Capillary Force Lithography: Large-Area Patterning, Self-Organization, and Anisotropic Dewetting

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This article gives an overview on a new lithographic technique called capillary force lithography for large-area patterning. The technique simply involves placing a polydimethylsiloxane mold on a polymer film, which is then heated above the glass-transition temperature of the polymer. Various useful microstructures can be obtained by sequential applications of the technique through self-organization. Dewetting, which can be observed in capillary force lithography for relatively thin films, is also described as a new pathway for realizing anisotropic dewetting.

1. Introduction

Future integrated circuits will require reliable lithographic techniques for fabricating nanometer-scale devices. Photolithography or optical lithography, which has been and will be the mainstay of lithography for the near future, is expected to be limited to ~100 nm resolution. Because of the limitations photolithography has for sub-100 nm features, extensive efforts have been made during the last decade to develop new patterning methods that can replace photolithography. The efforts have been fruitful and given birth to several novel techniques such as capillary force lithography,[1] nanoimprint lithography,[2-3] and soft lithography.[4-9]

“Nanoimprint lithography” (NIL)[2] generates resist relief patterns in a thermoplastic layer such as polystyrene by physically compressing the resist that has been thermally softened, rather than by modifying the resist’s chemical structure by irradiation. An anisotropic etching process, such as reactive ion etching (RIE), is used to remove the residual resist in the compressed region. In the typical NIL (hot-embossing lithography), a clean release of the resist from the mold is one of the critical requirements. Usually, anti-adhesion agents are used to achieve a clean mold release.[2] Distortion of the imprinted structures or mold features due to the thermal cycle of heating followed by cooling is another problem to contend with.

To overcome these shortcomings, room-temperature nanoimprint lithography (RT-NIL)[10] has recently been developed based on a plastic flow with free-volume contraction and plastic deformation of the resist layer. This room-temperature process has the unique features of enabling step-and-repeat and multiple imprinting, which is impossible using the conventional high-temperature imprint processes.

However, the imprinting method requires a very high pressure, typically on the order of 10⁷ Pa (N/m²), which could cause breakage of the substrate. In addition, a recent study[11] reported that material transport limits the performance of NIL in general, especially when the mold is negative or has recessed features within a large elevated surface level. In this case, the pattern transfer turned out to be unsatisfactory and bubble-like defects were observed, possibly due to a lack of conformal contact. As a result, much effort has been made in our group to lower the contact load, which led to two unique methods. One is the solvent treatment of resists,[12] which softens the polymer layer with a suitable solvent and enables room temperature processing. The other is the introduction of an elastomeric mold such as polydimethylsiloxane (PDMS), which has led to the advent of capillary force lithography.[1]

The elastomeric polymer, PDMS, has extensively been used for a number of useful techniques, which are often referred to as “soft lithography”, a term that was originally proposed by Whitesides and co-workers. The lithography involves the use of PDMS to print patterns of “inks” (microcontact printing or μCP),[3] to mold polymers (micromolding in microcapillaries, or MIMIC,[4] solvent-assisted microcontact molding, or SAMIM,[5] microtransfer molding, or μTM,[6] or replica molding[7-8]), or to perform photolithography in the near optical field.[9]

By simply combining nanoimprint and soft lithographies, we developed a new lithographic technique called “capillary force lithography” (CFL).[1] When a patterned (positive or negative) PDMS mold is placed on a polymer surface and heated above the polymer’s glass-transition temperature ($T_g$), capillarity forces the polymer melt into the void space of the channels formed between the mold and the polymer, thereby generating a negative replica of the mold as shown in Figure 1. When the polymer film is thick enough to completely fill the cavity of the
mold, a residual polymer layer remains on the substrate as shown in Figure 1A. However, if the polymer film is thin and the interaction between the polymer and the substrate is sufficiently weak, the substrate surface can be exposed as depicted in Figure 1B. This experimental procedure is similar to that for nanoimprint lithography except that a hard mold is replaced by a soft mold and no pressure is applied.

In CFL, the essential feature of nanoimprint lithography—molding a polymer melt—is combined with the prime element of soft lithography—the use of a PDMS mold. As a result, the advantage of the imprint lithography over the soft lithography is retained in meeting the stringent pattern fidelity requirements as in the fabrication of integrated circuits while eliminating the need to use an extremely high pressure that is needed in nanoimprint lithography.

This article is divided into three sections. In the first section, we briefly summarize our recent experimental results on large-area patterning with an emphasis on direct exposure of the substrate and related pattern transfer. In the second, we present self-organization as a new route to physical self-assembly. In the last section, we deal with dewetting of thin polymer films confined by mold walls, which could be a unique method for realizing anisotropic dewetting\cite{13} for patterning purposes. A schematic representation of the three areas is shown in Figure 2.

2. Large-Area Patterning

CFL is a powerful method for patterning polymers on a large area. As PDMS mold spontaneously wets the surface and makes conformal contact with the substrate without any external force, an area up to ~4 x 4 cm² can be patterned in one step with high pattern fidelity. Here, we present several unique features of CFL.

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mold, a residual polymer layer remains on the substrate as shown in Figure 3A (also refer to Fig. 1A). However, if the polymer film is thin and the interaction between the polymer and the substrate is sufficiently weak, the substrate surface can be exposed as depicted in Figure 1B. In the latter case, a meniscus is observed at the protruding end of the polymer as shown in Figure 3B, which is the signature mark of the capillary rise.

In order to verify the exposure of the substrate, we fabricated 250 nm Cu lines by the Shipley electroless Cu deposition process.\textsuperscript{[14]} For the deposition, a palladium layer was first formed on the silicon substrate. A thin film (~50 nm) of polystyrene–butadiene–styrene copolymer (SBS) was then spin-coated onto the palladium layer. The CFL process was carried out and then the substrate was placed in an electroless plating solution for the deposition. Copper deposits only on the exposed surface where the palladium is exposed and not on the polymer-covered surface. After the growth, the remaining SBS was removed by dissolving in toluene. Figure 4 shows the atomic force microscopy (AFM) image of the as-grown Cu lines, verifying that the surface can be exposed by CFL. Considering the poor growth characteristics inherent in the electroless deposition, remarkably good Cu lines are seen to have formed.

\section*{2.1. Direct Exposure of Substrate}

Figure 3 shows the scanning electron microscopy (SEM) images of typical polymer patterns realized by CFL. When the polymer film is thick enough to completely fill the cavity of the mold, a residual polymer layer remains on the substrate as shown in Figure 3A (also refer to Fig. 1A). However, if the polymer film is thin and the interaction between the polymer and the substrate is sufficiently weak, the substrate surface can be exposed as depicted in Figure 1B. In the latter case, a meniscus is observed at the protruding end of the polymer as shown in Figure 3B, which is the signature mark of the capillary rise.

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\begin{figure}[h]
\centering
\includegraphics[width=0.8\textwidth]{figure4.png}
\caption{Fabrication of 250 nm Cu lines by a Shipley electroless Cu deposition process. In the process, copper grows only on the exposed surface, thus leading to alternating Cu lines.}
\end{figure}

\section*{2.2. Fabrication of Three-Dimensional Structures}

Figure 5 shows the cross-sectional SEM images of various three-dimensional and complex polymer structures formed by CFL in one step. As shown in the figure, CFL can preserve the fidelity of the original structure without causing any distortions or defects. Furthermore, the edge definition, which is an important characteristic for the subsequent etching step, is also satisfactory. Note in this regard that distorted or textured structures are frequently observed in NIL\textsuperscript{[5]} and SAMIM.\textsuperscript{[5]} If the conventional photolithography is used, several mask steps are needed to fabricate the multi-level structures in Figures 5B and 5D. These results clearly demonstrate the effectiveness of capillary force lithography in fabricating three-dimensional and complex structures.
2.3. Pattern Transfer to an Underlying Substrate

The transfer of a polymer pattern to an underlying SiO₂ substrate by reactive ion etching (RIE) can be completed in one step as shown in Figure 6 for 800 nm line-and-space and 1 μm dot patterns. A relatively thin film (60 nm) was used for the line-and-space pattern with a negative PDMS mold; a relatively thick film (170 nm) was used for the dot pattern with a positive PDMS mold. To examine the etching characteristics of the polymer resist, the etching time was varied from 5 min (Fig. 6A) to 15 min (Fig. 6B) for the line-and-space pattern. One notable finding is that the shape of the etched structure is dependent on the etching time due to the meniscus in the original polymer structure. Because of the presence of a meniscus, the minimum point of the meniscus is vulnerable to RIE as the underlying substrate will be exposed first at that point as the etching time increases. Once the substrate is exposed, both polymer and SiO₂ are etched simultaneously, thus leading to a rough surface structure as shown in Figure 6B. Therefore, a thicker film is needed to etch more deeply into the underlying substrate.

The transfer of a dot pattern was also carried out as shown in Figures 6C (before etching) and 6D (after etching). In this case, there is ample space between the dots so that the residual film thickness is not negligible. Therefore, the residual polymer is also involved in the etching process, which makes the surface of the etched SiO₂ surface a little rough. However, the pattern was successfully transferred to the underlying SiO₂ without any observable change as there is little residual polymer present in the patterned, depressed region. The roughness can be eliminated if a conventional two-step etching is used.

2.4. Nanostructures and Structures of High Aspect Ratio

Two examples of nanostructures are shown in Figure 7: one is 150 nm dots (Fig. 7A) and the other is 100 nm lines (Fig. 7B). The dot and line features on the original mold were poor due to our limited electron-beam capability, which is replicated in the structures formed. In general, however, it becomes very difficult to prepare good molds as the pattern size gets smaller. As PDMS is a very viscous melt (~3900 cP), it cannot completely fill up the cavity of the original hard mold when the cavity is deep, which often leads to a rearranged shape of the PDMS mold. In addition, the weak mechanical properties of the PDMS mold leads to broken or isolated structures for smaller features when detached from the original hard mold. For example, an aspect ratio only up to 1.4 and 1.0 could
be realized in our experiment for 800 nm and 400 nm line-and-space patterns, respectively (see Fig. 8 for an 800 nm line-and-space pattern). When the pattern size gets smaller than 100 nm, however, the maximum aspect ratio attainable would become less than 0.25, as revealed in our preliminary experiments.

![Fig. 8. An example of the polymer structure with an aspect ratio of 1.4 for an 800 nm line-and-space pattern.](image)

However, a recent study has shown that the PDMS mold prepared from mixtures of vinyl and hydroxiane compounds can provide sufficiently good mechanical properties to allow replication of high-density patterns at the 100 nm scale. With further improvement in the mold preparation, CFL may also be effective in patterning high-density sub-100 nm features.

### 2.5. Theoretical Background of CFL

A simple geometric calculation shows that if the maximum step height \( h \) of the mold is larger than the polymer thickness \( t \), i.e., \( h > t \), and only nearby polymeric material participates in the CFL process, the substrate surface can be made exposed. However, it has been observed that the whole mass of polymer in contact with the mold spontaneously migrates to planarize the film thickness such that a significantly large mass transport occurs during the CFL. In fact, one can observe two drastically different colors of the polymer film after the CFL between the molded parts and the rest for low \( T_g \) polymers such as SBS \( (T_g \sim 36 \degree C) \). As a result, one may need a mold with a step height much higher than the polymer thickness to safely expose the substrate surface.

The most important concept of CFL is that wetting of the wall of PDMS mold by polymer melt lowers the total free energy. If the interaction at the polymer/substrate interface is negligible and the polymer melt is liquid-like, the maximum height the polymer melt can rise to through a narrow channel of width \( L \) by the capillary action can be obtained by equating the capillary force to the resistant gravitational force, which gives

\[
h_{\text{max}} = \frac{2 \gamma_{\text{polymer/air}} \cos \theta}{\rho G L}
\]

where \( h_{\text{max}} \) is the maximum height, \( \gamma_{\text{polymer/air}} \) is the surface tension at the polymer/air interface, \( \theta \) is the contact angle at the polymer/mold interface, \( \rho \) is the density of the polymer, and \( G \) is the gravitational constant. From the meniscus of the cross-sectional SEM image, the contact angle is estimated to be about 85\(^\circ\) for SBS films. When we insert into Equation 1 the surface tension (~30 dyne/cm) and density (0.95 g/cm\(^3\)) of SBS and an \( L \) value of 300 nm, a maximum height of 1.87 m is obtained, which indicates that the height of the patterned polymer can be made quite large. In fact, we were able to fabricate polymer structures with a step height as high as 5 \( \mu \)m for 80 \( \mu \)m line-and-space pattern. If we take into account the interaction at the polymer/substrate interface and the solid-like properties of the polymer melt, the theoretical maximum height would become smaller. We believe that the polymer melt can fill up the cavity of any step height as long as the mold is available and the film is sufficiently thick.

The time it takes for the polymer melt to fill up the void space between the mold and the polymer film can be estimated. If we neglect the effect of gravity, then the surface tension and viscosity of the polymer melt, and the size of the capillary determine the rate of flow such that the time is given by

\[
t = \frac{2 \eta z^2}{R^2 \gamma_{\text{polymer/air}} \cos \theta}
\]

where \( z \) is the length of the capillary to be filled, \( t \) is the time, \( \eta \) is the viscosity of the polymer melt, and \( R \) is the hydraulic radius (the ratio of the volume of the liquid in the capillary section to the area of the solid and liquid interface), which is approximately one half the width, \( L \). The calculated time is in satisfactory agreement with the observed value.

Capillary force lithography is a simple and yet robust method for large-area patterning. With further development, the technique could become a strong candidate for an alternative to photolithography in fabricating large-scale integrated circuits.

### 3. Self-Organization

“Chemical” self-assembly involving a monolayer has proven to be quite fruitful. To control the chemical and structural properties of surfaces, self-assembled monolayers (SAMs) have been extensively used in many practical applications such as patterning, chemical sensing, and catalysis. Here, we introduce a concept of “physical” self-assembly on a micro-scale, involving self-organization of an existing microstructure into a new microstructure that is driven by capillarity.

The capillary forces drive the pattern formation in CFL, which does not involve any external forces or modifications. Therefore, capillarity could lead to self-organization of a polymer into an ordered structure if utilized properly. A simple meniscus forms when capillarity acts on a flat surface, but unexpected structures could result when capillarity acts upon an underlying polymeric structure, since then the reorganization dynamics of the underlying structure could lead to self-organization. When CFL is used to cause self-organization, only sequential applications of two molds are involved.

A schematic diagram of the two-step method is shown in Figure 9A. Typically, a mold is placed on the surface of a polymer layer spin-coated onto a silicon substrate. After annealing at 150 \( \degree \)C for 1 h with the first mold in place, an underlying poly-
mer structure forms as shown in Figure 9B. Then, another mold is placed on the pattern formed with this second mold rotated by a certain angle with respect to the first mold and the sample is heated above the glass-transition temperature, typically 130°C, for various annealing times.

We show first the polymeric microstructures that result when the second mold is rotated 90° with respect to the first mold (Fig. 10) and annealed at 130°C for 90 min. In this case, the underlying polymer structure had been formed with the first mold having a 700 nm line-and-space pattern (Fig. 9B). Two molds were used as the second mold, each having a line-and-space pattern with a step height of 220 nm but a line width of 1 and 3 µm, respectively. In the case of the 1 µm mold pattern, one row of holes formed along the pattern direction of the second mold, which is perpendicular to the underlying polymeric pattern as shown in Figure 10A. The holes are located at the valleys between two adjacent mesas (Fig. 9A) of the underlying polymer structure that were originally formed with the first mold. In the initial stage, the polymer on the mesa, which is in contact with the void of the second mold, selectively wets the mold walls, leading to the formation of the first meniscus within the void channel. The polymer rises continuously until it reaches the ceiling of the mold. In the meantime, a uniformly curved surface develops at the mesa position due to Laplace pressure. As the mass is continuously supplied into the void of the second mold by the capillarity from the region of the mesa that is not exposed to the void, the mass in this region gets depleted, leading to the lowering of the mesa height, and eventually the mold is in contact with the valley of the underlying polymer structure. A second meniscus starts forming when the mold becomes in contact with the valley. In the final stage, the height of the first meniscus is nearly flattened and the lowest part of the second meniscus keeps rising by the capillarity, which leads to the formation of regularly spaced holes at the center of the valley. If the annealing time increases further to 3 h, the holes are filled completely.

Two rows of holes form at the valleys instead of one for the 3 µm mold pattern as shown in Figure 10B. This feature arises from the second meniscus being broken into two, thereby forming two rows of holes near the edge of each wall. The breakdown of the meniscus is related to the competition between Laplace pressure and surface-energy minimization[20].

The step height of the underlying pattern is an important factor in the self-organization. For instance, no hole formation occurs if the step height is relatively large. Two examples are shown in Figure 11 where the step heights are 400 nm (Fig. 11A) and 600 nm (Fig. 11B), respectively. In this case, the second meniscus cannot start forming, as seen from the figure, such that isolated line structures result. Furthermore, the underlying structures are not completely destroyed even for long annealing time.

Next, we show the microstructures that result from the self-organization when the second mold is oriented 45° with respect to the first. Figure 12 shows typical results for the 1 and 3 µm patterns when annealed at 130°C for 40 min. In this case, twisted microstructures form with an apparent directionality. The directionality of the polymer along the wall is quite surprising as the polymer is expected to move freely in every direction. The phenomenon can be understood from the wet-
ting behavior that tends to drive the system toward meeting the equilibrium contact angle in both directions.

As was the case with SAM, the self-organized microstructures presented here would open the door to a variety of applications in biomedical, electronic, and optical areas.

4. Anisotropic Dewetting

Dewetting of a liquid film on a substrate has drawn much interest in the last ten years due to a fundamental interest in basic wetting phenomena and potential applications to nanopatterning technology.13,21-27 Much of the theoretical and experimental work has been devoted to polymer films because of the technological importance and convenient experimental time scale that polymer films offer due to their high viscosity.13,25-27

Recently, anisotropic spinodal dewetting of polymer films has been reported13 as a route to patterning surfaces on the nanometer scale. Due to the isotropic nature in spinodal dewetting, the authors used mechanically treated substrates, which gives rise to the anisotropic behavior.

Here, we present an anisotropic dewetting of the polymer films confined laterally by PDMS walls when CFL is carried out without any treatment of the substrate. As the annealing is prerequisite to CFL, dewetting of thin polymer films cannot be avoided if the annealing time exceeds a certain incubation time. However, if the film is thick enough such that the mold void is completely filled, no dewetting takes place. Only when the film is thin such that the mold void is partially filled would the dewetting be initiated above the glass-transition temperature (see Fig. 1B).28

Figure 13 shows a three-dimensional AFM image of the typical dewetting structure of laterally confined SBS films for a 1 µm line-and-space pattern. The pattern consists of six strips: five separated and one isolated line. The samples were annealed at 120 °C for 27 h. Transient morphologies26 reveal that up to 19 h, there is no observable change on the polymer surface. However, regularly spaced holes are detected after 21 h and some holes grow across the strip. Finally, all the holes propa-
gate to the edge in the lateral direction, which leads to the regularly separated block formation (Fig. 13).

The incubation time that is needed for the onset of the instability depends on the film thickness and annealing temperature. For example, it is about 20 h for 67 nm thick films and about 12 h for 42 nm thick films at 120 °C for SBS. If the temperature increases to 150 °C, the incubation time decreases drastically to 1–3 h.

One notable finding in our dewetting experiment is that the wavelength of the instability is on the order of micrometers. A capillary wave model predicts that the fastest growing mode in the film is given by[23]

$$q = a h^2$$

(3)

where $a = (A_{\text{eff}}/4\pi)^{1/2}$, $A_{\text{eff}}$ is the effective Hamaker constant for the van der Waals interaction of the film with the surrounding media, $\gamma$ is the surface tension of the polymer, and $h$ is the film thickness in the channel. The effective Hamaker constant in our experimental geometry should be expressed in terms of three Hamaker constants that are responsible for three interfaces (polymer–air, polymer–substrate, and polymer–PDMS wall). However, its value ranges between $10^{-20}$ and $10^{-19}$ J regardless of the individual Hamaker constants.[29] Therefore, the conventional capillary wave model gives a wavelength on the order of several hundred of micrometers for the conditions used in our experiment. If we adjust the effective Hamaker constant to fit the observed value, it takes a value higher than $10^{-17}$ J, which is not realistic. Furthermore, the theory predicts that the hole density in a given strip length should be correlated with $h^{-2}$. In our experiment, the power index range between $-0.7$ to $-1$, which deviates severely from $-2$ that is predicted from the capillary wave model. The power index increases from $-1$ to $-0.7$ as the strip width decreases. This fact reveals that the conventional capillary wave model cannot explain our observations.

One possible origin that is responsible for this anomaly is the viscoelastic properties of the polymer films. Wang et al.[27] reported that the power index is not 2 but rather ranges from 0.8 to 1 for high molecular weight polyvinyl pyridine (PVP) and polystyrene (PS) ($M_n = 9 \times 10^3$) thin films. They attributed this anomalous behavior to solid-like or viscoelastic properties of the polymers as PVP and PS have high molecular weights and strongly react with SiO$_2$ and Si substrates, respectively. As the molecular weight of the polymer used in our experiment is sufficiently high ($> 10^4$) for both SBS and PS, it would show a more solid-like behavior such that elastic effects may come into play.[30] Another possible origin is the thermal stress generated at the film-substrate interface due to a thin glass layer.[31] If there exists a thin glass layer at the interface, which is frequently observed for high molecular weight polymers, the built-in thermal stress plays an important role in the dewetting, which can modify the dewetting behavior. The exact origin of the anomalous dewetting behavior is currently under study.

The simple experimental method proposed in this section would be quite useful for the studies of polymer films with anisotropic geometry.

5. Conclusions and Outlook

In this article, we have described capillary force lithography as a tool for general-purpose lithography for large-area patterning. The technique was originally developed to overcome a few shortcomings of imprint lithography such as limited mass transfer and high contact load. In the course of studying CFL for the patterning purpose, we subsequently found that self-organized polymeric microstructures can be made by sequential applications of the technique and that anisotropic dewetting occurs for relatively thin polymer films.

Capillary force lithography is a simple and yet effective tool for patterning. It does away with the high-pressure requirement of the imprint lithography while maintaining the excellent pattern fidelity offered by the method that soft lithography cannot match. A feature unique to CFL is that the photographic lithography involving exposing, developing, and baking can be accomplished in one simple step without involving reactive ion etching, which other non-photographic techniques require. The ease with which three-dimensional and complex structures can be fabricated is another advantage. For the method to be applicable to high-density patterns with a feature size less than 100 nm and a high aspect ratio, however, better mold preparation techniques should be developed.

In the area of self-organization, we have found that simple sequential applications of CFL lead to various useful polymeric microstructures. The dynamics of capillarity renders itself to self-organization of polymeric microstructures when a mold is placed on the underlying microstructure that was formed with the first mold. The pattern shapes of the two molds and the rotation angle of the second mold with respect to the first together with the annealing time and temperature can be used to obtain various self-organized microstructures. This physical self-assembly opens the door to applications in optics and separation.

In the area of dewetting, we have discovered that anisotropic dewetting takes place when CFL is applied to relatively thin films of polymer, given a sufficient time. This new route to dewetting cannot be explained by the conventional theories. Since the experimental method is quite simple, it would serve as an effective technique for studying the polymer behavior under confined microstructures, which could lead to unexpected findings.

Capillary force lithography is in its infancy and yet the findings so far exceed expectations that we had. With further development, it would open the door to new discoveries and useful applications in the areas of patterning, self-organization, and dewetting.

Received: February 25, 2002

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