

# Microcontact printing of self-assembled monolayers: applications in microfabrication

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**Abstract.** This paper describes applications in microfabrication using patterned self-assembled monolayers (SAMs) formed by microcontact printing. Microcontact printing ( $\mu$ CP) is a flexible new technique that forms patterned SAMs with regions terminated by different chemical functionalities (and thus different physical and chemical properties), in patterns with  $\mu$ m dimensions. Patterns of SAM are formed using an alkanethiol as an 'ink', and printing the alkanethiol on a metal support with elastomeric 'stamp'. We fabricate the stamp by moulding a silicone elastomer using a master prepared by optical or x-ray microlithography or by other techniques. SAMs of long-chain alkanethiolates on gold and other metals can act as nanometer resists by protecting the supporting metal from corrosion by appropriately formulated etchants: the fabrication of microstructures of gold and silicon demonstrates the utility of patterned SAMs (formed by  $\mu$ CP) as nm resists. Patterned SAMs formed by  $\mu$ CP can also control the wettability of a surface on the  $\mu$ m scale. The organization of liquids in patterned arrays with  $\mu$ m dimensions, and the patterned deposition of microcrystals and microcrystal arrays illustrate the use of controlled wettability for microfabrication.

## 1. Introduction

Self-assembled monolayers (SAMs) of organic compounds on inorganic or metal surfaces are becoming increasingly important in many areas of materials science [1–3]. Although there are many different systems of SAMs based on different organic components and supports, the best developed systems are those of alkanethiolates,  $X(\text{CH}_2)_n Y(\text{CH}_2)_m \text{S}-$ , on gold films [4–6]. Alkanethiols chemisorb spontaneously on a gold surface from solution and form adsorbed alkanethiolates with loss of hydrogen. A wide variety of organic functional groups ( $X$ ,  $Y$ ) can be incorporated into the surface or interior of the monolayer. SAMs can therefore be tailored to provide a wide variety of material properties: wettability and protection against corrosion by chemical etchants are especially relevant to many applications.

We described previously [7–10] a technique that forms patterned SAMs with geometrically well defined regions of different chemical functionality and thus different physical and chemical properties. This technique,  $\mu$ CP, uses an elastomeric 'stamp' and alkanethiol 'ink' to form patterned SAMs of alkanethiolates on gold films with dimensions ranging from 200 nm to several cm. Patterned SAMs formed by  $\mu$ CP have many applications, including microfabrication [7, 8, 11–14], studies of wetting

and nucleation phenomena [12, 15–19], protein [20, 21] and cellular adhesion [22]; and in analytical studies involving scanning electron microscopy [23], and scanning probe microscopies [24]. In this paper, we focus on applications related to the fabrication of structures with micrometer and sub-micrometer dimensions.

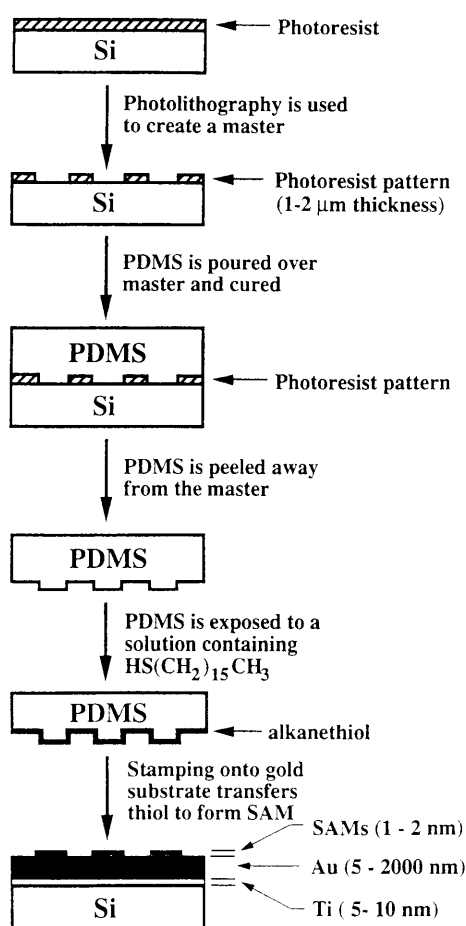
## 2. Results and discussion

### 2.1. Microcontact printing ( $\mu$ CP) transfers by contact alkanethiol 'ink' from an elastomeric 'stamp' to a gold surface: if the stamp is patterned, a patterned SAM forms [7, 8]

The stamp is fabricated by casting polydimethylsiloxane (PDMS) on a master having the desired pattern. Masters are prepared using standard photolithographic techniques, or constructed from existing materials having microscale surface features.

In a typical experimental procedure (figure 1), we placed a master in a glass or plastic petri dish, and poured a 10:1 ratio (w:w or v:v) mixture of SYLGARD silicone elastomer 184 and SYLGARD silicone elastomer 184 curing agent (Dow Corning Corporation) over the master. The elastomer degassed for approximately 30 min at room temperature, cured for 1–2 hr at 60 °C, and was peeled gently from the master. The resulting stamp replicated in reverse the relief of the features on the master: raised

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**Figure 1.** Schematic of the procedure for  $\mu$ CP. See text for details.

regions of the stamp corresponded to recessed regions of the master.

We 'inked' the elastomeric stamp by exposing the stamp to a 0.1–1.0 mM solution of alkanethiol in anhydrous ethanol, usually by rubbing the stamp gently with a Q-tip saturated with inking solution. The stamp dried until no liquid was visible by eye on the surface of the stamp (typically about 60 s), either under ambient conditions, or by exposure to a gentle stream of nitrogen gas. Following inking, the stamp was applied by hand to a gold surface. We used 50–2000 Å-thick gold films, prepared by electron-beam evaporation on Si[100] wafers, with 10–100 Å of Ti as an adhesion promoter (between the gold and the Si). Very light hand pressure aided in complete contact between the stamp and the surface. The stamp was then peeled gently from the surface.

## 2.2. Microcontact printing formed patterned SAMs with features having $\mu$ m dimensions [7, 8]

Figures 2(a), (b) show SEM images of complex patterns produced by standard lithographic techniques (photolithography, etching, metallization and lift-off) that were used as masters for  $\mu$ CP. We selected these masters to demonstrate the complexity of features that could be produced by  $\mu$ CP. The masters had features with a range of sizes ( $< 1\mu\text{m}$  to hundreds of  $\mu\text{m}$ ), arranged in patterns similar to those used in microelectronics fabrication. The relief

in these features was between 0.8 and 1.5  $\mu\text{m}$ , and gave rise to the contrast between different regions of the SEM images in figures 2(a), (b): raised regions of the master appeared lighter (in the SEM image) than recessed regions of the master.

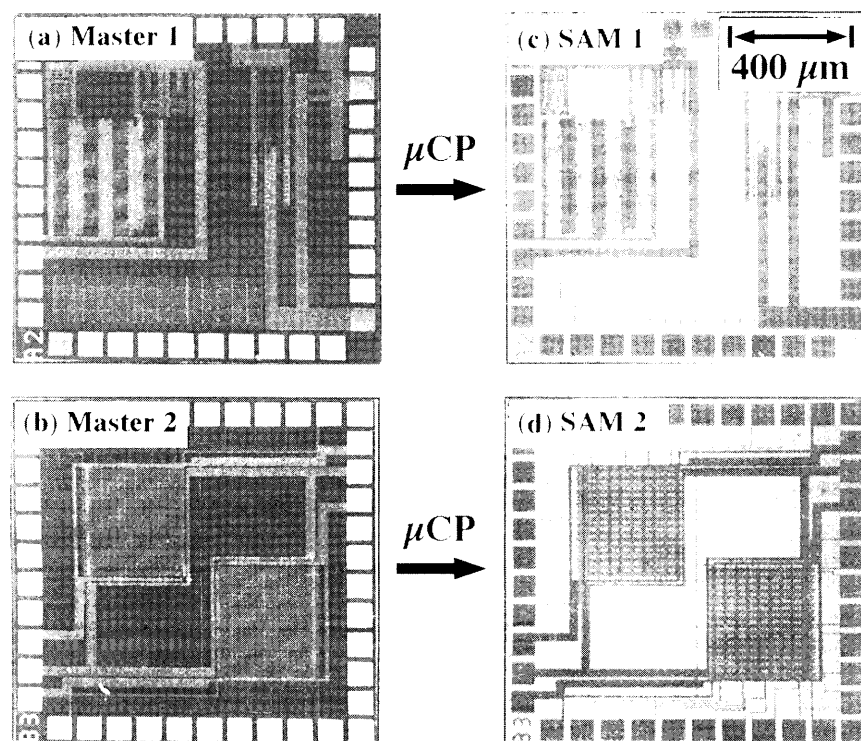
Stamps cast from these masters had recessed regions that corresponded to raised regions of the master. Microcontact printing of  $\text{CH}_3(\text{CH}_2)_{15}\text{SH}$  with these stamps on a gold film produced a patterned SAM terminated by  $\text{CH}_3$  (lighter, figures 2(c), (d)); washing the surface with  $\text{HO}(\text{CH}_2)_{11}\text{SH}$  formed patterned SAMs terminated by OH in regions of the gold surface not derivatized by the  $\mu$ CP step (darker, figures 2(c), (d)). Since the stamped features of the patterned SAM appear bright in the SEM image, the patterned SAMs are direct negatives of the corresponding masters. The complexity and scale of the features shown in figure 2 is typical of patterned SAMs that  $\mu$ CP can produce routinely.

Close inspection of figures 2(c), (d) (and other patterned surfaces in this paper) revealed the small defects in the patterned surfaces. At present, stamping is conducted by hand under ambient laboratory conditions: presumably, more sophisticated methods of stamping will improve the quality of features produced by  $\mu$ CP.

At present, the primary advantage of microcontact printing is the simplicity and ease with which micron-scale patterned surfaces can be produced. Microcontact printing is experimentally simple, and can be conducted in a conventional chemical laboratory—no routine access to clean rooms or photolithographic equipment is required (although some microfabrication technique is required to make the master). Several stamps can be fabricated from a single master and individual stamps can be used hundreds of times (while stored under ambient lab conditions) without degradation in performance. Patterned SAMs of alkanethiolates formed by  $\mu$ CP are robust—samples exposed to the laboratory ambient for several months showed no detectable degradation, and could be washed repeatedly in organic solvents without damage.

## 2.3. Microcontact printing of alkanethiols on gold and wet-chemical etching produced microstructures of gold [7]

We formed patterned SAMs by  $\mu$ CP with  $\text{CH}_3(\text{CH}_2)_{15}\text{SH}$  on a 2000 Å-thick gold surface (prepared by electron-beam evaporation of gold on a Si[100] wafer using 10 Å of Ti as an adhesion promoter). Submerging this patterned surface in a basic solution of cyanide ion (0.01M KCN, 2M KOH) [19,25,26], with continuous stirring and bubbling of oxygen gas, removed the gold in regions of the surface not protected by SAMs [7]. Using this procedure, we have produced features having dimensions from 0.2  $\mu\text{m}$  to several hundred  $\mu\text{m}$  (figure 3) [7,8]. Figures 3(a), (b) suggest the size and complexity of features produced routinely by  $\mu$ CP and wet-chemical etching. A feature profile of 2  $\mu\text{m}$  lines of gold revealed the depth of the gold features (figure 3(c)). The smallest features of gold fabricated to date by  $\mu$ CP are lines of gold 200 nm wide that are separated by 200 nm (figure 3(d)).



**Figure 2.** Microcontact printing ( $\mu\text{CP}$ ) using an elastomeric ‘stamp’ and alkanethiol ink formed patterned SAMs on gold: the pattern of the SAMs formed by  $\mu\text{CP}$  corresponded to the features of the masters used to cast the elastomeric stamps (a), (b). Scanning electron micrographs of masters used to cast elastomeric stamps. Standard lithographic techniques were used to fabricate the masters. The relief in the features of the masters was between 0.8–1.5  $\mu\text{m}$ . (c), (d) Scanning electron micrographs of patterned SAMs formed by  $\mu\text{CP}$  with stamps cast from masters in (a, b). Microcontact printing with  $\text{CH}_3(\text{CH}_2)_{15}\text{SH}$  (light regions) formed the initial pattern; washing the surface with  $\text{HO}(\text{CH}_2)_{15}\text{SH}$  (dark regions) formed SAMs (terminated by OH) on gold not derivatized by the  $\mu\text{CP}$  step.

Typically, hexadecanethiol ( $\text{CH}_3(\text{CH}_2)_{15}\text{SH}$ ) was the best resist for etching experiments. Longer chain alkanethiols provided comparable protection against the  $\text{CN}^-$  etch and were less convenient to use; alkanethiols of shorter chain length provided decreasing protection. For best cases, we have observed [8] (by SEM) 5 etchable defects/ $\text{mm}^2$  over regions of gold protected by SAMs of  $\text{CH}_3(\text{CH}_2)_{15}\text{SH}$ . These defects may arise from imperfections in the patterned SAM formed by  $\mu\text{CP}$  and contamination (dust, hydrocarbons) present in the laboratory ambient.

#### 2.4. Microcontact printing of alkanethiols on gold and wet-chemical etching produced microstructures of silicon [8–11]

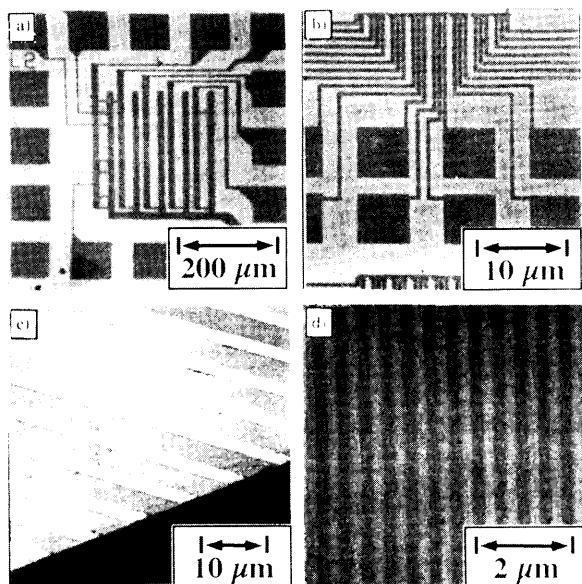
We used microstructures of gold fabricated by  $\mu\text{CP}$  and wet-chemical etching (see above) as resists for the anisotropic etching of Si. Figure 4 shows our procedure for the fabrication of Si microstructures. Microcontact printing formed a patterned SAM (figure 4(a)); exposure to  $\text{CN}^-$  solution created gold microstructures corresponding to the pattern of the SAM formed by  $\mu\text{CP}$  (figures 4(b), (c)). Anisotropic etching of the Si wafer, in areas exposed by the removal of the gold and Ti layers, was accomplished by submerging the substrate in a stirred solution of KOH (4 M) in isopropanol (15% by volume) at 60 °C for approximately 30 min (figure 4(d)) [10, 27]. Aqua regia (1:1  $\text{HNO}_3/\text{HCl}$ ) was then used to dissolve the remaining gold layer (used

previously as a resist for Si etching, figure 4(e)). Washing with a 1% solution of HF removed the  $\text{TiO}_2$  layer. The resulting structures of Si had features with sub-micrometer dimensions (figure 4(f)). Small variations in etching times resulted in slightly different features.

#### 2.5. Patterned SAMs formed by $\mu\text{CP}$ organized spatially liquids on a gold surface [8, 15, 16, 28–30]

Microcontact printing prepared patterned SAMs with well defined hydrophobic and hydrophilic regions, creating a pattern in the wettability of the surface. We exploited this pattern in the wettability of the surface to form arrays of liquids organized on the  $\mu\text{m}$  scale.

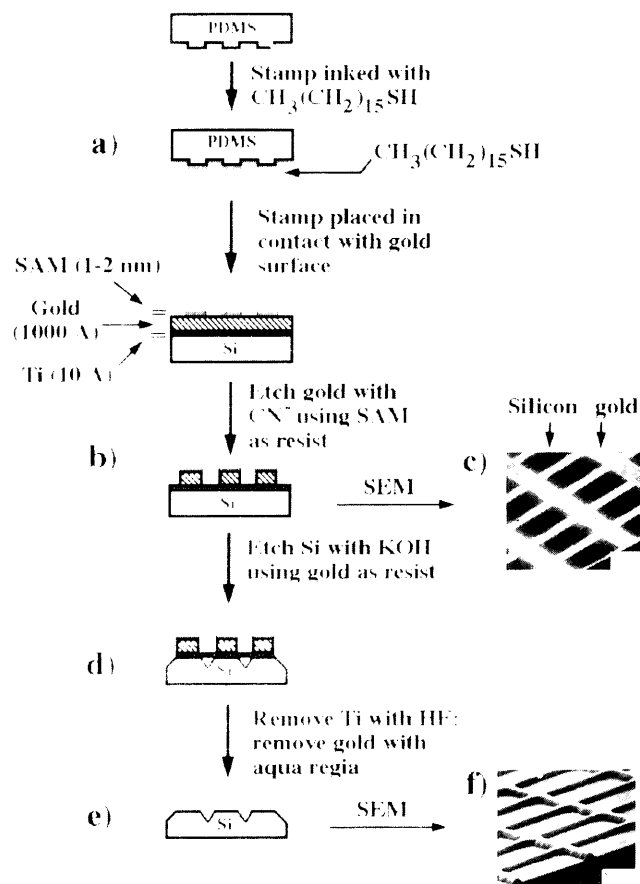
Figure 5(a) shows a SEM image of a patterned SAM formed by  $\mu\text{CP}$  with regions terminated by  $\text{COOH}$  (darker, hydrophilic) or  $\text{CH}_3$  (lighter, hydrophobic). When the temperature of the surface was lowered, droplets of water condensed selectively on the hydrophilic regions [16, 28]. These ‘condensation figures’ provided an image of the patterned SAM surface [28]. Condensation figures on patterned SAMs also acted as optical diffraction gratings: following formation of a condensation figure, the light from a He:Ne laser ( $\lambda = 632.8 \text{ nm}$ , 1 mW) reflected off the surface giving a diffraction pattern (inset, figure 5(a)) [16]. The patterned SAM in figure 5(a) had a lattice of small features with small spacing and a lattice of larger features with larger spacing. Two periodicities corresponding to these different lattices were evident in



**Figure 3.** Microcontact printing and wet-chemical etching produced microstructures of gold. (a, b) SEM images of microstructures of gold fabricated by μCP with  $\text{CH}_3(\text{CH}_2)_{15}\text{SH}$  on a 2000 Å-thick gold surface and etching with a  $\text{CN}^-$  /2M KOH solution. Light areas of the image corresponded to gold not removed by the  $\text{CN}^-$  etch: the patterned SAMs protected these regions of gold. Dark areas of the image corresponded to regions of the sample where the etch removed gold not protected by SAMs. (c) SEM image showing a fracture profile of gold lines fabricated by μCP and chemical etching. The width of the gold lines is 2 μm, and the thickness of the gold is ~ 2000 Å. (d) SEM image of 200 nm wide gold lines with 200 nm wide spacing fabricated by μCP and etching. Features on scales of 1 μm are produced easily through μCP by hand; 200 nm features such as those shown in (d) are the smallest features fabricated to date by μCP and are not achieved routinely.

the diffraction pattern. Quantitative measurements of the intensities of the diffraction spots measured the time-evolution of condensation figures formed on patterned SAMs. Since the conditions (temperature, humidity) of the surrounding environment influenced directly the evolution of a condensation figure, this type of analysis produced a simple humidity sensor [16].

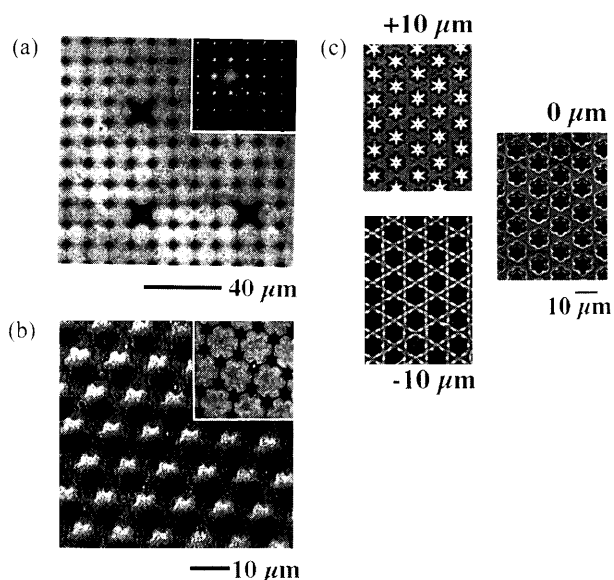
Passing patterned SAMs through an interface between water and an immiscible hydrocarbon fluid formed droplets of hydrocarbon localized on hydrophobic regions of the surface [12, 29, 30]. Figure 5(b) shows drops of hexadecane organized on hydrophobic regions of a SAM terminated by  $\text{CH}_3$  (lighter, inset figure 5(b)) and  $\text{COOH}$  (darker, inset figure 5(b)). The shape of these drops reflected the shape of the hydrophobic regions in the patterned SAM surface. We formed permanent structures using a UV-curable optical-quality polyurethane (NOA 60, 'Norland') as the hydrocarbon liquid. These polyurethane structures acted as microlens arrays (figure 5(c)) [12, 29].



**Figure 4.** Microcontact printing and wet-chemical etching formed Si microstructures with ~ 1 μm features. (a) Microcontact printing with  $\text{CH}_3(\text{CH}_2)_{15}\text{SH}$  formed patterned SAMs on a 1000 Å-thick gold surface: the gold surface was deposited by electron beam evaporation on a Si[100] wafer, with ~ 10 Å of Ti as an adhesion promoter. (b) Immersing the sample in a basic  $\text{CN}^-$  solution for approximately 15 min removed the gold film in regions not derivatized by SAMs. (c) SEM image of a patterned SAM/gold formed by μCP with  $\text{CH}_3(\text{CH}_2)_{15}\text{SH}$  and etched selectively. Dark regions in the image corresponded to regions unprotected by SAMs and etched away to expose the underlying Si; light regions in this image corresponded to gold derivatized with SAMs and protected during etching. (d) Gold structures formed by μCP and etching were resists for anisotropic etching of Si. Submerging the substrates in a stirred solution of KOH (4 M) in isopropanol (15% by volume) at 60 °C for ~ 15 min etched anisotropically Si not protected by gold. (e) The remaining gold layer was removed with aqua regia (1:1  $\text{HNO}_3$ : $\text{HCl}$ ); the Ti layer was removed by washing with a 1% HF solution. (f) SEM image of Si microstructures formed by etching the sample in (c). The SEM images in (c) and (f) were obtained with the sample tilted by approximately 70° to improve imaging of the surface relief of the sample. The tilt of the sample distorts the image: the rectangular patterns in (c) and (f) would appear as squares if the sample were not tilted.

**2.6. Patterned SAMs with regions of different wettability directed the formation of microcrystals and microcrystal arrays [8]**

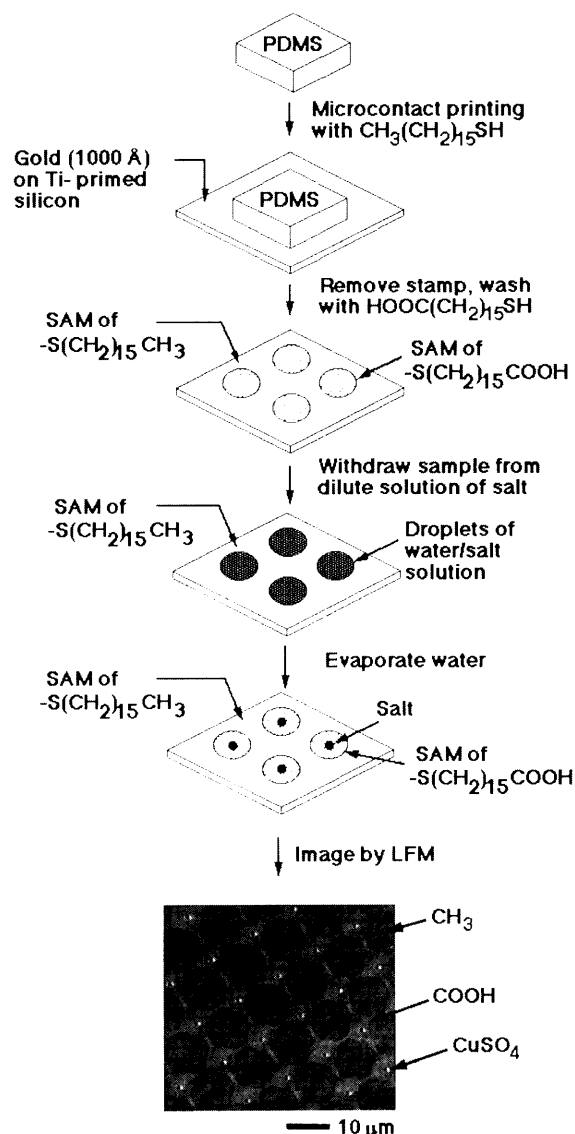
We demonstrated the principle of patterned crystallization by producing an array of droplets that contained dissolved salts, and crystallizing these salts by evaporation of the droplets. Microcontact printing formed patterned SAMs with regions terminated by hydrophilic ( $\text{COOH}$ ) and



**Figure 5.** Patterned SAMs formed by  $\mu$ CP organized liquids into simple three-dimensional structures: the spatial arrangement of these structures on the surface reflected the pattern in the wettability of the surface. (a) SEM image of a patterned SAM formed by  $\mu$ CP. Dark regions corresponded to SAMs terminated by COOH; light regions corresponded to SAMs terminated by CH<sub>3</sub>. Drops of water condensed selectively on the hydrophilic (COOH terminated) regions of the sample: these 'condensation figures' acted as optical diffraction gratings when a He:Ne laser reflected off the surface (inset). (b) Drops of hexadecane formed on a patterned SAM with spatial arrangement and shapes corresponding to the pattern of the SAM. The inset in (b) shows an SEM image of the patterned SAM used to assemble these hexadecane drops: dark regions corresponded to SAMs terminated by COOH and light regions corresponded to SAMs terminated by CH<sub>3</sub>. (c) Polyurethane structures, similar to the hexadecane drops shown in (b), assembled on a patterned SAM on transparent gold, acted as microlenses. The array of these structures showed lensing properties; three plates correspond to the images focused at (0), above (+10  $\mu$ m), and below (-10  $\mu$ m) the focal plane.

hydrophobic (CH<sub>3</sub>) groups. By slowly withdrawing these patterned surfaces from aqueous solutions of inorganic salts, liquids containing the salts organized on hydrophobic regions of the pattern: by varying the pattern in the wettability of the surface (lines, circles, etc) we controlled the structures of the liquids on the surface (microtubules, microdrops, etc).

Figure 6 shows the procedure for producing microcrystals and microcrystal arrays, and shows an image (obtained by lateral force microscopy [31] (LFM)) of a representative sample. Microcontact printing with CH<sub>3</sub>(CH<sub>2</sub>)<sub>15</sub>SH formed the initial hydrophobic pattern (dark crosses in LFM image [24]); washing with HOOC(CH<sub>2</sub>)<sub>15</sub>SH formed a hydrophilic SAM on regions of the gold not derivatized by the  $\mu$ CP step (light ovals with channels in LFM image [24]). This sample was withdrawn slowly (less than 2 cm s<sup>-1</sup>) from a 1 M CuSO<sub>4</sub> solution, forming an array of microdroplets containing dissolved CuSO<sub>4</sub>. Evaporation of these droplets at room temperature (27 °C) and ambient humidity (~40–60%) formed a periodic array of CuSO<sub>4</sub> microcrys-



**Figure 6.** Patterned SAMs formed by  $\mu$ CP directed spatially the deposition of inorganic solids on a SAM surface, forming microcrystals and microcrystal arrays. Microcontact printing with CH<sub>3</sub>(CH<sub>2</sub>)<sub>15</sub>SH and washing with HOOC(CH<sub>2</sub>)<sub>15</sub>SH formed a patterned SAM with regions terminated by CH<sub>3</sub> (hydrophobic, dark crosses in LFM image) or COOH (hydrophilic, light ovals with channels in LFM image). Withdrawing the sample slowly from a dilute (1 M) solution of CuSO<sub>4</sub> organized microdroplets of water containing dissolved CuSO<sub>4</sub> on the patterned surface. Evaporation of these droplets formed an array of CuSO<sub>4</sub> microcrystals. Imaging this sample by lateral force microscopy (LFM) revealed contrast between regions of the SAM terminated by CH<sub>3</sub> (dark) or COOH (light) and between the CuSO<sub>4</sub> and the SAM surface. Individual microcrystals had a diameter of ~1  $\mu$ m and height of ~250 nm (measured by atomic force microscopy (AFM)).

tals on the surface. The CuSO<sub>4</sub> crystals were localized approximately in the center of the hydrophilic regions: evidently, the droplets withdrew from the edges of the hydrophobic regions (during evaporation) before the CuSO<sub>4</sub> deposited. The crystals in figure 6 were ~1  $\mu$ m in diameter and ~250 nm high (relative to the plane of the SAM) by AFM [32]. Reducing the concentration of the CuSO<sub>4</sub> solution reduced the size of the crystals: a 0.1 M solution

of  $\text{CuSO}_4$  produced  $0.5 \mu\text{m}$ -diameter  $\text{CuSO}_4$  crystals in an extended (several mm) patterned array [32]. Using a similar procedure, we formed patterns of microcrystals with other salts ( $\text{KI}$ ,  $\text{LiClO}_4$ ,  $\text{K}_3\text{Fe}(\text{CN})_3$ ) [8]. For these salts, changes in the temperature and relative humidity of the environment during evaporation resulted in crystals with different shapes and morphologies [8].

### 3. Conclusions

Microcontact printing, which uses a patterned elastomeric stamp to deliver alkanethiols to a gold surface, is at present the most flexible technique for forming patterned SAMs of alkanethiolates on gold. Although the technique is experimentally simple—with the exception of the photolithographic processing of the masters, the experiments described in this paper were conducted in a conventional chemical laboratory—patterned SAMs with complex features having micrometer dimensions are produced routinely.

We have described several applications for  $\mu$ CP in the area of microfabrication. The capacity to form patterned SAMs, and the ability of SAMs to act as nm resists against selected wet-chemical etchants, allowed the formation of microstructures of gold and Si [7, 8, 10, 11]. The ability to pattern the wettability of surfaces by  $\mu$ CP allowed for the organization of liquids on gold surfaces: applications included condensation figures of water [16], microlenses and microlens arrays formed from organic liquids [12, 29], and patterned arrays of microcrystals [32].

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