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Surface Micropatterning and Lithography with Poly(Ferrocenylmethylphenylsilane)

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The use of poly(ferrocenylmethylphenylsilane) (PFMPS) is discussed as etch resist in soft molding applications for large-area pattern transfer into silicon. The presence of Fe and Si in the main chain of this polymer is responsible for the high etch resistivity toward oxygen and CF_4 plasmas. PFMPS patterns consisting of lines, stripes, and circles were fabricated on Si by capillary force lithography and by solvent-assisted dewetting. Pattern formation during capillary force lithography was rationalized by wedge wetting models. Negative resist patterns were transferred into the Si substrates by reactive ion etching. The influence of film thickness of PFMPS resists prior to molding on the features transferred into Si was studied as a function of transfer (etching) conditions.

Introduction

The fabrication of new functional submicron and nanoscale devices with chemical or true three-dimensional patterns requires new complementary soft-lithography approaches.[1,2] Polymers come naturally as ideal “inks” or building blocks for soft lithography because of their defined architecture, wide range of chemical functionality that can be incorporated, and ease of processing on micro and nano scales.[3] Optical systems,[4−6] and microelectronic devices such as transistors[7] and light-emitting diodes[8] have been realized with polymers and soft lithography. Among the most promising polymer-based microfabricaction strategies are nanoimprint lithography,[9,10] microcontact printing,[8,11] micro-fluid-contact printing,[12] lift-up,[8,13] micromolding in capillaries,[14] replica molding,[6] solvent-assisted micromolding,[15] and its variations.[16,17] Polymer patterns can also be formed on chemically heterogeneous surfaces prepared by microcontact printing of self-assembled monolayers[18,19] and by performing photolithography in the optical near field of an elastomeric mask.[20,21] However, one of the drawbacks is that most polymers possess poor etch resistivity. Thus the choice of macromolecular “inks” that can be shaped or transferred with the stamp to, e.g., a silicon wafer and used as a “single-step” resist is rather limited.[22] Poly(ferrocenylsilanes)[23] (PFS) constitute a group of polymers that combine both macromolecular properties and etch resistivity and are ideal materials for one-step resists. Because of the presence of iron and silicon in the main chain, the polymer was found to be relatively stable toward oxygen and carbon tetrafluoride reactive ion etching. This property of PFS was used in block copolymer thin film lithography where structures with smallest dimensions in the order of 20−30 nm could be etched into underlying substrates.[24−27] The potential of poly(ferrocenyl-

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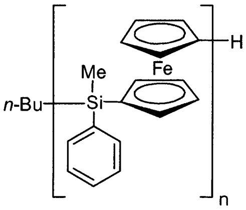
Scheme 1. Poly(ferrocenylmethylphenylsilane) (PFMPS)



dimethylsilane) as an etch barrier in O_2 and CF_4/O_2 plasmas and its use in micromolding in capillaries (MIMIC) has also been shown.[28]

This paper describes the fabrication of microstructures of poly(ferrocenylmethylphenylsilane)[29] (PFMPS) and their use as etch resist. Two soft lithography approaches for pattern formation are presented. One relies on the concept of capillary force lithography,[30,31] and the other employs solvent-assisted polymer dewetting.

Experimental Section

N,*N*,*N*′,*N*′-Tetramethylethylenediamine (TMEDA), ferrocene, dichloromethylphenylsilane, and *n*-butyllithium were purchased from Aldrich. Methylphenylsilyl[1]ferrocenophane was prepared as described earlier.[29,32] Polymerizations were carried out in THF in a glovebox purged with prepurified nitrogen. *n*-Butyllithium was used as initiator, and the reaction was terminated after 2 h by adding a few drops of methanol. The polymer (Scheme 1) was precipitated in methanol and dried. Molar mass characteristics of the polymer were determined by gel permeation chromatography (GPC) measurements in THF, using polystyrene calibration. A Perkin-Elmer Pyris 1 differential scanning calorimeter was used for the determination of glass transition temperatures (T_g) at a scan rate of 10 K/min. PDMS molds were prepared with Sylgard 184 silicone elastomer (Dow Corning). Prepolymer was mixed with curing agent in a 10:1 ratio. The mixture was then poured onto a pre-patterned silicon master and cured at 100 °C. Thin polymer films were prepared by spin-coating a toluene solution of PFMPS on silicon wafers. Prior to spin-coating, all silicon substrates were cleaned with oxygen plasma. Film thicknesses were determined with a Plasmos SD 2002 ellipsometer, using a wavelength of 632.8 nm.

The patterns in capillary force lithography experiments were fabricated by placing the PDMS mold in contact with the thin polymer film and keeping it overnight at 100 °C under vacuum. For solvent-assisted dewetting, the surface of the PDMS stamps was oxidized for 1.5 h in an ozone cleaner (PRS-100). Following the ozone treatment, a 10 mg/mL solution of PFMPS in toluene was spin-coated onto the stamp for 30 s, using a rotation speed of 2000 rpm. The stamp was then placed in contact with the cleaned surface of the Si wafer and left in contact for several hours. For contact angles of PFMPS on silicon substrates and on PDMS, pieces of polymer were placed on a standard silicon wafer and on a wafer coated with Sylgard 184. They were kept overnight at 100 °C in vacuo. Actual contact angles were measured with an OCA 15 Plus contact angle microscope (Dataphysics Instruments) equipped with a hot stage. Static contact angles were measured on both sides of the drops and the results were averaged.

Reactive ion etching (RIE) was carried out in an Elektrotech PF 340 apparatus. During oxygen RIE the pressure inside the etching chamber was 10 mTorr, the substrate temperature was set at 10 °C, and an oxygen flow rate of 20 cm^3/min was maintained. The power was set at 75 W. The films were etched under the conditions mentioned above for 10−20 s. For resist etching, O_2−RIE was used at 5 mTorr and 150 W forward power (750 V bias voltage) for up to 3 min. CF_4/O_2−RIE was performed at a pressure of 3−5 mTorr, with a substrate temperature of 10 °C. The power used was set at 20 W, which resulted in a bias voltage of approximately 190−220 V.

Atomic force microscopy (AFM) experiments were performed on a Digital Instruments NanoScope IIIa. Standard Si_3N_4 tips were used (Pointprobe, Nanosensors) with an average tip radius of 10 nm. The images were acquired in ambient air using the tapping mode. Films were studied with an amplitude of oscillation at free vibration $A_0 = 2.0$ V and with operating setpoint ratios $A/A_0 = 0.6-0.9$. Scanning electron microscopy images were captured by a LEO Gemini 1550 FEG-SEM.

Results and Discussion

Synthesis and characterization of PFMPS, an amorphous, unsymmetrically substituted poly(ferrocenylsilane), have been reported earlier.[29,32] We measured the dependence of its glass transition temperature on the number average molar mass M_n and fitted the results with O'Driscoll's equation.[33] The extrapolated value of T_g of the polymer with infinite molar mass was found to be 92 °C. A low molar mass polymer ($M_n = 3700$ g/mol, $N = 12$, $M_w/M_n = 1.07$) was used for its liquidlike behavior above T_g. Its T_g of 74 °C allows for processing at considerably lower temperatures than those used in similar experiments so far.[34] The low molecular weight polymers employed in this study are suitable "inks" as they possess a low viscosity above T_g, but preserve their shape when quenched to below T_g. We found that the right balance between molar mass, T_g, and processing conditions is essential for successful patterning. On one hand polymers with too low a molar mass will more easily dewet; on the other hand polymers with too high a molar mass will not yield patterns within the experimental time scale.

Capillary Force Lithography of Thin Polymer Films. The PDMS stamp geometry used consisted of 3-μm wide recessed lines spaced by 2-μm wide protruding lines (denoted as $2 \times 3\,\mu$m). Stamps with dimensions $3 \times 5\ \mu$m and $2 \times 5\ \mu$m were also used. The polymer, initially confined in a thin film, is squeezed out from areas of contact between stamp and substrate. It diffuses into the grooves where structures are formed along the vertical walls of the stamp due to capillary rise (Figure 1). Polymer structures, which are approximately 110 nm high and 500 nm wide, were fabricated. Section analysis of the AFM height images reveals a meniscus of the capillary rise. The contact angle between PFMPS and PDMS calculated from these AFM profiles (~70°) is in good agreement with contact angle microscope measurements ($\theta_{PDMS/PFMPS} = 67°$). The receding contact angle between the silicon substrate and PFMPS estimated from AFM section analysis is 5°, wheras the static contact angle $\theta_{Si/PFMPS}$ is 45°. This inconsistency is likely due to the large contact angle hysteresis, which

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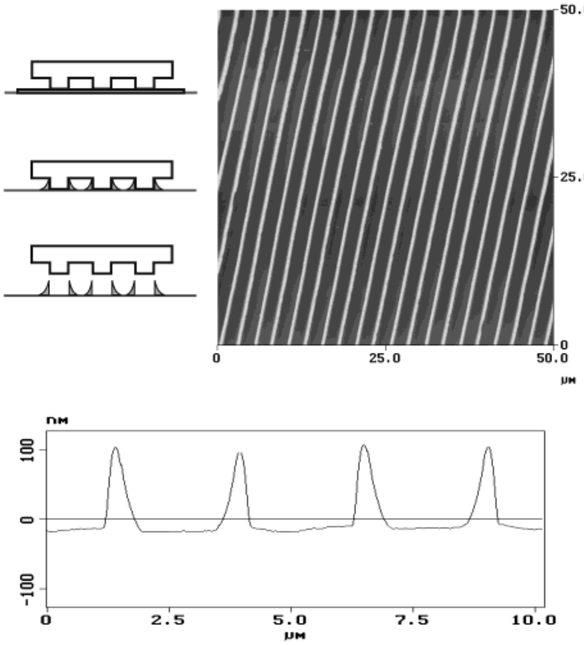

Figure 1. Capillary force lithography of thin PFMPS films with a 2 × 3 μm PDMS stamp. Schematic diagram (left) and AFM height image with section analysis. The height of the resulting features is 110 nm and the initial thin film thickness was 27 nm.

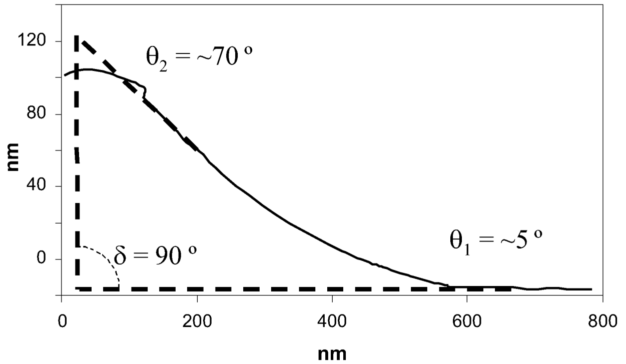

Figure 2. Meniscus profile of polymer confined in a wedge from AFM section analysis. The vertical wall is the PDMS stamp and the horizontal wall is the silicon substrate.

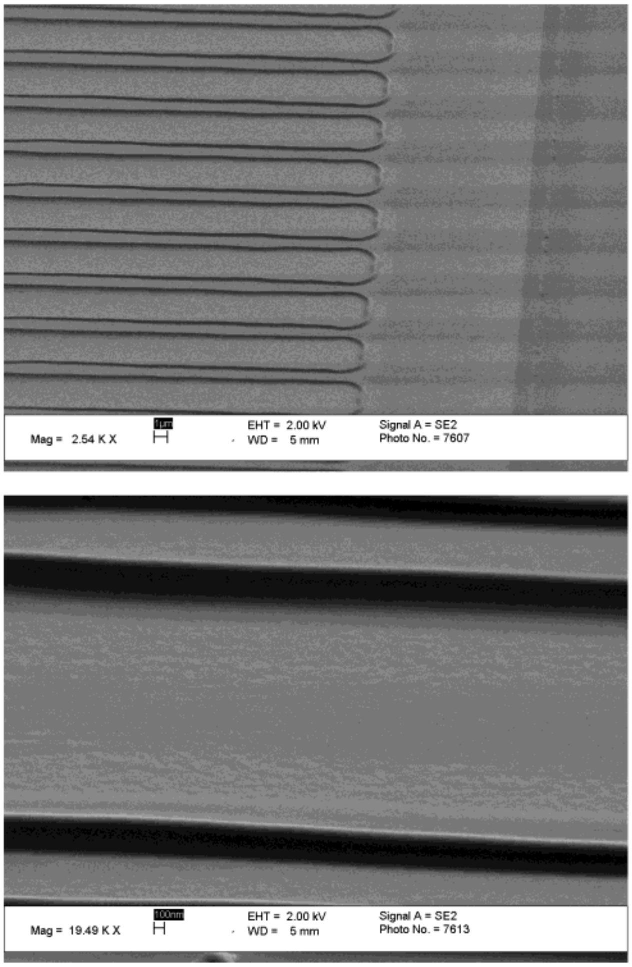

Figure 3. Scanning electron microscopy images of polymer structures obtained with a 2 × 5 μm stamp. The lower image shows an enlarged area, which was not directly in contact with the stamp. Two menisci and bare Si wafer between can be seen.

can be further reinforced by the presence of residual polymer on the substrate.

The shape of a liquid meniscus in a corner can be predicted by considering the problem of wedge wetting.[35−37] For nonvolatile liquids such as polymers, the equilibrium wedge profile is determined by the drive to minimize the surface free energy of the liquid under the constraint of a prescribed volume.[38] The equilibrium interface shape at a macroscopic wedge or step can be addressed through the Gibbs−Wulff[39] or Winterbottom[40] constructions, which predict different liquid shapes depending on the liquid−substrate contact angles and on the opening angle of the wedge. From theoretical considerations it follows that if the wall attractions are large enough, but still in a partial wetting regime, one obtains a concave equilibrium shape, which is described by the "summertop" construction.[41] Particularly for the case when the vertical (or inclined) wall is the less attractive one, a critical angle δ_S exists, above which the droplet in a corner between two walls becomes concave.[42] This angle is given such that for $\delta - \theta_2(\delta) > \theta_1$ a concave equilibrium profile described by the "summertop" construction is obtained. This is also valid for the case described here where the angles δ, θ_1, and θ_2 are 90, 5, and 70 degrees, respectively, and indeed a concave polymer (liquid) profile can be seen in the AFM section analysis (Figure 2).

Very thin polymer films do not provide enough material to fill the grooves of the stamp completely. As a result, two polymeric lines are formed per one groove. This is clearly seen in AFM profiles and was also confirmed by SEM (Figure 3). A more quantitative explanation of meniscus break down was given by Lee and co-workers.[43] Increasing the thickness of the initial polymer film results in thicker double lines which

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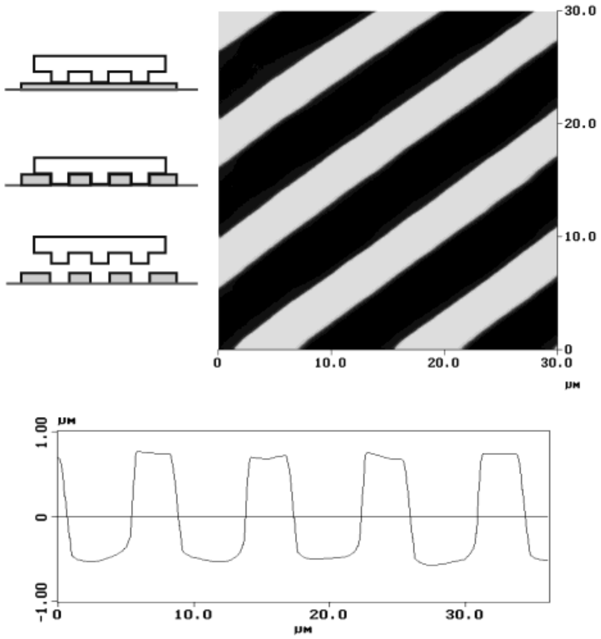

Figure 4. Capillary force lithography of thick PFMPS films. Schematic diagram (left) and AFM height image with section analysis. The features are 1.2 mm high and the initial film thickness was ~0.5 μm.

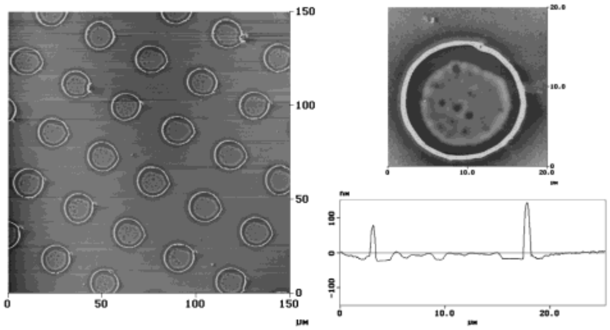

Figure 5. Circles fabricated by capillary force lithography of PFMPS. AFM height images with section analysis (bottom right) of the single circle (top right). Initial film thickness was 30 nm.

eventually merge when the film thickness exceeds ~140 nm. This allows one to tune the lateral dimensions of the polymer lines (shown later in Figure 8). Finally, when the initial film thickness is on the order of 500 nm, there is enough material for the polymer to fill the grooves of the stamp completely. In this case negative replicas of the stamps are obtained (Figure 4). In a similar way circular patterns can be prepared. Figure 5 shows structures molded with a stamp with holes of 15-μm diam. A circular rim with a height of 100−160 nm is formed. There is a depletion area outside of the circle but it is considerably smaller than the one inside. Thus, it can be concluded that most of the polymer used for capillary rise comes from the middle of the circle and was not in contact with the stamp. Unlike the polymer inside of the circle, the remainder of the thin film, which was sandwiched between the stamp and a substrate, shows no signs of dewetting. The circles appear slightly deformed, probably due to mechanical stress during peeling off the stamp.

Solvent-Assisted Polymer Dewetting in Physical Confinement. Stamps prepared from PDMS have a very low surface tension (around 20 mN/m) compared to that of other hydrocarbon-based materials.[44] One of the main consequences of such a low surface tension is the very poor wettability of the stamp surface. This is why attempts to use PFS as an ink in classical microcontact printing (μCP) were not successful. Wettability of the PDMS stamps used for μCP can be improved by treating the stamp with an oxygen plasma prior to inking. Exposure to an O_2-based plasma oxidizes the surface of the stamp and increases the free surface energy of PDMS.[45] However, the thin silica-like layer that is being formed during plasma treatment[46] thermally expands and poses a mechanical stress on the stamp upon cooling. The relaxation of this stress induces buckling of the brittle surface layer, and a periodic wavy structure with an orientation perpendicular to the pattern of the stamp is introduced.[47] This fine structure can also be replicated by the PFS used as an ink and a pattern on two independent length scales is formed.[48] In this paper we use a UV ozone generator to enhance wettability of the PDMS stamps. This provides much milder oxidation conditions and no buckling of the stamp surface is observed. Furthermore, the stamps exposed to ozone for more than an hour have a high energy, hydrophilic surface. An increase in surface roughness is also being observed. This results in a remarkably low contact angle between O_3-treated PDMS and water.[49] Patterns revealed after using these stamps have a spacing that corresponds to the periodicity of the stamp structures. This suggests that during printing the polymer solution dewets between the stamp and Si surface, forming continuous lines in the middle of the protruding stamp contact areas (Figure 6). Similar results were observed by Braun and co-workers using a hexagonally structured stamp.[12] As shown in Figure 6, these lines with a width on the order of 1 μm can be etched into the underlying silicon substrate.

Pattern Transfer into Silicon. Upon oxygen plasma treatment, a Si/Fe oxide layer is formed at the surface of poly(ferrocenyldimethylsilane).[28] The presence of such an oxide results in a high etch resistance of the PFMPS layer in O_2 and CF_4/O_2 plasmas. The high etch resistance enables a direct pattern transfer into silicon. Figure 7 displays profiles of microstructures obtained with PFMPS before and after etching, showing that approximately 300 nm of silicon was removed in a 10-min treatment. The remaining resist (polymer with oxide layer) still present at the top of the silicon structures can be stripped easily in HNO_3. The etch rate contrast between silicon substrate and PFMPS resist, estimated from the AFM height images, is 6:1. The final size of the silicon features depends on the initial polymer film thickness. This allows one to tune the lateral

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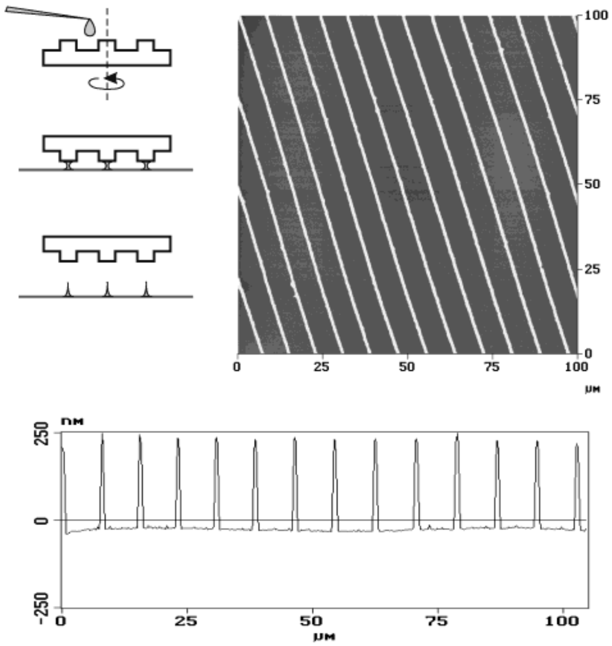

Figure 6. Solvent-assisted dewetting of PFMPS confined between PDMS stamp and silicon substrate. Schematic diagram (left) and AFM height image (right) with section analysis after 10 min of CF_4/O_2−RIE treatment.

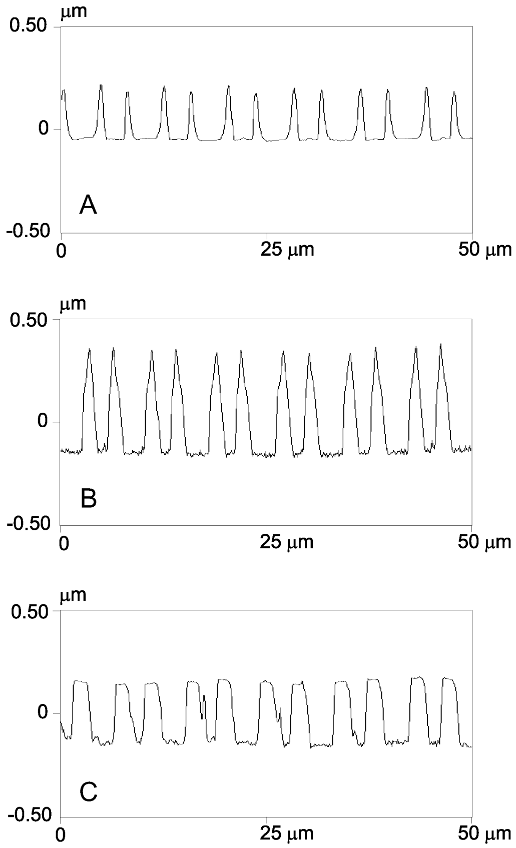

Figure 7. Pattern development in capillary force lithography (3 × 5 μm stamp) with poly(ferrocenylmethylphenylsilane). AFM section analysis of (A) PFMPS mask, (B) structures after etching, and (C) Si structures after removal of the resist in HNO_3.

dimensions of the lines in silicon. For the 3 × 5 μm stamp, lines with widths of 1.1, 2.1, 2.4, and 5.4 μm were produced. They correspond to initial film thicknesses of 30, 80, 100, and 150 nm, respectively. The gap between the lines can be adjusted from 2.5 μm for a 30 nm film to 840 nm for a 100 nm film (Figure 8). The circular patterns (Figure 5) can also be etched even though there is a thin layer (~17 nm) of polymer that covers the wafer outside and inside the circles. This thin resist layer can be removed by physical sputtering in O_2−RIE at high power and low-pressure settings, followed by CF_4/O_2−RIE. Figure 9 shows the different patterns obtained under various etching conditions.

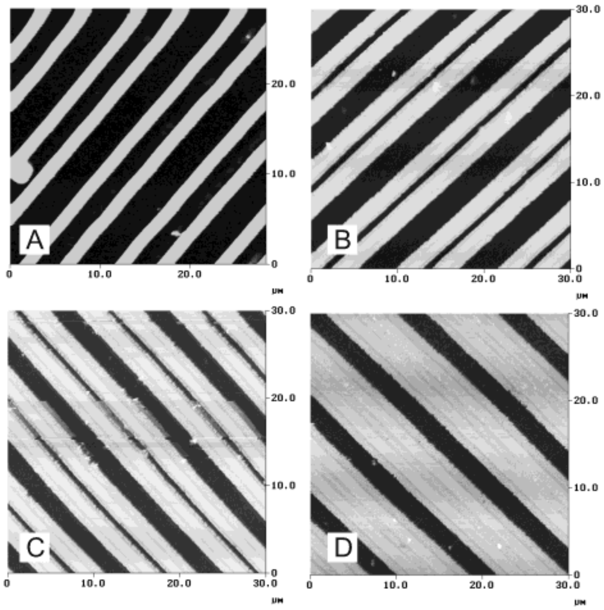

Figure 8. Patterns in silicon after etching and resist stripping. The initial polymer film thickness was (A) 30 nm, (B) 80 nm, (C) 100 nm, and (D) 150 nm.

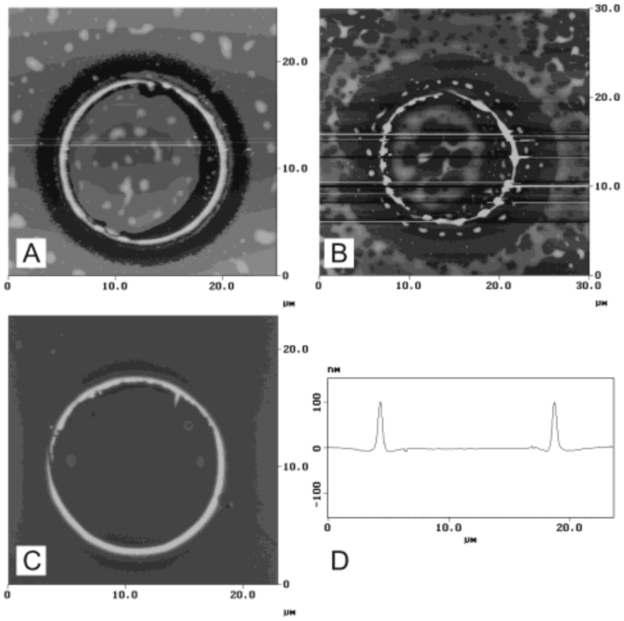

Figure 9. Etching of circles. O_2−RIE, DC bias of 750 V for (A) 20 s, (B) 60 s, (C) 120 s. AFM height images. (D) Section analysis of the circle in image (C).

Conclusions

The potential of poly(ferrocenylmethylphenylsilane) as an ink and resist in soft lithography has been demonstrated. Different soft molding approaches allow one to structure this polymer on micron and submicron scales. The patterns obtained depend not only on the geometry of the stamp but also on the molding conditions. It has been shown that a stamp with one geometry can be used to give three different line patterns. In all of the cases substrate areas with sizes of several cm^2 can be structured. Circular patterns which are not accessible at all by methods such as MIMIC, and are technologically difficult to obtain otherwise,[19] were produced. All the polymer structures were transferred into silicon substrates by plasma etching. This lithographic approach can also be scaled down to the submicrometer scale provided that stamps with desired geometries are available.

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