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Sub-10-nm Wide Trench, Line, and Hole Fabrication Using Pressed Self-Perfection

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ABSTRACT

We report a new approach to adjust and improve nanostructures after their initial fabrication, which can reduce the trench width and hole diameter to sub-10 nm, while smoothing edge roughness and perfecting pattern shapes. In this method, termed pressed self-perfection by liquefaction (P-SPEL), a flat guiding plate is pressed on top of the structures (which are soften or molten transiently) on a substrate to reduce their height and guide the flow of the materials into the desired geometry before hardening. P-SPEL results in smaller spacing between two structures or smaller holes in a thin film.

As conventional nanofabrication techniques are approaching their limits (because of their working principles or intrinsic noise), the fabricated nanostructures will be determined primarily by the limitations, and the improvement of these nanostructures by improving their fabrication processes becomes increasingly less effective. A path-changing approach is to improve a nanostructure after its fabrication. Recently, we have proposed and demonstrated one such approach, termed self-perfection by liquefaction (SPEL), which improves nanostructures after their original fabrication by selectively melting the nanostructures on a substrate for a short period of time while applying a set of boundary conditions to guide the flow of the molten materials into a desired geometry before solidification.[1] It has been demonstrated that SPEL can significantly smooth line-edge roughness (LER), and by placing a flat plate above the structure, capped or guided SPEL can increase the sidewall slope, flatten the top surface, maintain or narrow the width. SPEL works for soft materials as well as hard (high melting-temperature) materials such as Si and Cr.

Here, we propose and demonstrate a new approach to SPEL, termed pressed SPEL (P-SPEL), in which a guiding plate with a smooth surface is pressed on the top of the structures on a substrate (Figure 1). If the structures consist of two lines (or mesas) separated by a spacing or a thin film with a hole, the spacing and the hole diameter will be reduced by P-SPEL. Different from previous SPEL methods (capped or guided), where the resultant structure height was the same as or taller than the original height, P-SPEL reduces the height of the structures (when they are molten or softened) and forces materials to flow laterally between the guiding plate and the substrate, expanding their lateral size (to keep

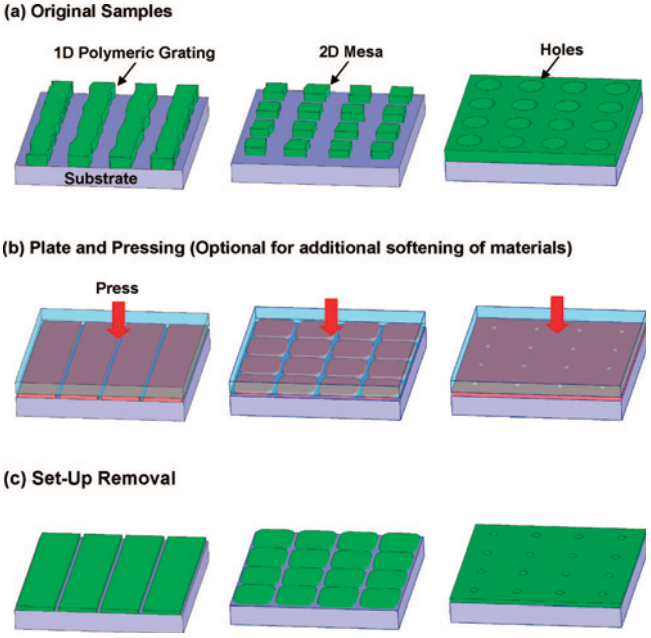

Figure 1. Schematic of pressed self-perfection by liquefaction (P-SPEL) process. P-SPEL improves nanostructures on a substrate after their original fabrication, by selectively melting (softening) the nanostructures while pressing a flat plate on top of the structures to guide the flow of the molten (softened) materials into a desired geometry before solidification. P-SPEL will reduce the height of the nanostructures while expanding their lateral size, hence reducing the spacing between the two adjacent structures or the diameter of a hole in a thin film, plus smoothing edge roughness and perfecting the shapes.

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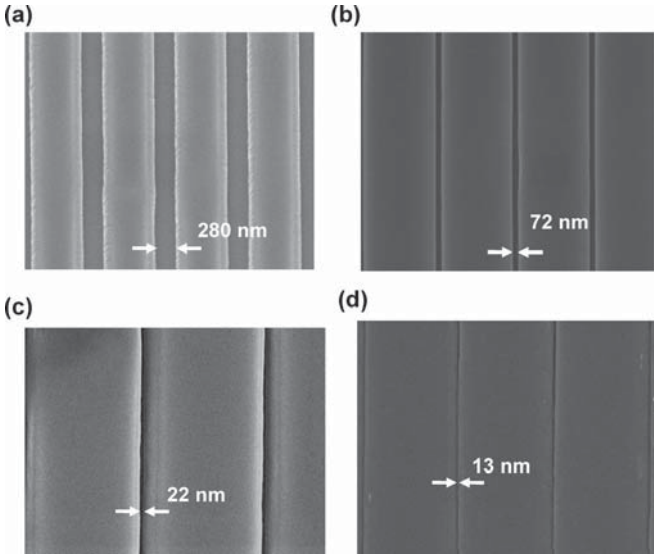

Figure 2. SEM of 1 μm period polymer grating on a Si substrate before and after P-SPEL, where the spacing between 2 grating lines is reduced from 280 nm to an average 13 nm. (a) The original resist grating with a 280 nm spacing before P-SPEL, fabricated by nanoimprint using a grating mold; (b) after P-SPEL for 8 min, the spacing became 72 nm; (c) after P-SPEL for 20 min, the spacing became 22 nm; and (d) After P-SPEL for 40 min, the spacing became an average 13 nm less than 1/20th of original width. The pressure is 150 psi, and the temperature is 70 °C.

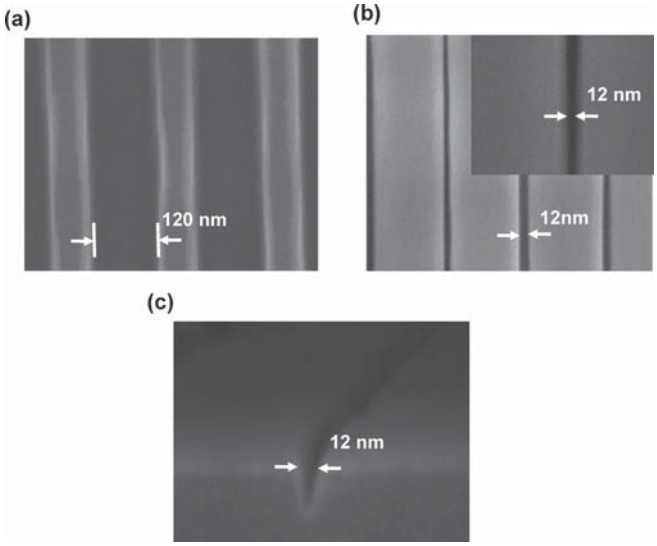

Figure 3. SEM of 200 nm period polymer grating on a Si substrate before and after P-SPEL, where the spacing between 2 grating lines is reduced from 100 to 12 nm. (a) The original imprinted gratings with a spacing of 120 nm; (b) after 20 min P-SPEL process, the spacing shrank to 12 nm (an average 16 nm); inset: the zoom-in image of spacing; and (c) a cross section of the spacing; the foot of the grating seems in touch. The pressure is 150 psi, and the temperature is 65 °C.

the volume constant). The lateral size expansion reduces the spacing between two adjacent structures (the trench width) and reduces the diameter of the holes in a thin film. The pressing combined with the effects of surface tension can smooth out edge roughness and perfect a round shape in the case of holes. Depending upon the material type, isothermal heating (for polymers) or selective pulsed laser heating (for

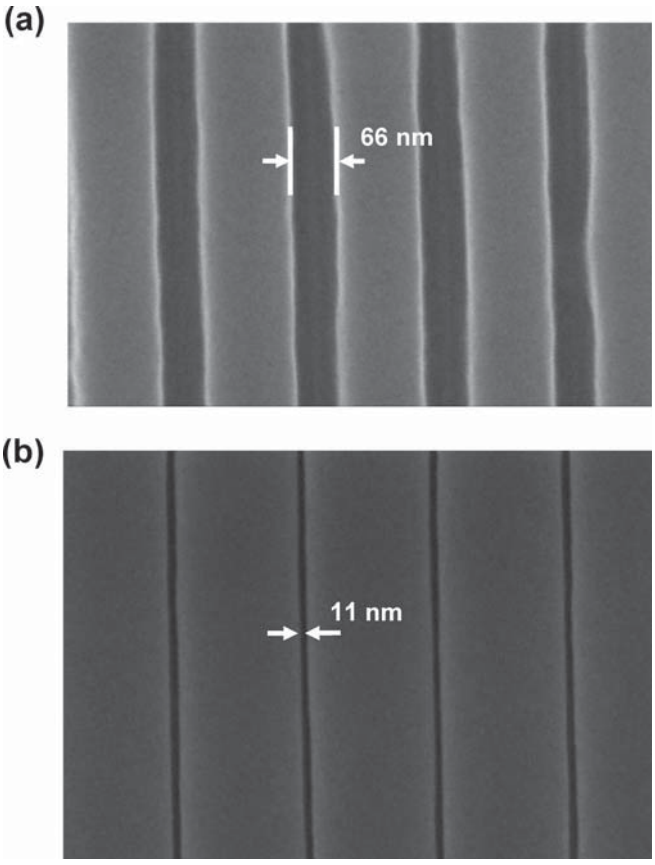

Figure 4. SEM of another 200 nm period polymer grating on a Si substrate before (a) and after (b) P-SPEL, where the spacing between 2 grating lines is reduced from 60 nm to an average 11 nm. The final spacing is rather uniform. The pressure is 150 psi; the temperature is 100 °C, and the time is 0.5 min.

hard high-melting temperature materials such as Si or Cr)[2] can be used in the softening step. Because P-SPEL is based on a different physical principle, it can overcome the limitations of electron beam lithography, photolithography, and other conventional patterning approaches to produce smaller trenches, lines, and holes.[3–6] Furthermore, P-SPEL works well for very dense patterns and is scalable to large-area wafers, hence offering unique nanofabrication capabilities.

We have demonstrated P-SPEL in thermoplastic polymer nanostructures on Si or SiO_2 substrates. The initial nanostructures were fabricated by thermal nanoimprinting for patterning and reactive ion etching (RIE) for removing the residual resist layer. We used Si wafer with a featureless smooth surface as the guiding plate, which was chemically treated with Nanonex NXT-100 surfactant to avoid sticking to the polymers when removing the plate. Either an Air Cushion Press™ (ACP)[7] in Nanonex NX-2000 imprinter or a parallel plate press (PPP) is used to apply pressure to the sample. The final gap between the guiding plate and the substrate, and thus the final spacing between the two adjacent structures or the final hole diameter in a thin film, were controlled by pressing pressure and processing time.

Figure 2 shows the results of P-SPEL for 1 μm pitch grating lines (line/spacing of ~720/280 nm) made of a linear acrylate polymer (thermoplastic with a glass transition

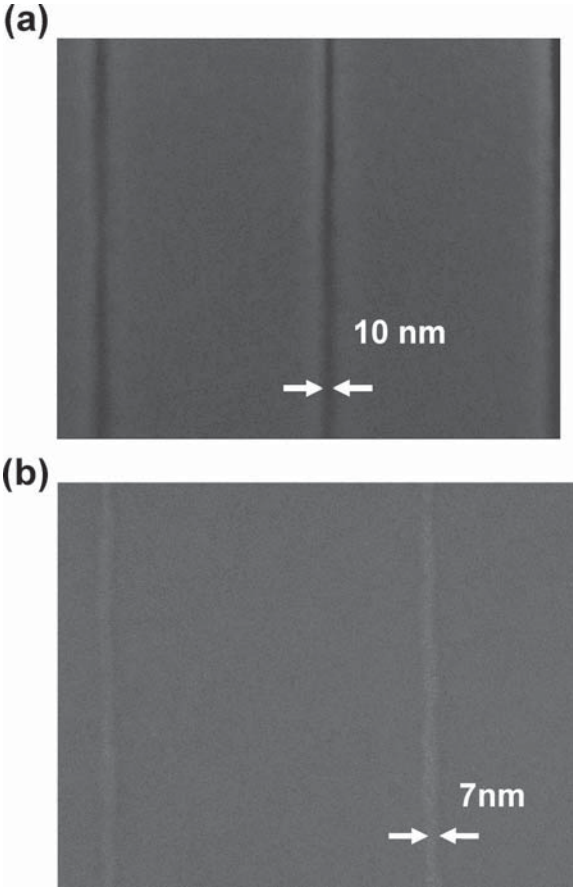

Figure 5. SEM of the patterns transfer using the resist profile obtained by P-SPEL in Figure 3. (a) 10 nm wide trench (200 nm period) etched into a SiO_2 substrate (the resist as a mask and RIE etching), and (b) 7 nm wide Cr lines by a lift-off.

temperate of 55 °C; we call it type-I polymer) at a processing temperature of 70 °C and pressure of 150 psi (by PPP) with various processing times. At 70 °C, the thermoplastic has a high viscosity leading to a slow but stable viscoelastic polymer flow. Initially, the spacing between two grating lines (1 μm period and 225 nm height) was ~280 nm. By using P-SPEL, the spacing was reduced to ~72 nm, 22 nm, and 13 nm, respectively for 8, 20, and 40 min processing times. The initial spacing was reduced as much as a factor of 2,100%. Furthermore, our analysis using the fractal method[8] shows that, as in other SPEL processes, the 3σ LER is also significantly reduced from 24.2 to 12 nm (see discussion below).

Similarly, an initial 120 nm spacing in a 200 nm period grating (80 nm height) of the same type-I polymer was reduced to as small as 12 nm after P-SPEL at 65 °C under 150 psi pressure (PPP) for 20 min (Figure 3a,b). From the cross-sectional scanning electron micrograph (SEM) (Figure 3c), it was observed that a typical line spacing exhibits a notch shape, and its depth is ~30 nm, less than the original line height, indicating that, as the spacing size is reduced to ~12 nm, two adjacent lines have been merged partially at their base.

We have tested P-SPEL in different thermoplastic materials and found that a different material might change the flow time but not the ability of P-SPEL to reduce the spacing

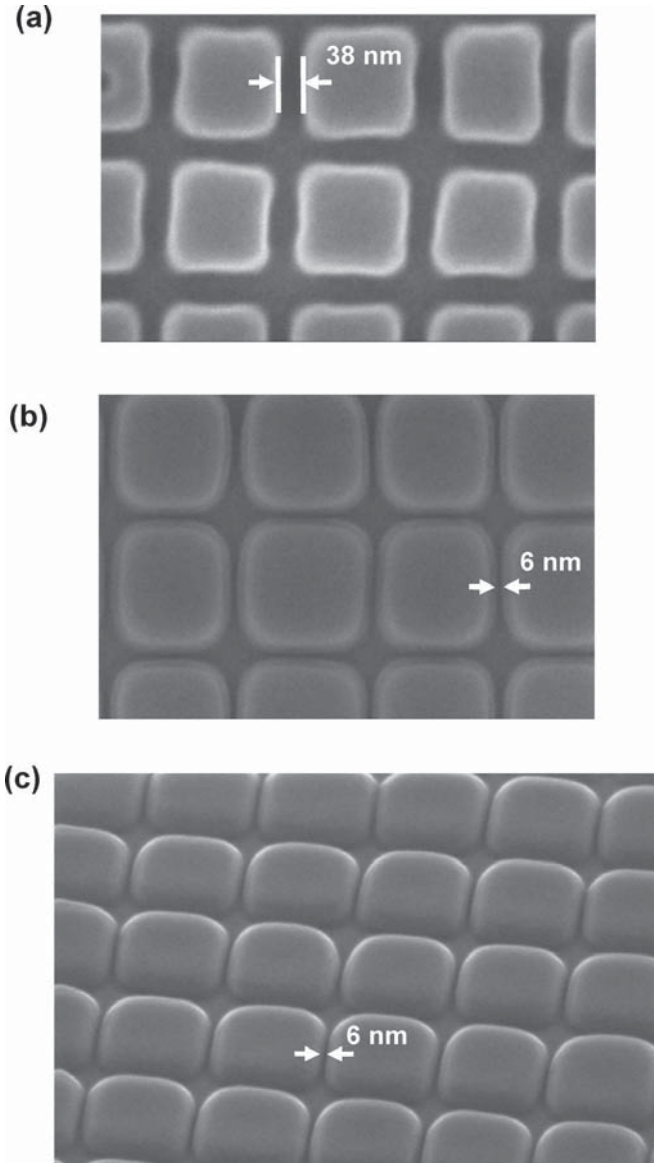

Figure 6. SEM of 200 nm period polymer 2D square tile array (55 nm height) on a Si substrate before (a) and after (b, c) P-SPEL, where the spacing between 2 tiles is reduced from 38 to 6 nm. P-SPEL used 230 psi pressing pressure and 90 °C temperature for 1 min.

between two adjacent structures. Figure 4 shows P-SPEL results in a polymer grating (type-II: acrylic thermoplastic with glass transition temperature about 45 °C). The original 66 nm spacing (line width 134 nm) of the grating with 200 nm pitch and 80 nm height became an average 11 nm spacing (189 nm line width) after a P-SPEL process at 150 psi (by ACP) and 100 °C temperature for 0.5 min. Again, the LER of grating lines is noticeably reduced (from 13.4 to 6.8 nm).

The patterns created in polymer by P-SPEL have been transferred into either nanotrenches in a substrate or nanometal lines on a substrate. The first step in the resist pattern transfer was to shadow a thin layer of Cr on top of the resist, followed by oxygen-based RIE to remove the residual polymer between the two grating lines. For making trenches, a second CF_4/H_2-based RIE with the same etching mask as the first RIE was used to etch trenches in SiO_2. After etching, the resist (and Cr on top) was removed by solvent. The result

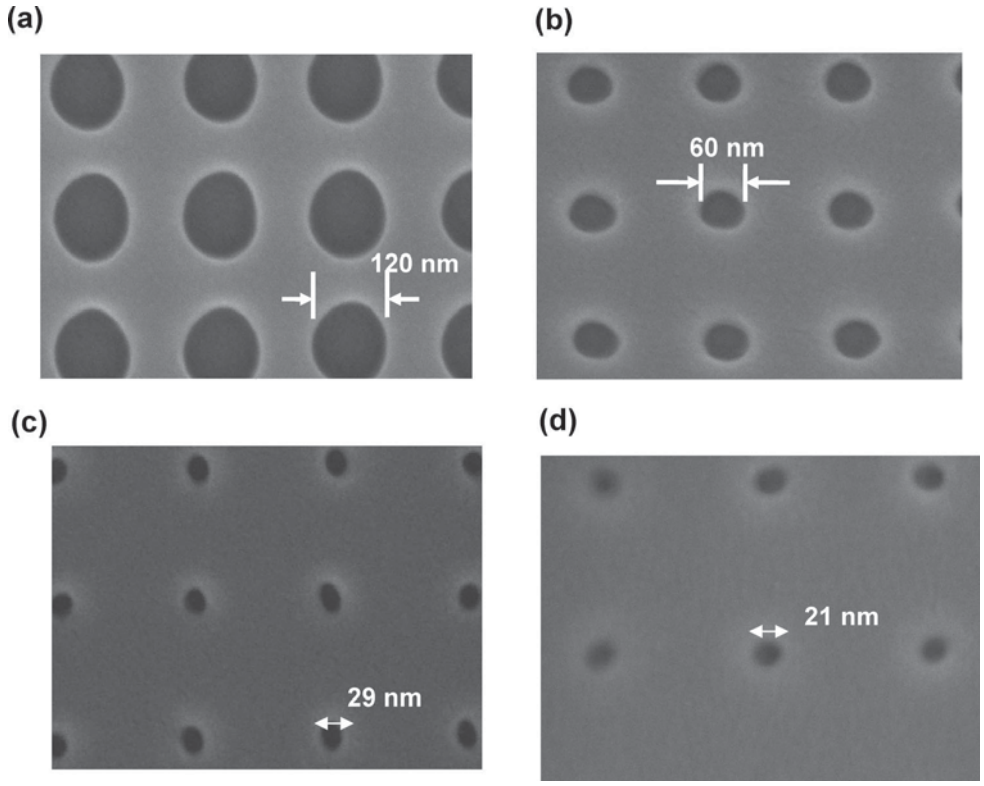

Figure 7. SEM of the holes in a polymer thin film (200 nm period) on a Si substrate before (a) and after (b) P-SPEL, where the diameter is reduced from 120 to 20 nm. (a) Original hole having less-round shape and 120 nm diameter; (b) after 2 min P-SPEL, the hole's shape becomes a near perfect round shape and the hole diameter is reduced to 50 nm; (c) after 5 min P-SPEL, the hole diameter shrank to 29 nm; and (d) after 10 min P-SPE, the hole diameter is reduced to 20 nm.

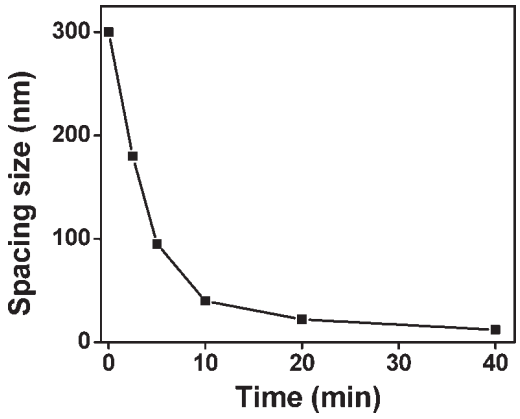

Figure 8. Final spacing size as a function of P-SPEL time (with fixed 150 psi pressure and 70 °C temperature) for 1 μm pitch P-SPEL grating of 300 nm initial spacing and 225 nm initial thickness (Figure 2). When the spacing becomes less than 20 nm, the spacing reduction rate slows drastically, making P-SPEL nearly self-limiting.

is 10 nm wide trenches in SiO_2 (Figure 5a). For making metal lines, a second Cr (8 nm thick) layer was evaporated after O_2 RIE but at an incident angle normal to the wafer, followed by a lift-off in a solvent, which removes the resist and the Cr on top of the resist, leaving 7 nm wide Cr lines on the substrate (Figure 5b). The Cr line can be used as a part of a device or another etching mask for additional pattern transfer.

We have applied P-SPEL to other types of nanostructures, including a two-dimensional (2D) square tile array. Using

Table 1. Average Size, Size Variation, and Edge Roughness from 10 Different Spacing or Holes on Each Sample

samples		av spacing (nm)	3σ for av spacing (nm)	3σ for av LER (nm)
1 μm grating (Figure 2)	before	277	20.1	24.1
	after	22	28.7	12.0
200 nm grating (Figure 3)	before	120	23.6	13.4
	after	16	10.8	6.8
200 nm grating (Figure 4)	before	61	14.3	6.6
	after	11	5.5	3.5
squares	before	32.3	18.3	
	after	7.9	6.0	
holes	before	120	12.3	
	after	21	12.0	

P-SPEL, we reduced the spacing of a 200 nm period polymer 2D square tile array (55 nm height) on a Si substrate from 38 nm to as small as 6 nm (Figure 6). The P-SPEL process used 230 psi pressing pressure (by ACP) and 90 °C temperature for 1 min. As shown, the tile profile after P-SPEL has a rather smooth and straight edge, with slightly rounded corners.

The third type of nanostructures to which we applied P-SPEL was holes in a thin polymer film. P-SPEL can significantly reduce the hole diameter while rounding the hole shape. As shown in Figure 7, the hole diameter in a polymer thin film (200 nm hole period and 90 nm film thickness) on a Si substrate was reduced from 120 to 50 nm, 39 nm, and 20 nm, respectively, after P-SPEL at 150 psi (by PPP) and 100 °C for 2, 5, and 10 min, respectively.

Interestingly, we observed that the final spacing size in a P-SPEL process is nearly self-limiting, when the spacing between two adjacent structures is reduced below 20 nm. For example, Figure 8 shows the final spacing versus the pressing time of P-SPEL for a 1 μm period grating with 300 nm initial spacing and 225 nm initial thickness. The pressing was carried out at 150 psi pressure (by PPP) and 70 °C temperature. It clearly shows that initially the spacing was reduced at a rate of ~40 nm/min, but when the spacing is reduced below 40 nm, the rate significantly slows down to 0.9 nm/min, allowing good control of the final spacing.

It should be pointed out that, unlike capped or guided SPEL, for which the structures need to transiently become a liquid with a relatively low viscosity (so that they can change the shapes on their own), in P-SPEL, structures are only required to expand laterally when pressed vertically. In fact, we have adjusted the processing temperature to make the polymer either in the viscoelastic state (i.e., a flow have both viscous and elastic characteristics) or in the nearly viscous state. We found that the viscoelastic flow allows results in a relatively sharper final corner than that in a viscous flow. The examples shown in the paper are in viscoelastic flow regime.

Furthermore, we studied the average spacing and average LER before and after P-SPEL by performing fractal analysis of high-resolution SEMs of different 10 spacing or holes for each sample, as listed in Table 1. Although locations of SEM before and after P-SPEL are not the same, the averaging of 10 spacing offers a good indication of the trend. In all experiments, 3σ LER has been reduced by at least a factor of 200% (defined as $3\sigma_{Initial}/3\sigma_{Final}$) after P-SPEL. We believe that the pressing may enhance LER smoothing, since material expansion due to pressing will be more on a concave surface than a convex surface, therefore reducing the topographic fluctuation at the spreading edge of polymer and thus LER. As to the 3σ variation of the spacing size between two structures, our data analysis shows that, in two experiments, the average 3σ variation in spacing size after P-SPEL has been reduced by 220−260%. But in one experiment (1 μm grating shown in Figure 2), the 3σ variation in spacing size was increased after P-SPEL by 140%, which we believe is due to the poor quality of SEM images for the narrow spacing after P-SPEL. We need to study these variations further.

Obviously, the control of the final distance between the substrate and the top guiding plate is crucial to P-SPEL applications. The pressing time based control is simple but less precise. A better approach is to add local microstoppers that will stop, once the limit is reached, any further reduction of the distance between the substrate and the guiding plate. The microstoppers also can improve the large-area wafer uniformity of P-SPEL. The control of the sidewall profile depends on the wetting properties of the substrate and the guiding plate, as well as the properties of the material during its flow (Newtonian or viscoelatic flow) and others.

Clearly, P-SPEL can be expanded to other materials including hard (high-melting-temperature) semiconductors, metals, and insulators (since it has been demonstrated that these hard materials be melt and reshaped in ~100 ns[1,2]) and can have a variety of applications in electronics, optics, magnetics, and biology, hence opening up a new nanofabrication path to broad applications.

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