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Print-to-Pattern Dry Film Photoresist Lithography

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Abstract

Here we present facile microfabrication processes, referred to as Print-to-Pattern dry film photoresist (DFP) lithography, that utilize the combined advantages of wax printing and DFP to produce micropatterned substrates with high resolution over a large surface area in a non-cleanroom setting. The Print-to-Pattern methods can be performed in an out-of-cleanroom environment making microfabrication much more accessible to minimally equipped laboratories. Two different approaches employing either wax photomasks or wax etchmasks from a solid ink desktop printer have been demonstrated that allow the DFP to be processed in a negative tone or positive tone fashion, respectively, with resolutions of 100 μm . The effect of wax melting on resolution and as a bonding material was also characterized. In addition, solid ink printers have the capacity to pattern large areas with high resolution which was demonstrated by stacking DFP layers in a 50 mm $\times$ 50 mm woven pattern with 1 mm features. By using an office printer to generate the masking patterns, the mask designs can be easily altered in a graphic user interface to enable rapid prototyping.

Keywords

Dry film photoresist; lithography; solid ink printing; DIY; rapid prototyping

1. Introduction

Scaling processes down to devices with features on the order of millimeter and micron scales can yield several advantages, such as increased precision, accuracy, and smaller reagent volumes [1]. To produce features on this scale, photolithographic methods have dominated industrial production. However, standard photolithography relies on an expensive exposure system with high-intensity collimated UV light source, a complicated dispensing method (e.g., spin or spray coating), and environmentally unfriendly photoresists and solvents. In addition, all of the aforementioned equipment must be maintained within a costly cleanroom environment to prevent damage to features from environmental particulates. Therefore, a significant technical barrier exists for non-expert end users to develop and explore new applications of microdevices.

Several groups, including ours, are currently exploring the use of off-the-shelf consumer electronics and intriguing low-cost materials to produce micro- and nanoscopic features with the objective of enabling complete out-of-cleanroom microfabrication capacity [2]. One of the emerging and most promising technologies for micropatterning is to utilize the state-of-the-art consumer office printer, which claims a maximum feature resolution up to 6 μm at 2400 DPI. To further increase the Ink jet printers have been used to pattern heat shrinkable polyolefin sheets that when shrank produce a reduced-scaled microstructures from the printed patterns that serve as a soft lithographic mold [3]. They also can be fitted with cartridges whose ink is replaced with solutions of cell adhesive compounds that when printed on substrates can selectively pattern cells upon seeding [4]. A more recent printer technology to enter the market is the solid-ink printer that deposit solid, water impermeable wax onto the printing substrate and has been taken advantage for a number of applications [5, 6]. When wax is patterned on solid substrates, the resulting microfeatures can be used as a mold [5, 7] or an etch mask [8, 9], and can even function as localized adhesive activated upon thermal treatment [9, 10]. Moreover, wax printers also have appreciated widespread use for the facile generation of paper-based microfluidic devices [6, 11-13]. While these printers are useful for generating micropatterns, the resulting channel heights of molds are shallow (typically $< 20 \mu\text{m}$ [5]) with circular profiles and difficult to control, particularly if taller channels are desired.

Solid-phase photoresist, also known as dry film photoresist (DFP), is a resin-based photosensitive material that offers an alternative to traditional photolithography processing. Initially employed in printed circuit board applications, DFP have been recently re-introduced as an excellent rapid-prototyping material for three-dimensional structures and microfluidics [14-16] as well as for biological micropatterning [17]. DFP is typically produced and packaged as sheets of pre-defined thicknesses ranging from 10 to 600 μm with removable plastic protective layers and can be laminated to glass, metals, plastics, or itself [18] which eliminates the need for spin coaters used in liquid photoresist dispensing. It is photopatternable (usually negative tone) with UV light, is only moderately sensitive to white light, and can be developed by an alkaline solution of sodium carbonate, a relatively safe chemical. The result is a flat, uniform patterned layer that can be bonded with other patterned dry film layers to create 3D structures. A limitation of DFP, as with all photoresists, is that a photomask is required to achieve the intended design features. While traditional chrome photomasks are typically used to pattern dry film, they are costly in time and money to produce and do not facilitate rapid prototyping process.

Here we present facile microfabrication processes, referred to as Print-to-Pattern dry film photoresist lithography, that utilize the combined advantages of wax printing and DFP to produce micropatterned substrates with high resolution over a large surface area in a non-cleanroom setting. Two different approaches employing either wax photomasks or wax etchmasks from a solid ink desktop printer, have been demonstrated that allow DFP to be processed in a negative tone or positive tone fashion, respectively. These methods can be performed in an out-of-cleanroom environment, which make DFP processing much more accessible, and can extend microfabrication capacities to the most basic laboratories. In addition, because patterns are deposited by an office printer, the mask designs can be easily altered in a graphic user interface to enable rapid prototyping.

2. Methods

2.1. Print-to-pattern process by wax photomask method (negative tone method)

This processing method, depicted in Fig. 1a, uses printed wax as a photomask. Dry-film photoresist (Alpho NIT215, Nichigo-Morton, Japan) of 15 μm thickness was taped to a piece of paper and a wax layer was patterned on one protective layer of the DFP by a solid ink printer (Phaser 8560, Xerox). The DFP was then detached from the paper and laminated onto a glass slide (Corning) after removing the unpatterned protective layer and subsequently soft baked at 90°C for 5 min. A 13 W compact fluorescent black light was used to expose the DFP through the printed mask for 6-10 s at a measured intensity of 5 mW/cm² at 365 nm. The patterned protective layer was successively removed and post-baked at 90°C for 5 min. In the following step, the exposed DFP was then developed in a solution containing a 1% Na₂CO₃ (Sigma-Aldrich), typically for 45 s. Optionally, a hard bake can be performed after development at 90°C for 5 min to further stabilize the DFP.

2.2. Print-to-pattern process by wax etchmask method (positive tone method)

Processing DFP using an etch mask approach shares a similar operational protocol as described in the wax photomask method with some alterations, shown in Fig. 1b. Prior to printing, one of the protective layers was removed and the wax patterns were deposited directly onto the DFP surface. In this processing scheme, the patterned DFP features are left standing on the remaining protective layer and are not laminated to another substrate unless otherwise indicated. The patterned wax was then briefly remelted at 100°C for < 1 min followed directly by etching the DFP in 1% Na₂CO₃ for 30 s on its remaining protective layer. The wax can then be subsequently removed by either an organic solvent or by tacking with an adhesive material, e.g. tape. Alternatively, the wax can be left on the surface, laminated onto another surface, and briefly remelted at 100°C to reversibly seal the two layers together. The capacity for the printed wax to attenuate UV light was determined by measuring the steady-state light intensity by a UV meter (OAI Model 308) before and after a DFP protective layer covered in printed wax was placed over the sensor.

2.3. The effect of wax remelting on resolution

Wax was printed in a comb pattern with lines of single wax dots at the resolution limit of the printer on either the protective layer of the DFP (the wax photomask method) or directly on the DFP itself (etchmask method). A subset of the patterned substrates were either directly etched in 1% Na₂CO₃ solution, or remelted at 105°C and then etched. Bright field images at 4 $\times$ were taken before and after each processing step.

2.4. Resolution testing

Using the photomask or etchmask method, circles and lines between 100 and 500 μm were patterned on DFP in a positive (pillars, ridges) or negative (wells, grooves) fashion and imaged with a stereomicroscope. Higher magnification images of 100 and 200 μm 2D features were processed to match the color of lower magnification images. A detailed analysis of the features was performed by measuring the printed feature width and the corresponding etched result from bright field micrographs. For features produced by the wax

etchmask method, the mask size was considered the top wax layer and the resulting features were measured from the DFP patterns imaged on back side. Shape error was determined by imaging several micropatterned rectangles with varying aspect ratios made by the wax photomask method. Using custom software, the feature width and height was measured and the relative difference was determined between the calculated area ($L \times W$) and the integrated area using image thresholding.

2.5. Bond strength testing

Using the wax etchmask method, a DFP layer with a circle printed wax 1 cm in diameter was laminated wax side up onto a 25 mm $\times$ 75 mm glass slide (Corning). A subset of test slides were backside exposed to fully crosslink the film. Two slides of either exposed or unexposed dry film were coupled perpendicularly with the wax circles in contact and were remelted at 100°C to bond the layers. The top slide was subsequently fixed to a crossbar held by a ring stand while a plastic container was hung from the bottom slide using twine. Water was added to the container until the wax bonding failed, after which the mass of the water was recorded. As a reference, polydimethylsiloxane (PDMS, Dow Sylgard 184) mixed at a 10:1 base:curing agent ratio was cured at 80°C for 3 h and then cut into 1 cm circles that were oxygen plasma-assisted bonded to glass slides (100 W, 5 min), which were subsequently bonded by oxygen plasma to another PDMS-glass construct and tested the same way as the dry film/wax bonding pairs.

3. Results and Discussion

3.1. The desktop dry film processing method

Conceptually, the print-to-pattern DFP lithographic processing method provides a facile, out-of-cleanroom lithographic method for the creation of sub-millimeter structures in DFP using an off-the-shelf solid ink printer. DFP is an aqueous-based photosensitive semi-solid resin (negative tone photoresist) that is commonly used in the microelectronics industry to pattern printed circuit boards. Prior to exposure, it is highly adhesive to hydrophilic surfaces and can be laminated in a multiple layer configuration. Its easy handling and extensive compatibility with high throughput machinery increases its usefulness not only in this field, but also with use in the laboratory as a masking or molding material, e.g. for patterning and soft lithography, respectively [14, 16, 17]. Our processing methods extend the processability of this material by using a commercially available solid ink printer as a pattern generating device and further eliminates the bottle-neck of mask production in traditional photolithography. There are two proposed methods in Figure 1 using print-to-pattern DFP lithographic processing: 1) a wax etchmask method (top) where wax is deposited directly onto the DFP and the result can be realized by directly etching the printed product; and 2) a wax photomask method (bottom) where wax is printed directly onto DFP protective layers and processed similarly to a typical photolithographic scheme. Both schemes possess their own unique advantages and can be tailored to overall design requirements and are cross-compatible with one another to achieve the final desired result.

The wax photomask method allows processing very akin to traditional photolithography to take place. In this method, the DFP is patterned and exposed like a negative photoresist.

However, some simplifications can be made to the process to remove the need for technical expertise, expensive equipment, and hazardous materials. Because the resolution limit of the printer is on the order of 100 μm , reasonable results can be achieved without the use of a collimated light source. All photoexposures of DFP in this approach were performed by a \$5 compact fluorescent black light purchased at a local store. These lights have a strong 365 nm UVA component of 5 mW/cm^2 at a distance of 10 cm. While white fluorescent light can be used to expose the type of DFP used here, the intensity of active wavelengths of room light were not strong enough to significantly impact the resolution of the resulting features for the short processing times. Therefore a gold room is no longer required, but could be inexpensive enough to be realized by inexpensive polymeric lamp sleeve filters if long processing times are required. Importantly, the type of DFP used here can be etched by a non-toxic sodium carbonate solution that is safe to handle using minimal personal protective equipment.

In contrast to the wax printmask, the wax etchmask method patterns wax directly onto the DFP. The masked dry film can then be directly etched, allowing for positive imaging of dry film. Subsequently, the result can be backside exposed to fully crosslink the material to increase durability. The remaining wax can be removed by either a sticky surface (e.g. tape), wiped off with an organic solvent (ethanol or similar alkyl alcohol), or left to remain on the surface which can act as a gluing layer between other layers of dry film or another substrate that is capable of being wetted by the wax. The etchmask method also provides the quickest result, as the exposure and prebake steps are not used. Optionally, the product can be left as a free standing structure on its protective layer or laminated onto a substrate; unexposed film can be laminated onto hydrophilic surfaces while exposed film can adhere well to another unexposed dry film layer.

3.2. Printed wax features and remelting

Solid ink printers apply single spots of wax at their maximum resolution that come together to form the final image on the printed substrate. Unlike liquid ink from inkjet printers that wet and absorb into the substrates they print on, wax deposited by solid ink printers retains a rounded droplet shape as it rapidly cools below its glass transition temperature ($\sim 100^\circ\text{C}$) upon deposition and largely remains on the surface rather than absorption into the substrate. While macroscopically these imperfections are not discernible, they can have a negative impact when features near their length scale are desired. In our Print-to-Pattern processing methods, the resulting pin holes between the applied wax dots could result in bleed-through exposure, rough edges, and pitted surfaces. Figure 2a shows a one-dot wide wax comb pattern that is deposited directly onto the surface of dry film as would be the case in the wax etchmask method. The pinholes are very apparent in the micrograph, and when etched, result in breaks in thin features, holes throughout larger areas, and rough edges (Fig. 2b). However, if the wax pattern is heated above its glass transition temperature and melted, the pinholes become filled in (Fig. 2c) due to surface tension and restores the intended pattern (Fig. 2d) after etching the DFP. Only brief melting of the wax is necessary (<5 s) to achieve the desired result which occurs immediately after the wax appears shiny and wet. Prolonged melting results in the loss of small features due to surface tension effects.

When wax is printed on the protective layer of the DFP, as would be the case in the wax photomask method, pinholes were not observed (Fig. 2e). Continued processing results in clean patterns as intended (Fig. 2f). Melting of the wax was found to cause the edges to diffuse (Fig. 2g) and results in UV intensity gradient upon exposure that yields rounded edges (Fig. 2h) in the DFP after processing. Therefore, melting of the wax degrades the resolution of the technique and becomes unnecessary for the wax photomask method. We found that a single layer of printed wax was able to block 95.7 ± 0.4 % of light from the UV source used in this study.

3.3. Resolution testing

To investigate the resolution limits of the Print-to-Pattern processing methods, arrays of 1-D and 2-D features of varying sizes were created in single layer of DFP using both the wax etchmask (Fig. 3a) and wax photomask methods (Fig. 3b). Figure 3 shows patterned wells and pillars or ridges and grooves with dimensions ranging between 100 and 500 μm in section (i). High resolution images of the 100 and 200 μm 2D features are provided in section (ii). To determine the fidelity of the features to their respective masks, both the dimensions of the masking features and the resulting DFP features were measured. In the case of the wax etchmask approach, the top wax layer was taken as the masking layer and the DFP feature was measured on the backside. The resulting dry film feature dimensions were measured and plotted against the mask feature size for both methods in all patterns, shown in Fig. 4ab. As expected, all measured data points showed a strong correlation between the patterns generated and the mask features, demonstrating high fidelity image transfer from the mask to the photoresist. Linear regressions of the data showed slopes indicated a less than 5% deviation from unity.

Like all etching processes, controlling the etching conditions and duration are critical to preventing significant undercut of the DFP when employing the wax etchmask method. For the DFP thickness and conditions used in this study, an estimated etch rate of 0.33 $\mu\text{m/s}$ was achieved. Temperature and concentration of the etchant can be adjusted as needed to achieve optimal etch rates for a particular DFP, resist thickness, and the aspect ratios and sizes of the desired features.

Another benefit to using the Print-to-Pattern processing techniques is that by printing directly on the cover layer of the DFP or the resist itself, the distance between the masking layer and the photosensitive material is minimized, increasing the resolution limit of the system which is determined, among other variables, by the thickness of the protective layer (16 μm), and the resolution of the printer (600 DPI $\approx 23 \mu\text{m}$). For a proximity exposure using 365 nm light and a 15 μm -thick photoresist, the expected minimum resolution is $\sim 9 \mu\text{m}$, which is below that of the manufacturer's declared minimum resolution for the photoresist used in this work (15 μm). It is also well below the theoretical resolution of the printer, indicating that the resolution limit of this processing method is that of the printer. Our data support this conclusion due to the near unity of the regressed slopes in Fig. 4ab, and Fig. 3 where the imperfections on the border of printed features are very characteristic of wax dots.

To determine the impact of the finite size of the wax droplets on the formation of features, rectangles of various aspect ratios were patterned into DFP using the wax photomask method and the resulting length and width were subsequently measured along with the integrated internal area from micrographs using custom software. The shape error was calculated as the percent error of the measured dimensional area ($L \times W$) to the integrated area and plotted against the nominal feature size (square root of the integrated area) shown in Fig. 4c. A decreasing logarithmic trend was observed, indicating that smaller features are more significantly affected by the resolution of the wax droplets than larger features as would be expected. Practical application of this knowledge that features that require relatively tight tolerances ($<10\%$) are behooved to have a minimum feature size of $750\text{ }\mu\text{m}$. This is consistent with the wax droplets of being $\sim 70\text{ }\mu\text{m}$ in diameter. However, the values reported in Fig 3c are specific to the printer used in this study and are expected to vary with other models.

3.4. Bond strength testing

One of the convenient features of DFP is that laminated unexposed layers typically yield a strong and irreversible substrate adhesion for clean, hydrophilic surfaces, including to itself. However, exposed DFP shows little to no adhesive capacity. We were unable to test the bonding strength between DFP pairs as delamination from their substrates occurred before the bond between DFP layers failed. To provide an alternative bonding approach, we investigated the use of the printed wax as a reversible bonding agent between DFP layers. By using the wax etchmask method, the remaining wax layer patterned on the surface of the DFP can be melted to bond it any other surface that the wax layer is capable of wetting. Bond strength tests were performed between layers of either exposed or unexposed dry film layers. Figure 5 shows that bond strengths of more than 300 kPa are easily achieved (313 ± 57 and 348 ± 67 kPa for unexposed and exposed dry film samples, respectively). For reference, the bond strength of oxygen plasma-bonded PDMS, a commonly used polymer and bonding method in MEMS fabrication, was measured and found to be very comparable (380 ± 17 kPa) to that of the wax bonding technique. Wax bonding is therefore suitable for a variety of devices that are presently constructed through PDMS plasma bonding. As an added advantage over PDMS bonding, wax bonding is reversible by simply peeling the layers apart by hand or through remelting of the wax. This property allows devices to be disassembled if the recovery of its contents is needed for further testing or analysis.

3.5. 3D Woven DFP pattern

Another benefit to using a solid wax printer to deposit the masking layer is that it can produce high resolution patterns over a large surface area ($8.5\text{ in} \times 11\text{ in}$). To demonstrate this capacity, a $50\text{ mm} \times 50\text{ mm}$ woven DFP pattern, shown in Fig. 5, was created using two layers produced by the wax photomask method. Individual features were 1 mm in width and the layers were laminated using manual alignment. Higher resolution multilayer devices could be fabricated with the aid of patterned fiduciary markers and a stereomicroscope for alignment.

3.6. Comparison with existing DIY technologies

The Print-to-Pattern method is a versatile technique that possesses several advantages. Table 1 lists similar out-of-cleanroom technologies that provide DIY patterning methods. Print-to-Pattern lithography fills a similar niche as the direct projection on dry-film photoresist (DP²) [14] and shrink-film fabrication methods [3] in that it is a rapid, low-cost, out-of-cleanroom technique that is capable of sub-millimeter resolution. However, the Print-to-Pattern technique has the advantages of larger working areas and eliminates extra design considerations such as shrinking ratios encountered in DP² and shrink-film fabrication, respectively. In comparison with 3D printing and laser micromachining methods [19], Print-to-Pattern is significantly less expensive and can also generate a product faster due to the parallel patterning method of desktop printers with nearly the same resolution. However, if micrometer-scale resolution is required, techniques such as stereomask lithography [17] and plasma lithography [20] provide excellent alternatives at the expense of higher equipment costs.

3.7. Future work and developments of the Print-to-Pattern technique

The goal of the Print-to-Pattern technique is to utilize existing commercially available technology to achieve out-of-cleanroom microfabrication for classrooms and laboratories with limited resources. Therefore resolution limit of the Print-to-Pattern technique relies on the capabilities of currently available commercial printing technology. As this technology develops, the improvements in precision and throughput will be extended to the Print-to-Pattern technique. The present state-of-the-art in printed material deposition can be exemplified by the Fujifilm Dimatix™ which boasts a droplet resolution of 20 μm . While acquisition of this technology would currently be forbiddingly expensive to most classrooms or under-equipped laboratories, the prices of these printers will drop as the technology matures and will set the future resolution limit of the Print-to-Pattern technique. Other factors that affect the resolution of the Print-to-Patten methods, such as surface wettability and contact angle of the wax droplets, should be the subject of future investigations.

4. Conclusion

Here we demonstrated two 3D, out-of-cleanroom DFP fabrication schemes using basic laboratory equipment and a solid ink printer. An image can be designed and printed directly onto the DFP protective layers and subsequently exposed and processed to fabricate layers (wax photomask), or wax can be directly printed onto dry film to serve as an etchmask (wax etchmask). The wax photomask and etchmask methods allow for the rapid prototyping of devices and can achieve minimum, printer-limited resolutions of 100 μm . The printed wax layer was found to have the capacity to reliably bond two dry film layers together with the approximate bonding strength of plasma bonded PDMS. In addition, solid ink printers have the capacity to pattern large areas with high resolution which was demonstrated by stacking DFP layers in a 50 mm $\times$ 50 mm woven pattern with 1 mm features. These techniques can allow relatively unequipped labs and classrooms to perform microfabrication without the necessity for expensive equipment or extensive expertise. This accessibility extends the opportunity for others to construct and utilize microscale devices for themselves.

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(a) Wax Etchmask Method

(b) Wax Photomask Method

Figure 1.

The Print-to-Pattern dry film photoresist lithography fabrication process for (a) the wax etchmask (positive tone) and (b) the wax printmask (negative tone) methods. The illustration on the right depicts if the printed wax (black letter A) is deposited on the photoresist itself (a) or on the protective layer (b).

Figure 2.

Effect of remelting printed solid ink on resulting features using (a-d) the wax etchmask and (e-h) the wax printmask (bottom) techniques. Remelting the printed wax eliminates pinholes in the wax etchmask method but blurs edges in the wax printmask method. Scalebar = 200 μm

Figure 3.

Resolution tests of (a) the wax etchmask and (b) the wax printmask methods of negative and positive features with features ranging from 100 to 500 μm in section (i). Lower pairs of images in section (ii) are high magnification images of 100 and 200 μm features. (i) scale bars = 1 mm; (ii) scale bars = 100 μm .

Figure 4.

Analysis of resolution. Resulting feature sizes are shown after development vs mask feature size using (a) the wax etchmask and (b) the wax printmask methods. A high degree of correlation indicated that the resolution was limited by the printer. Diagonal correlation lines are added for reference. Data points given as mean $\pm$ SD. (c) Calculated shape errors of printed rectangles vs nominal feature size. Digital deposition of ink droplets affects the fidelity of the designed feature as its dimensions decrease.

Figure 5.

Dry film photoresist bonding characterization. Measured bonding strengths between unexposed (UDFP) and exposed (EDFP) layers of dry film photoresist bonded together by remelted printed wax. As a reference, the bonding strength of oxygen plasma-bonded PDMS layers were measured by the same method. Bond strengths between laminated dry film photoresist layers was greater than the adhesion between dry film photoresist and glass substrates and could not be tested. Data given as mean $\pm$ SD.

Figure 6.

A 3D dry film photoresist weave pattern over a 50 mm $\times$ 50 mm area. Top and bottom layers, whose design is shown on the left, were patterned using the wax photomask method and laminated together on a 2 in $\times$ 3 in glass slide to form the final structure.

Table 1

A comparison of DIY out-of-cleanroom technologies.

Technique	Construction Area	Resolution	Fabrication Time	Initial cost	References
Plasma lithography	100 cm ²	0.1 μm	minutes	Moderate	[20]
Direct projection on dry-film photoresist	5 cm ²	10 μm	minutes	Low	[14]
Stereomask lithography	10 cm ²	10 μm	minutes	Moderate	[17]
Laser engraving	2000 cm ²	100 μm	hours	High	[19]
Shrink-film fabrication	300 cm ²	100 μm	minutes	Very low	[3]
3D printing	1000 cm ²	100 μm	hours	High	--
Print-to-Pattern lithography	600 cm ²	100 μm	minutes	Low	This work