OH boiling (alkali treatment), HCl boiling (acid treatment), rinsing in deionized water, and blow-drying with nitrogen, proposed by Ishizaka and Shiraki to remove contaminants.[29] A thin oxide layer was grown on the Si substrate by chemical oxidation with peroxy-sulfuric acid. The thin oxide layer of the Si substrate was etched away by placing in a saturated NH_4F solution (40% in water) for 4 min, followed by a short (1−2 s) water rinse and a 20-s rinse in 2-propanol. The NH_4F treatment has been shown to produce a hydrogen-terminated Si substrate nearly free of carbon and oxygen. Alkylsiloxane SAMs were formed by immersing the oxidized Si substrates in a 2.5 mM solution of octadecyltrichlorosilane (OTS) in hexadecane−chloroform (4:1). The samples were washed in carbon tetrachloride to remove excess reactants and dried with nitrogen. The TiO_2 thin films were deposited onto the Si substrates by the atomic layer deposition (ALD) using $Ti(OCH(CH_3)_2)_4$ and water as ALD precursors. Ar gas served both as a carrier and a purging gas. The $Ti(OCH(CH_3)_2)_4$ and water were evaporated at 80 and 20 °C, respectively. The number of ALD cycles in a run was kept 100−200. The cycle consisted of 2-s exposure to $Ti(OCH(CH_3)_2)_4$, 5 s to Ar purge, 2 s to water, and 5 s to Ar purge. The total flow rate of the Ar gas was 20 standard cubic centimeters per minute. The TiO_2 thin films were grown at 150 °C under 2 Torr. The thickness of the TiO_2 films was about 10−20 nm. Gold substrates used in this research were prepared by evaporating 200 nm of gold onto the Si substrates that were precoated with 10 nm of chromium to promote adhesion. Platinum substrates were also prepared by evaporating 200 nm of platinum onto the Si substrates.

Analysis Techniques. Atomic force microscopy images of the samples were obtained on a PSIA XE-100 operating in tapping mode.

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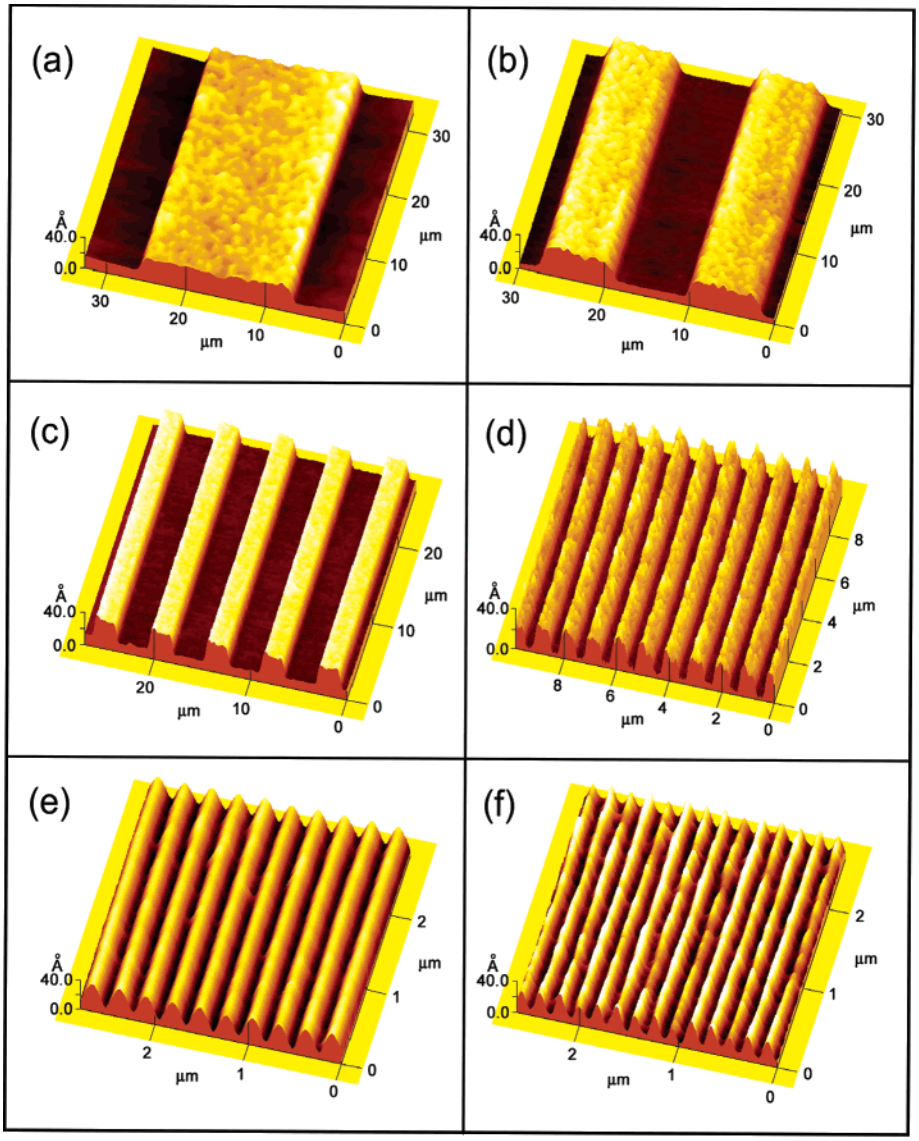

Figure 3. AFM images of the patterned PDMS fabricated by using LSL on Si substrates.

The X-ray photoelectron spectroscopy (XPS) measurements were conducted using the ESCALAB MKII XP spectrometer. Water contact angles of the samples were determined on a model A-100 Rame-Hart NRL goniometer in the ambient air by using the sessile drop method. The thickness of the TiO_2 thin films was estimated using transmission electron microscope (TEM, JEOL) and UV spectrometer (Scinco S-3100).

III. Results

A. Formation of PDMS Patterns on the Si Substrate. Several PDMS patterns were made on the Si substrate by using the LSL method. The masters we used for fabrication of stamps are silicon wafers with line-patterned resists, on scales from 20.2 to 0.090 μm. The masters with lines larger than 1 μm were fabricated using photolithography on silicon wafers, whereas high-resolution masters were fabricated using e-beam lithography. The stamps were fabricated by casting PDMS on the masters. After curing, the PDMS stamps were peeled away from the masters. When the stamps were made using these masters, the raised lines of the master corresponded to the recessed spaces of the stamps. The patterned PDMS stamps were placed in contact on the Si substrates. Then, the Si substrates were exposed through the PDMS stamps to an ORIEL 450 W Xe lamp (UV Enhanced) with total intensity of 50 mW/cm^2 at a working distance of 20 cm for 2 min. The UV irradiation induces a strong surface bonding between the PDMS stamps and the substrates because the primary wavelength of the lamp is 254 nm and PDMS is optically transparent above 230 nm. After inducing the surface reaction, the stamps were peeled away carefully. The PDMS patterns were left on the substrates exclusively in the areas of contact. When the PDMS patterns were made using these stamps, the negative and hydrophobic patterns of the original masters were produced on the substrates.

Parts a−c of Figure 3 show AFM images of the PDMS patterns fabricated by using the masters having 20.2-, 8.4-, and 3.5-μm parallel lines with 18.8-, 8.8-, and 2.2-μm spaces, respectively. These images clearly show that the PDMS patterns retain the dimensions of the masters without noticeable line spreading. Parts d−f of Figure 3 show AFM images of the PDMS patterns fabricated by using the masters having 395-, 120-, and 95-nm parallel lines with 505-, 175-, and 90-nm spaces, respectively. The lines of the PDMS patterns are 510, 177, and 95 nm wide and separated, respectively, by 390, 118,

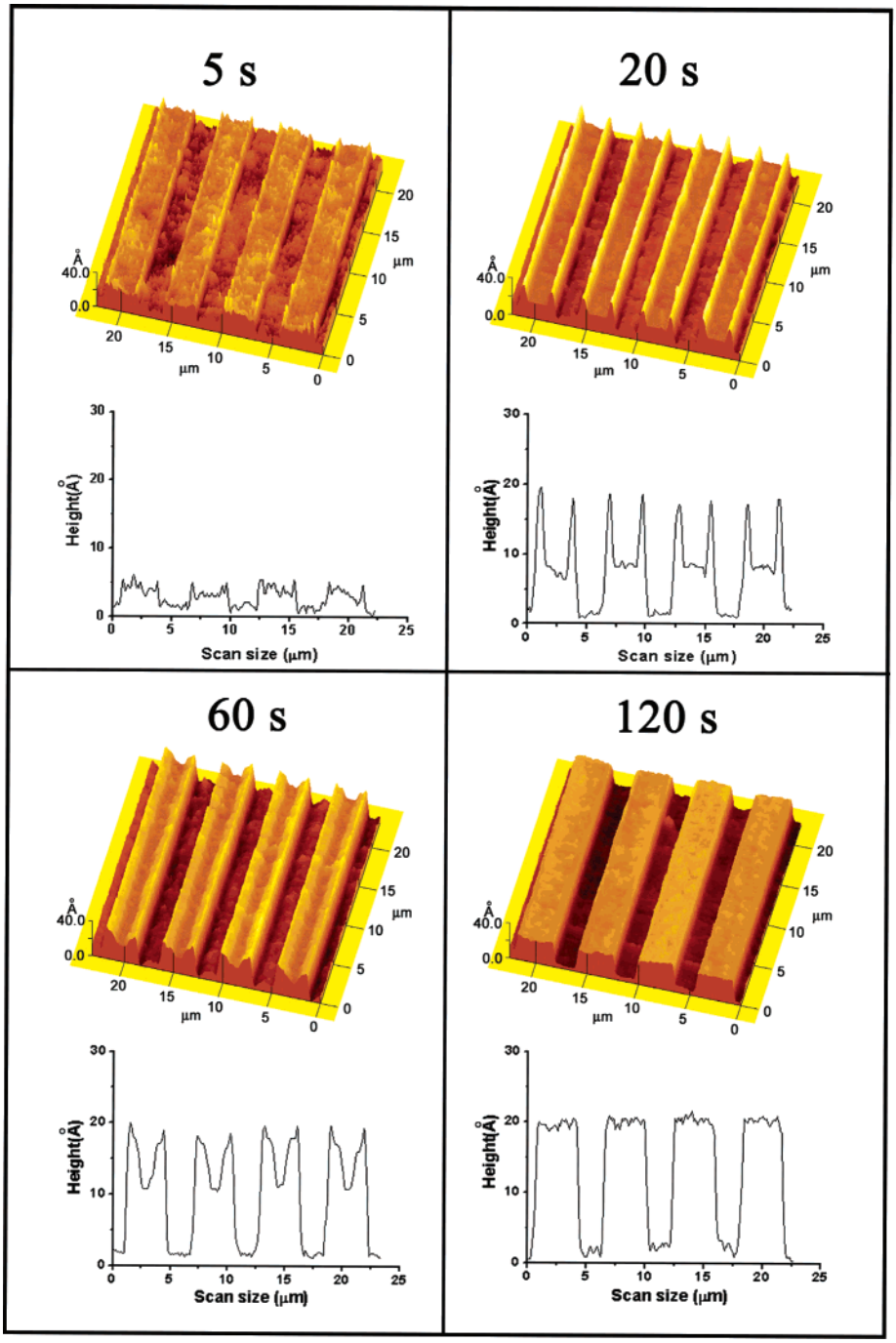

Figure 4. AFM images and cross sections of the patterned PDMS fabricated by using LSL on Si substrates as a function of UV irradiation time.

and 90 nm, indicating that the PDMS patterns almost retain the dimensions of the masters without noticeable line spreading. Additionally, the height of each PDMS pattern is about 19 Å, which is close to that of a densely packed alkylsiloxane monolayer (∼20 Å).[30−32] The regions covered by the PDMS patterns were hydrophobic, low free-energy surfaces, whereas the regions exposing the silanol groups of the Si substrates were hydrophilic, high free-energy surfaces. The water contact angle of the PDMS-patterned Si substrates was about 106°, which is close to that of the alkylsiloxane SAMs that had been patterned on Si by microcontact printing.[25−27]

B. AFM and XPS Studies for the Light-Stamping Process. To elucidate the pattern transfer processes in LSL, the PDMS patterns made on Si substrates were analyzed by AFM. At first, we fabricated several PDMS stamps using a master composed of 2.3-μm parallel lines and 3.6-μm spaces, and then the PDMS stamps in contact on the Si were exposed to UV for diverse periods of time. Figure 4 shows AFM images and cross sections of the Si substrates that have been obtained after exposure of the PDMS stamps to a 254 nm UV lamp for 5, 20, 60, and 120 s. After a 5-s irradiation, PDMS patterns with a mean height of about 5 Å are visible on the Si substrate. After a 20-s irradiation,

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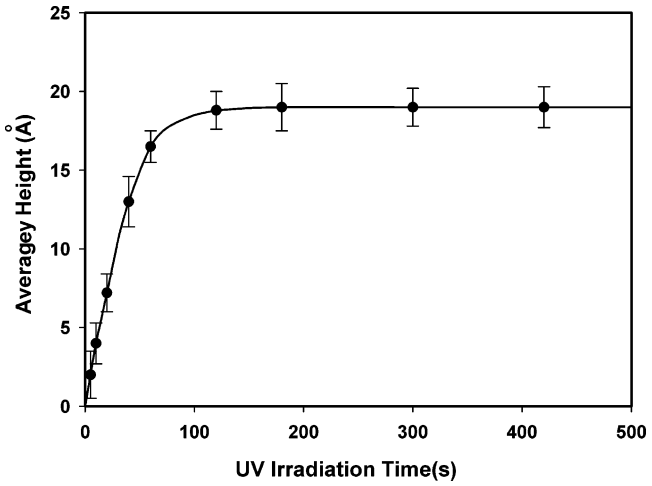

Figure 5. Average height of the patterned PDMS fabricated by using LSL on Si substrates as a function of UV irradiation time.

line edges are seen to increase in height by ~18 Å. After prolonged irradiation, the spaces between the line edges are filled, maintaining the initial height for the line edges. After a 120-s irradiation, smooth PDMS patterns with a mean height of about 19 Å are observed on the Si substrates. These images clearly indicate that the PDMS patterns retain the dimensions of the master patterns without noticeable line spreading. To obtain more quantitative information, similar AFM measurements were made for patterns generated by UV irradiation for up to 600 s. Figure 5 displays the mean height of the PDMS patterns on Si thus obtained as a function of UV irradiation time. It is seen that the mean height of the patterns increases with increasing irradiation time, approaching a limiting value (~19 Å) after 120 s. This is intriguing that the limiting height, 19 Å, is in fact comparable to that of a densely packed tetradecylsiloxane monolayer (~19 Å).

Separately, a PDMS stamp without any pattern was brought into contact with a TiO_2 substrate and exposed to a 254-nm UV lamp for 2 min and peeled away from the substrate. The three kinds of the PDMS surface, i.e., the untreated PDMS, the PDMS after LSL, and the transferred PDMS film, were analyzed by glancing-angle XPS. All XP spectra were acquired at a 10° takeoff angle in order to investigate only the surface state of the sample. With mean free paths calculated from the equations given by Seah and Dench,[33] the sampling depth for Si(2p), O(1s), and C(1s) was about 17 Å. The survey spectrum of the TiO_2 substrate after LSL did not show any Ti peak, which means that the sampling depth was lower than the thickness of the transferred PDMS film. High-resolution spectra of the PDMS stamp retain the shapes and intensities after LSL, as shown in Figure 6. Furthermore, high-resolution spectra of the transferred PDMS film on the substrate are seen to be similar to those of the stamp. All these results indicate that both the PDMS stamp and the transferred film are not subjected to change in chemical states during the light stamping processes.

C. Substrate Effect on LSL. The PDMS patterns were created by using LSL on many substrates such as oxidized Si (100), hydrogen-terminated Si (100), OTS-coated Si (100), TiO_2, Pt, and Au. Figure 7 shows AFM images of the PDMS patterns on SiO_2, TiO_2, H−Si, and Pt. The stamp was fabricated by using the master having 395-nm parallel line and 505-nm space. The

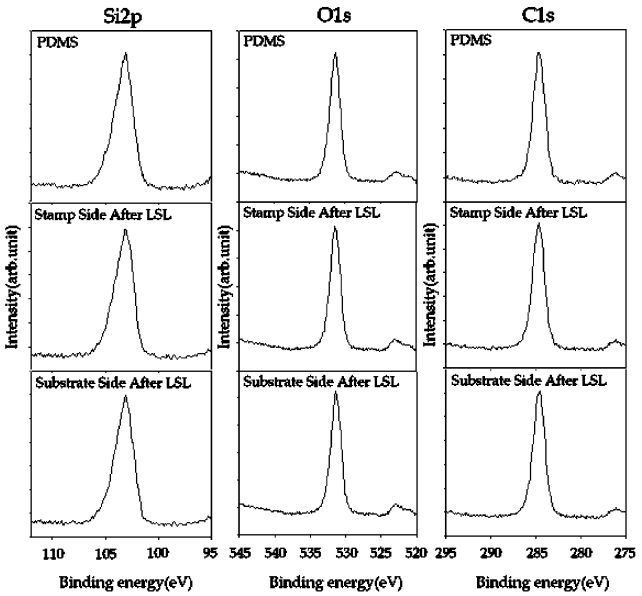

Figure 6. XP high-resolution spectra of the PDMS stamp before LSL and after LSL, and TiO_2 substrate after LSL.

lines of the PDMS patterns on all these substrates are 510 nm wide and separated by 390 nm, indicating that the PDMS patterns almost retain the dimensions of the masters without noticeable line spreading. Additionally, the height of each PDMS pattern is also about 19 Å. It, however, is difficult to form the PDMS patterns on OTS−Si and Au by using LSL. The surface free energies of these two substrates are too low so that the UV (254 nm)-induced surface reaction to form chemical bonds with the PDMS stamps hardly takes place. After a long irradiation time (more than 30 min), diffuse PDMS patterns with a low height (below 5 Å) are visible on these substrates, as shown in Figure 8. All these results indicate that the LSL method can be used to make PDMS patterns on many substrates as long as the UV (254 nm) light can induce a surface reaction to allow for the direct transfer of the PDMS patterns.

D. Selective Deposition of TiO_2 Thin Films on the Si Substrate with PDMS Patterns. The TiO_2 thin films were selectively deposited onto the Si substrates with PDMS patterns by atomic layer deposition using $[Ti(OPr^i)_4]$ and water as ALD precursors, as shown in Figure 9. The selective ALD of the TiO_2 thin films was done at 150 °C, and the number of ALD cycles in a run was kept to be 150.[34−36] The thicknesses of the TiO_2 thin films, measured by AFM and TEM, were about 17 nm. Figure 10 illustrates AFM images of patterned TiO_2 thin films selectively deposited onto the Si substrate with PDMS patterns by ALD. The patterns of the TiO_2 thin films were defined and directed by the PDMS patterns generated with LSL. It is seen that TiO_2 thin films are deposited exclusively on the regions composed of silanol groups since there is not any functional group, in the regions covered by PDMS, to react with ALD precursors. These images clearly show that the patterned TiO_2 thin films retain the dimensions of the PDMS patterns with no noticeable line spreading.

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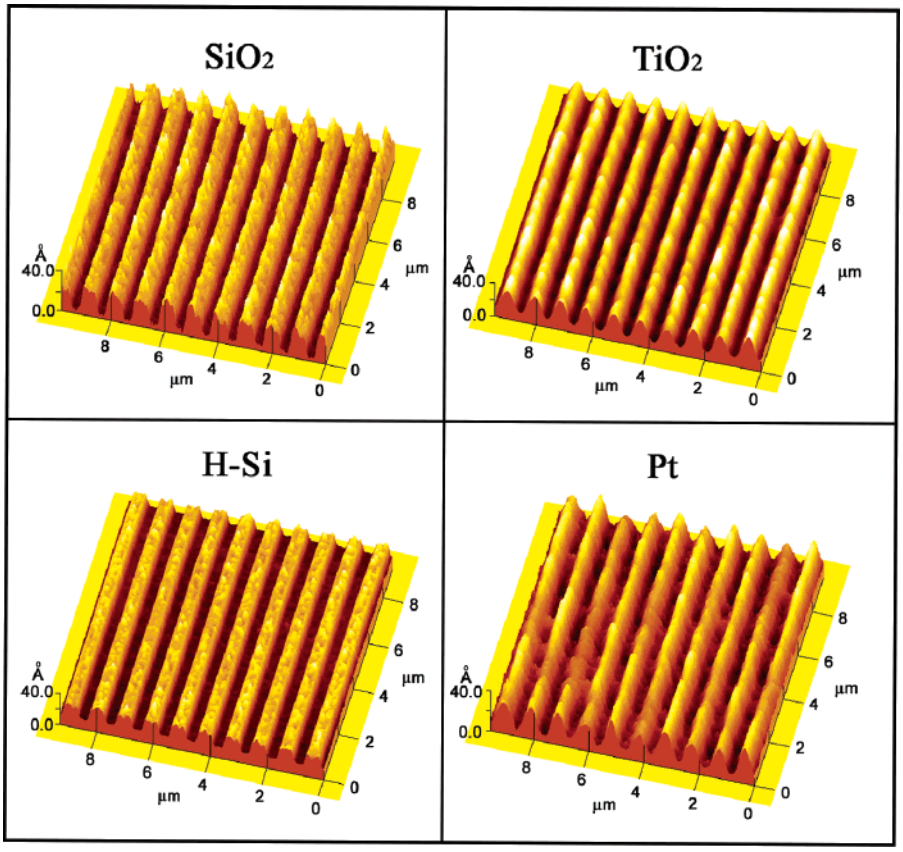

Figure 7. AFM images of the patterned PDMS fabricated by using LSL on SiO_2, TiO_2, H−Si, and Pt substrates.

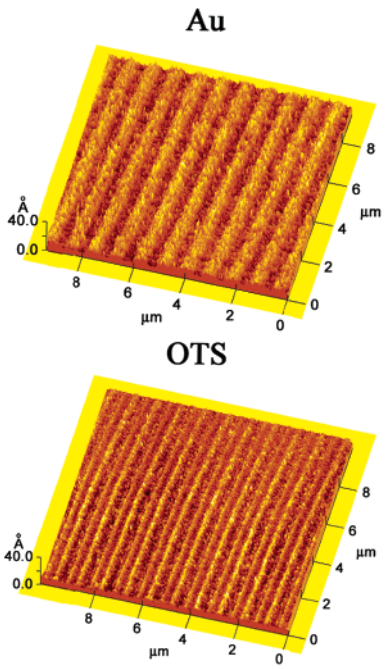

Figure 8. AFM images of the diffuse PDMS patterns on Au and OTS−Si substrates after a long irradiation time (more than 30 min).

The CMF method of DTL had some problem for pattern transfer with large feature sizes. CMF patterning with component feature sizes over 100 μm resulted in a loss of fidelity in the pattern transfer, a complex locus of cohesive failure, and irregular film distortions.[25] The LSL method, however, does not have this kind of limitation, as shown in Figure 11.

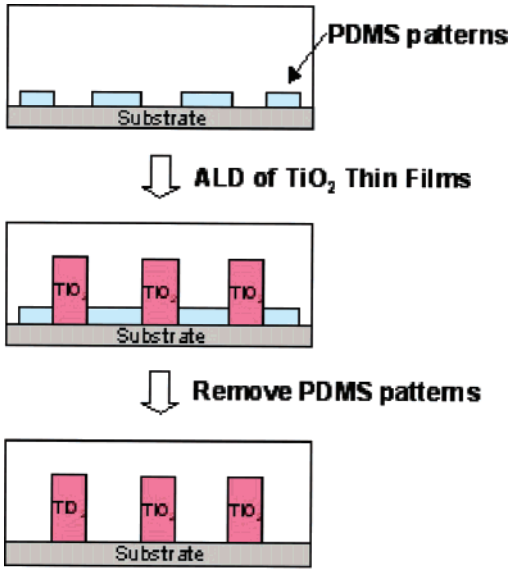

Figure 9. Schematic outline of the procedure to fabricate patterned TiO_2 thin films by using selective ALD on the PDMS-patterned Si substrates.

Microscopic images clearly illustrate that TiO_2 thin films are selectively deposited onto the Si substrate with PDMS patterns of large feature-sizes over 100 μm. These images clearly show that the patterned TiO_2 thin films retain the dimensions of the stamps with no distortion.

E. Selective Etching of TiO_2 Thin Films with PDMS Patterns. PDMS patterns are efficient resists for conventional wet etching. The TiO_2 thin films were deposited onto the Si substrates at 150 °C by atomic layer deposition, and the

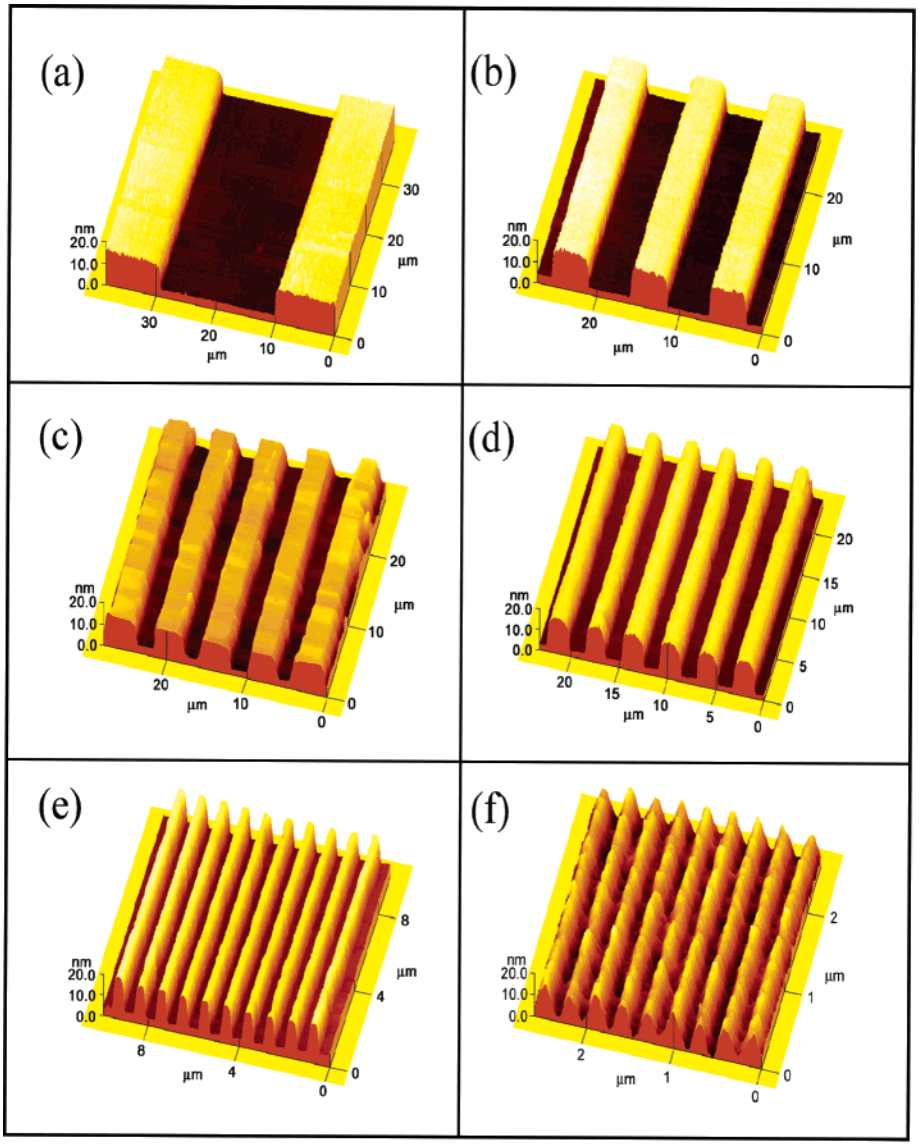

Figure 10. AFM images of the patterned TiO_2 thin films created by using selective ALD on the PDMS-patterned Si substrates.

thickness of the TiO_2 thin films, measured by TEM, was about 10 nm. The PDMS patterns were formed on the TiO_2/Si substrates by using the LSL method. The TiO_2 thin films on the Si substrates with PDMS patterns were selectively etched away by placing them in a saturated NH_4F solution (40% in water) for 1 min, followed by a short (1−2 s) water rinse. The residues of the PDMS patterns were then removed by submerging the substrate for 30 s in 1 M tetrabutylammonium fluoride in THF, as shown in Figure 12. The lines of the PDMS patterns on these substrates are 510 nm wide and separated by 390 nm, and the height is about 19 Å. Figure 13 shows the AFM image of the TiO_2 patterns that were fabricated by LSL followed by selective wet etching. The image indicates that the PDMS patterns are efficient resists for the saturated NH_4F solution, which is a wet etchant of the TiO_2 thin films.

IV. Discussion

DTL and LSL rely on adhesion-based transfer of PDMS patterns from stamps onto substrates. A major difference between DTL and LSL is the activation process of the PDMS stamp, which is the most critical step in both techniques and allows it to be irreversibly bound to the substrate. In DTL, the PDMS stamp is activated by exposure to UVO prior to its contact with the substrate, whereas in LSL, it is activated and bonded to the substrate by UV irradiation of 254 nm after its contact with the substrate.

Because of the simple activation and bonding process, the LSL method appears to have several potential advantages over other soft-lithographic methods. First, the alignments of the PDMS stamp can be easily inspected and errors can be corrected immediately. Deformation and distortions of the PDMS stamp would result in errors in the replicated patterns, as well as the misalignment of the patterns. These problems can hardly be corrected in soft lithography because the pattern transfer occurs immediately at the time of contact. In the LSL method, however, the position of the stamp can be adjusted even after its contact with the substrate because the pattern is not transferred to the substrate without UV irradiation. Second, the PDMS stamp in LSL can be used repeatedly without any treatment. The elastomeric properties allow the PDMS stamp to recover its

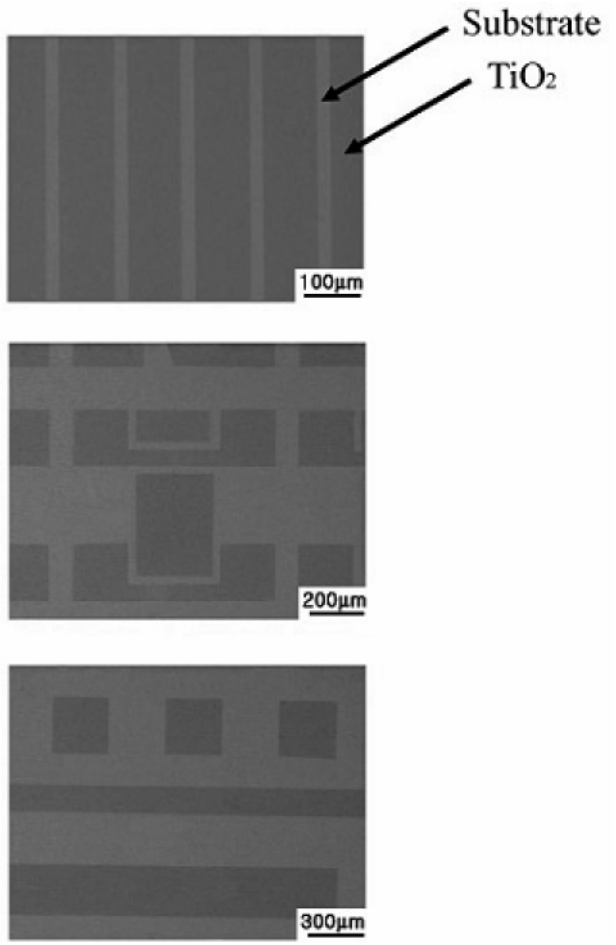

Figure 11. Optical microscopy images of the patterned TiO_2 thin films with wide feature widths.

original shape even after having undergone many cycles of patterning transfers. Since there is not needed any pretreatment, continuous printing is possible over large areas with very different feature sizes (hundreds of micrometers to tens of nanometer) until the patterns of the PDMS stamp were almost used up. Third, the PDMS patterns can be made on diverse substrates as long as PDMS can be bonded to the substrate by the irradiation of UV light. In fact, by the mediation of UV light at 254 nm, PDMS is irreversibly bound to various substrates including oxidized Si (100), hydrogen-terminated Si (100), TiO_2, and Pt; it is also noteworthy that PDMS is transparent down to ~230 nm. We believe that the LSL method can be adopted in automated printing machines that are used to generate patterns with a wide range of feature sizes on diverse substrates.

V. Conclusions

We describe a new soft lithographic method that was based on the transfer of PDMS to a substrate via UV (254 nm)-induced surface reaction to form chemical bonds between the PDMS stamp and the substrate. In the present LSL, the PDMS stamp is activated and bonded to the substrate surface by UV light after the stamp is brought into contact with the substrate,

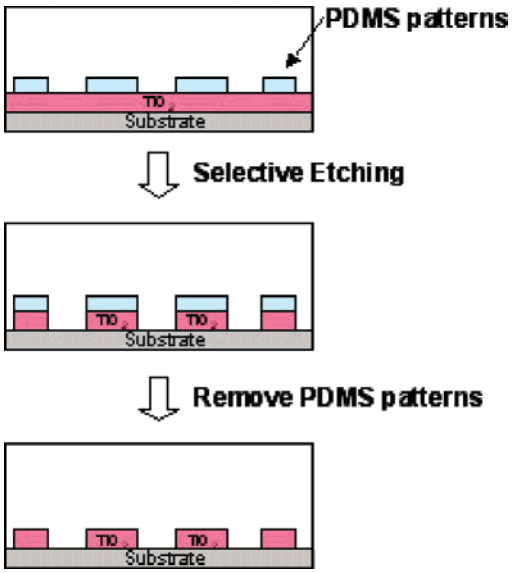

Figure 12. Schematic outline of the procedure to fabricate patterned TiO_2 thin films by using selective etching them on the Si substrates with PDMS patterns.

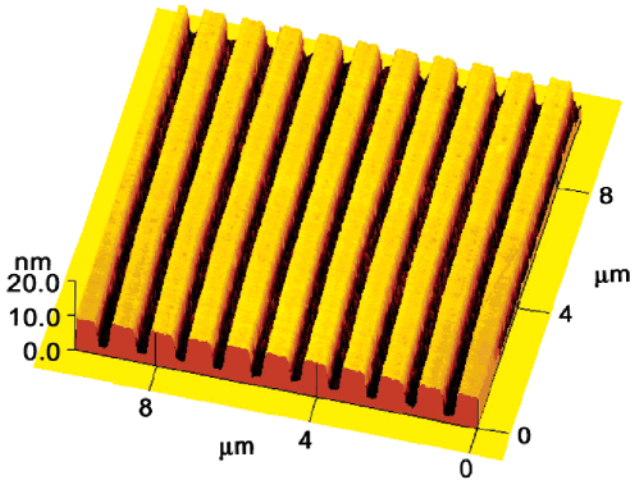

Figure 13. AFM images of the patterned TiO_2 thin films created by using selective etching them on the Si substrates with PDMS patterns.

differently from the previous cohesive mechanical failure (CMF) method. The LSL method was applied to the preparation of various PDMS patterns with a wide range of feature sizes on diverse substrates. The PDMS patterns thus prepared were used as templates to demonstrate the selective deposition of TiO_2 thin film, in addition to demonstrating their usage as resists for selective wet etching.

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