Colloidal pattern replication through contact photolithography operated in a ‘Talbot–Fabry–Perot’ regime

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Abstract
We describe a method for continuous colloidal pattern replication using contact photolithography. Cr-on-quartz masks are fabricated using colloidal nanosphere lithography and subsequently used as photolithography stamps. Hexagonal pattern arrangements with different dimensions (980, 620 and 480 nm, using colloidal particles with these respective diameters) have been studied. When the mask and the imaged resist layer were in intimate contact, a high fidelity pattern replica was obtained after photolithographic exposure and processing. In turn, the presence of an air gap in between was found to affect the projected image on the photoresist layer, with a strong dependence on the mask feature size and height of the air gap. Pattern replication, inversion and hybridization were achieved for the 980 nm period mask; no hybridization for the 620 nm one; and only pattern replication for the 480 nm one. These results are interpreted in the framework of a ‘Talbot–Fabry–Perot’ effect. Numerical simulations corroborate the experimental findings, providing insight into the processes involved and highlighting the important parameters affecting the exposure pattern. This approach allows complex subwavelength patterning and is relevant for three-dimensional layer-by-layer printing.

Keywords: Talbot effect, photolithography, subwavelength, replication
(Some figures may appear in colour only in the online journal)

1. Introduction
Colloidal particle assisted nanostructuring has become a powerful tool in nanotechnology [1–7]. The common approach involves sacrificial use of a colloidal monolayer and subsequent pattern transfer via surface or bulk structuring. While this approach has provided valuable results and access to structuring techniques and configuration that are otherwise difficult to attain, the original colloidal mask is destroyed and has to be fabricated repeatedly [8–11]. Many approaches have been developed for large-scale and easy assembly of colloidal particles, yet the one-time use methodology renders this approach less attractive. For example, nanoimprint lithography and photolithography are of interest for industrial applications because they allow repeated utilization of the mask and continuous processing. Merging colloidal lithography with one of these techniques is interesting because the cost of mask (master) fabrication could be diminished by using self-assembly techniques if the mask could be repeatedly used replicating the original colloidal pattern.

In this paper we explore this route and find that hexagonal arrangements of circular patterns can be easily produced using
a colloidal-templated Cr-on-quartz photolithography mask. As such, colloidal self-assembly is performed only once but this allows pattern photoreplication for virtually an infinite number of times. The mask feature size and arrangement are defined by the colloidal lithography mask (the diameter of the pristine particles defines the spacing between apertures whereas the size reduction step sets the opening diameter). The photolithography in hard-contact mode resulted in a series of patterns, dependent on the feature dimensions of the mask and especially on the air-gap distance between the mask and the imaging plane (photoresist layer).

A Talbot-like effect is behind the origin of those patterns. The Talbot effect, also known as the self-imaging effect, consists of periodic repetition of an image projected through a diffraction grating [12–15]. When a plane wave is transmitted through a periodic diffraction grating, repetitive images of the grating itself are obtained. The Talbot distance is then defined as the distance from the source where the first regular image is projected, also called Talbot image. Talbot imaging has a fractal nature with intermediate images appearing at smaller distances than the Talbot distance. These sub-images are characterized by a smaller feature size and pattern hybridization. Moreover, some works consider the Talbot distance in photolithography. For instance, the Talbot effect can be used to produce complex three-dimensional (3D) periodic patterns. Nevertheless, in the present work, while the typical Talbot distance should be in the micrometer range we show that complex patterns occur for a small air-gap distance. This is due to the fact that the Cr-on-quartz mask and silicon substrate of the photoresist layer act as a Fabry–Perot’ cavity which squeezes the Talbot effect. In the following the experimental set-up is described in section 2. Results are introduced and discussed thanks to numerical simulations in section 3.

2. Experimental section

The experimental part of this work is schematized in figure 1. Polystyrene colloidal particles with different nominal diameters were used in this study as-received (Microparticles GmbH; 480, 620 and 980 nm). Nanosphere lithography (NSL; figure 1(a)) was performed using previously documented techniques on quartz wafers [11, 16, 17]. Reactive ion etching (RIE; figure 1(b)) was used to reduce the size of the spherical particles. Next, ≈80 nm of metallic Cr was deposited by physical vapor deposition (PVD) followed by lift-off using adhesive tape (figures 1(c) and (d)). To remove any residual organic layer, samples were sonicated in dichloromethane and exposed to a short O2 plasma ashing. At this stage, the Cr-on-quartz mask was ready for use.

For the photolithography experiments, 180 nm of KMPR-series negative resist was coated on Si chips. In order to attain such thin photoresist films, the commercially available KMPR1025 formulation (MicroChem Corp.) was diluted (1/9 volumetric ratio) with cyclohexanone (Sigma Aldrich, boiling point 156 °C). No anti-reflective coating layer was used. Prior to the irradiation, the resist was soft-baked at 100 °C for 20 s (figure 1(e)). The exposure was done using a K&W Karl Süss mask aligner operated in hard-contact mode (Hg-lamp, i-line, cold mirror filtered; exposure wavelength centered at 365 nm, 205 W power) (figure 1(f)). The exposure time was varied between 2 and 3 s depending on the mask type. The resist was then post-baked at 100 °C for 15 s and the imaged pattern was revealed in a standard developing formulation (AZ 726 MIF) for 30 s at ambient temperature (figures 1(g) and (h)). The rather short soft-baking time (20 s at 100 °C) was found to be insufficient to completely remove the solvent and the coated films were occasionally found to be adherent and easy to indent (see further discussions). Nonetheless, the detailed protocol was found to be optimal for attaining high photolithographic contrast and resolution.

Morphological characterization was performed using scanning electron microscopy (SEM; XL 30 FEG) and optical microscopy. Maps of the square modulus of the electric field were obtained from a homemade numerical code which uses a rigorous coupled wave analysis (RCWA) approach jointly with a scattering matrix method [18–20].

3. Results and discussions

3.1. Description of the procedure

A schematized view of the experimental approach used in this work is shown in figure 1. As can be seen, the NSL is done only once, during the mask fabrication step, and the fabricated Cr-on-quartz mask is used repeatedly to replicate the NSL pattern. We have used the fabricated mask for more than 20 times without any sign of degradation. The only precaution required was rinsing the solvent from the mask after each utilization to remove the photoresist residues found to affect the quality of the photolithographic process. We chose to work with NSL for mask fabrication as it allowed us to rapidly fabricate and scan the influence of the feature size of the mask (just by simply using colloids of different sizes) on the photolithographic outcome. Furthermore, photolithographic masks are usually fabricated using electron-beam lithography (especially when sub-micrometer features are targeted), which is a time-consuming serial writing technique. In contrast,
colloidal lithography as a bottom-up approach can be performed over large areas, in a relatively short time and at low cost. Whilst the drawback is the constrained pattern design, some applications require just periodic nanostructuring without any special pattern designs like anti-reflective and super-hydrophobic surfaces or mass production of nanostructures for various applications [21–23].

### 3.2. Mask fabrication

A colloidal particles monolayer with hexagonal symmetry is obtained after NSL. The top view of an assembled monolayer composed of colloidal particles with a diameter of 980 nm is shown in figure 2(a). After the O₂ RIE step, the close-packing of the spheres is altered while the hexagonal arrangement is preserved (figures 2(b) and (c)). After Cr-metal deposition and lift-off, a holey-metal film is obtained. The elevation of the quartz plane with respect to the photoresist plane is expected to be of the order of 100 nm (corresponding to the thickness of the Cr layer). It is worth mentioning here that photoresist residues were often found on the quartz areas on the mask and pale iridescence was observed on the photoresist layer after exposure without being developed (data not shown here). This presumably implies that Cr features are partially percolating into the photoresist layer or, in other words, photoresist and the quartz plane are brought into intimate contact. The hard-contact mode used for the lithographic step, the soft nature of the photoresist layer as well as partial bake-out may be behind these experimental observations. However, the thickness of the Cr layer is only 100 nm and, as will be shown later, the critical separation is much higher than the above-mentioned value. Depending on the type of resist (positive or negative), a direct or inverted replica, respectively, can be obtained after exposure and revealing. Here we have used a negative tone photoresist (KMPR series). Consequently, holes in the Cr-on-quartz mask will normally yield photoresist posts after processing. To avoid thin-film interference, the thickness of the photoresist layer was set to 180 nm. As such, the pattern obtained after processing can be approximated to the plane projected image of the light passing through the holey-metal Cr-on-quartz mask.

Examples of fabricated masks with different feature parameters are shown in figure 3. Three mask types have been used, fabricated using different colloid diameters: mask M1, lattice constant \( S_{cc} = 980 \text{ nm} \) and hole diameter \( \varnothing = 540 \text{ nm} \); mask M2, \( S_{cc} = 620 \text{ nm} \) and \( \varnothing = 400 \text{ nm} \); mask M3, \( S_{cc} = 480 \text{ nm} \) and \( \varnothing = 380 \text{ nm} \). Careful inspection over the entire area of the mask showed that there are no inverted patterns (i.e. metallic dots) and there were no impurities at the center of the holes. These observations are important because inspection of the photolithographic results revealed the presence of unexpected patterns, especially for M1, not due to the presence of defects on the mask. These surprising patterns are related to the Talbot effect, as discussed later.

### 3.3. Lithography results

The photolithography results are shown in figure 3 (bottom row). Arrays of resist posts were primarily obtained, consistent with the processing conditions (openings in a Cr-on-quartz mask and negative tone photoresist). The dots have circular symmetry and preserve the hexagonal arrangement for all three types of mask. While the dot lattice spacing is fixed as defined by the imaging mask, their diameter can be modified by adjusting the exposure dose and post-processing parameters (developer concentration and time). Using mask M3 (figure 3, bottom row), the diameter of the dots is close to the resolution limit of \( \lambda/2 \approx 200 \text{ nm} \). Indeed, as shown later, the photoresist layer is still located in the radiative near-field zone, i.e. the Fresnel zone, and not in the non-radiative near-field region. As such, a simple mask fabrication procedure combined with standard photolithography enabled fast and large-area replication of 200 nm features.

Another interesting finding was the observation of inverted and multiplied (along with normal) patterns after photolithographic processing. Examples are shown in the bottom row of figure 3. Such a feature was not observed for mask M3, for which only photoresist posts were obtained. Using mask M2, pattern inversion was observed: instead of obtaining photoresist posts, holes in the photoresist films were obtained. Furthermore, three types of patterns were obtained using mask M1: normal photoresist posts, inverted holey photoresist film and a combination of both, i.e. holey films with posts at the interior of hole (donut-like structures). It should be noted here that even though hard-contact mode photolithography was used, due to the processing defects and non-planarity...
of both the mask and carrier sample, conformal it was not possible to achieve contact between both elements over the entire area of the sample. The presence of an air gap in between was visually identified by the appearance of the interference patterns (but limited to less than 30% of the entire sample area). The above-mentioned pattern shape anomalies were mainly observed in the respective areas and could be seemingly attributed to the air gap. We did not perform any extended experiment to precisely control the gap spacing between the imaging (Cr-on-quartz mask) and the imaged (photoresist layer) planes. While this could be implemented easily by deposition of a transparent dielectric layer [13] of desired thickness either on the mask or on top of the photoresist, we centered our interest in numerically interpreting the obtained results and finding the critical parameters that affect the outcome of the photolithographic processing [15].

3.4. Modeling

Simulations provide the square modulus of the electric field (i.e. the field intensity) and are obtained from a homemade numerical code which uses a RCWA approach jointly with a scattering matrix method [18–20]. The simulation configuration is depicted in figure 4. The Cr-on-quartz mask is numerically represented as an opaque film bearing hexagonally arranged holes. The thickness of the Cr layer is set to 75 nm while the quartz is considered to be of infinite thickness. Similarly, the silicon supporting substrate is simulated as infinitely thick while the photoresist is 180 nm thick. The
air gap (h) is defined as the distance between the upper surface and the lower boundary of the photoresist and the Cr layer, respectively. Assuming that \( \lambda = 365 \) nm, the optical constants of the materials are as follows: \( n_{\text{Cr}} = 1.40 + i3.26 \), \( n_{\text{quartz}} = 1.57 \), \( n_{\text{Si}} = 7.26 + i1.27 \), \( n_{r} = 1.61 + i5 \times 10^{-4} \) for chromium, quartz, silicon, [24] and resin (KMPR), respectively. In each figure, \( h \) is the separation between the chromium/air and the resin/air interfaces in the modeled system (see figure 4). In each figure, the electromagnetic field intensity scales are given for an incident electric field (in quartz substrate) equal to 1 V m\(^{-1}\).

Figure 5 shows the simulation and experimental results for the process using mask M1. For an air gap of 550 nm or less, direct imaging is observed, i.e. arrays of photoresist posts (bottom SEM image). It should be mentioned that no other patterns were observed in the respective areas. Simulation shows that the reverse pattern (middle SEM image) is obtained for \( h \) around 625 nm, whereas a hybridized pattern (top SEM image) is recovered for \( h \) about 650 nm. All other \( h \) values were found to recover the bottom SEM image. Interestingly, a small variation in \( h \) (≈25 nm) is found to dramatically affect the projected image, requiring precise mask and air-gap engineering/design if the respective patterns are targeted. Close inspection of the inverted and hybridized pattern area revealed intermixing of the respective patterns within. For example, in the inverted pattern (holey polymer film), polymer posts were occasionally found in the middle of the hole, and, conversely, the absence of the polymer post was noted in the hybridized pattern (marked with arrows in figure 5). The very small separation \( h \) required to differentiate the two patterns and possible non-uniformities of the mask features (for example small variation in the colloid size leading to different hole size) are the reasons for the pattern mixing.

Figure 5(c) shows the simulation results for conditions similar to those presented in figure 5(b), except that the silicon substrate and the resin layer were numerically removed in order to propagate the electromagnetic field in true Talbot conditions [15]. We clearly see that the electromagnetic field patterns strongly differ from those obtained in figure 5(b). This shows that the present experimental results depend on the interferences between the electromagnetic wave arising from the chromium mask and the reflected wave on the silicon–resin interface, which acts as a mirror. It should be noted that in the experiments no anti-reflective layer was used on silicon substrate. Eventually, the use of such a layer would eliminate mirror–cavity interference and lead to direct pattern replication without any special care with respect to the air-gap height [13].

Having confirmed the applicability of the simulation protocol to the observed experimental results, we analyzed the results obtained using masks M2 and M3. Figure 6 shows the experimental results obtained using mask M2 and the corresponding simulated electromagnetic field intensity at the surface of the photoresist layer. The inverted pattern, i.e. holey photoresist film (bottom SEM image), is roughly recovered for \( h \) between 500 and 600 nm. The top SEM image (the normal pattern) is obtained for the other \( h \) values (higher than 600 nm and lower than 500 nm). Pattern mixing was not observed here, either experimentally or in the simulations. The results for mask M3 are shown in figure 7. In this configuration, only direct patterning was observed. Polymer posts were obtained independently of the \( h \) value.

3.5. Discussions

As previously mentioned, the Talbot effect consists of periodic repetition of an image of a diffraction grating [12–14]. The
Talbot distance is the distance between the source and the first regular image. Talbot imaging presents intermediate images appearing at smaller distances than the Talbot distance. These sub-images are characterized by a smaller feature size and pattern hybridization. The Talbot distance ($Z_T$) can be approximated using the following equation provided that light propagates in air \[14\]:

$$Z_T = \frac{\lambda}{1 - \sqrt{1 - (\lambda/a)^2}}$$

where $\lambda$ is the incident wavelength and $a$ is the lateral grating period ($a = S_{cc} \sqrt{3}/2$, with $S_{cc}$ the lattice period of the mask). For the experimental set-up and the three different masks used in this study, values $Z_T$ are as follows: for mask M1, $Z_T = 3.76$ $\mu$m; for mask M2, $Z_T = 1.37$ $\mu$m; and for M3, $Z_T = 0.7$ $\mu$m. These large values lead to almost constant patterns when the distance $Z$ between the mask and photoresist slowly varies, as shown in figure 6. Nevertheless, in the present case, the silicon substrate dramatically modifies the lateral pattern behavior. Indeed, the Cr-on-quartz mask and the silicon-photoresist support act as a Fabry–Perot’ cavity allowing for fast lateral pattern variation when $Z$ varies. Since the silicon acts as a mirror, we can reasonably suppose that the new Talbot distance (squeezed Talbot distance) $Z'_T$ will be given by $Z'_T = Z_T/(2n + 1)$, where $n$ is an integer. Indeed, with $h = Z'_T$, the Talbot distance $Z_T$ is retrieved with $Z_T = (2n + 1)h$, i.e. when light has made $2n + 1$ trips between the source (the mask) and the mirror (the silicon substrate). An odd number of trips is required to get the final image of the source on the mirror. For each mask, the values of $Z'_T$ are shown in table 1. In addition, the Fabry–Perot’ cavity implies that the distance $h_{FP}$ for which the field can resonantly propagate between both plates is given by $h_{FP} = m\lambda/2$ ($m$ is an integer different from zero). Those values can be compared with the theoretical squeezed Talbot distance $Z'_T$ as shown in table 1. Then, for each mask, we can compare the numerically computed squeezed Talbot distance with the relevant values of $Z'_T$ (see table 1).

For masks M1 and M2 this approach matches quite well. For mask M3 we are closer to the true Talbot conditions and we lose the capability to get complex subwavelength patterns. Typical air-gap widths are between 350 and 700 $\text{nm}$, i.e. $Z_T$ and $Z_T/2$, and the mask pattern is retrieved all along those distances. Pattern deformation during photolithography induced by the presence of mask–substrate gaps is a known phenomenon; several studies have analyzed this effect and solutions to compensate the distortions have been proposed \[25\]. The major difference with respect to data presented in this work is that the air gaps utilized in previous reports as well as characteristic feature dimensions were far larger than the exposure wavelength. The documented distortions were attributed...
Figure 7. Left: SEM images depicting the obtained morphologies using a Cr-on-quartz mask with $s_c = 480$ nm and $\varnothing = 380$ nm. Neither pattern mixing nor inversion were observed for higher $h$ values. Right: simulated electromagnetic field intensity at the surface of the photoresist layer for different $h$ values.

Table 1. Comparison between theoretical squeezed Talbot distances $Z'_T$, Fabry–Perot’ wavelengths $h_{FP}$ and numerical Talbot distances $h$ in the range $h = 200–800$ nm.

| $Z'_T$ (nm) | $h_{FP}$ (nm) | $h$ (nm) |
|------------|---------------|-----------|
| M1         |               |           |
| 751        | 730           | 750       |
| 536        | 548           | 550       |
| 417        | —             | —         |
| 341        | 365           | 350       |
| 289        | —             | —         |
| 250        | —             | —         |
| 221        | —             | —         |
| 198        | —             | —         |
| 179        | 183           | 200       |
| M2         |               |           |
| 456        | 548           | Undefined |
| 274        | 365           | 350       |
| 196        | 183           | 200       |
| M3         |               |           |
| 700        | 730           | Undefined |
| 233        | 183           | Undefined |

to the divergence of the exposure source (in the combined optical system made of light source/mask/photoresist plane). Moreover, in our case, the characteristic dimensions are of the order of the wavelength and the source divergence is expected to have a minimal influence on the results. Finally, note that, even though the real part of the Cr permittivity is negative here, surface plasmons cannot be excited. Indeed, since the incident light is at normal incidence, any surface plasmon excitation is due to diffraction orders which become tangent to the metallic surface. In the present case, for the hexagonal lattice, no orders can provide a resonant excitation for wavelengths in the very close ‘vicinity’ of 365 nm, whatever the lattice parameter. As a consequence, surface plasmons play no role in the present study.

4. Conclusions

We have introduced continuous colloidal pattern replication using contact photolithography thanks to a Talbot-like effect. Cr-on-quartz masks have been fabricated using nanosphere lithography and used as photolithographic stamps. Dimensions of the pattern are experimentally controllable (the initial size of the colloids, the etching procedure, etc). Hexagonal pattern arrangements with different dimensions have been considered. Depending on the thickness of the air gap between the mask and the photoresist layer, various changes occur in the pattern replica. The present approach then allows complex bidimensional patterns to obtained at a subwavelength scale. For instance, not only dots can be obtained (high fidelity replica of the mask), but also subwavelength anti-dots or rings.
As a consequence, this patterning approach benefits from the ‘infinite’ use of the mask while allowing multiple patterns with a single mask. Numerical simulations and a simple model allow us to predict the obtained patterns against the air-gap thickness. Further improvements could be considered with a piezoelectric actuator allowing for accurate control of the air gap and, then, of the obtained patterns. This could also allow 3D layer-by-layer printing.

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