Rectangular Patterns Using Block Copolymer Directed Assembly for High Bit Aspect Ratio Patterned Media

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ABSTRACT We report a nanofabrication method that combines block copolymer directed assembly with e-beam lithography to achieve highly uniform rectangular patterns with a critical dimension of 16 nm, a full pitch of 27 nm, and arbitrary aspect ratio. This fabrication method enables geometries that are not natural to block copolymer assembly, preserves both the feature uniformity and the center-to-center spacing of the original block copolymer, sustains long-range translational order, and facilitates high-resolution, high-density patterns through feature density multiplication. These highly uniform arrays of dense rectangular features are particularly attractive for fabricating magnetic bit patterned media with high bit aspect ratio.

KEYWORDS: directed self-assembly · block copolymer · nanofabrication · lithography · patterned media

With the demonstration of feature density multiplication by e-beam directed self-assembly, block copolymer lithography stands as a promising method to generate master patterns for nanoimprint templates for magnetic bit patterned media technology.[1,2] Block copolymer self-assembly excels at forming periodic patterns with a highly uniform center-to-center spacing and a tight feature size uniformity.[3,4] When it comes to dimensions below 20 nm, they can outperform conventional resists in these two aspects. Block copolymer patterns are, nonetheless, limited by their short translational order, which typically decays over short-range distances if the pattern is not supported by an external condition to predetermine pattern registration.[5−7] Directed assembly has proved successful at providing registration together with long-range orientational and translational order.[8−10] In particular, electron-beam (e-beam) directed assembly also offers a path to enhanced resolution and reduction of e-beam writing times by means of feature density multiplication.[1,11−14] However, block copolymer patterns as lithographic masks are restricted to a limited set of geometries. Most typically, the morphologies considered for lithographic applications are those that form hexagonal close packed (hcp) arrays of dots from spherical or cyilindrical phase block copolymers or sets of periodic stripes from cylindrical or lamellar phase block copolymers.[4,15] Here we expand the capabilities of e-beam directed assembly to generate periodic arrays of rectangular features with a 16 nm critical dimension, 27 nm full pitch, and arbitrary aspect ratios. This technique is an enabler for applications that require both sublithographic dimensions and geometries that are not natural to self-assembly.

Magnetic bit patterned media (BPM) has been proposed as a solution for thermally stable magnetic data storage at densities in excess of 1Tb/in^2.[16,17] In BPM, individual magnetic bits are lithographically patterned onto the media disk with a full pitch below 27 nm placing an unprecedented lithographic challenge with a more aggressive roadmap for critical dimensions than the semiconductor industry roadmap.[18] E-beam directed assembly is one of the most promising methods to achieve the lithographic dimensions needed for BPM together with a high degree of size and placement uniformity.[19] Up to now, block copolymer lithography for BPM applications has been contemplated using block copolymer films that define circular features or "dots" in hexagonal close packed arrays either by spherical phase or cylindrical phase block copolymers with the cylinders oriented perpendicularly to the substrate.[1,15,20−23] The hcp array of dots maximizes the feature density for a given feature separation distance, but restricts the feature shape to be generally circular. In magnetic recording, certain hard disk drive architectures may require rectangular bit

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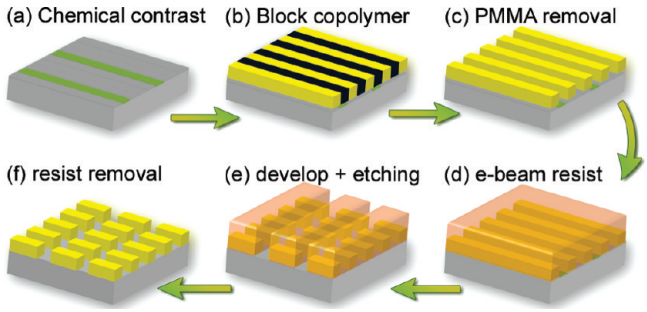

Figure 1. Fabrication of rectangular patterns with arbitrary aspect ratio from lamellar phase block copolymer stripes: (a) chemical contrast substrate with sparse lines; (b) block copolymer film in registration with the sparse pattern; (c) removal of one block from the polymer film; (d) deposition of e-beam resist and e-beam exposure; (e) e-beam resist developing and removal of exposed areas; (f) removal of remaining e-beam resist leaving the remaining polymer film in an array of rectangular features.

cells with bit shapes that are elongated in the cross-track direction and narrow in the down-track direction. Such bit cells may enable the use of wider write-head poles, which can be desirable to achieve the high write fields needed to write high-coercivity media.[24] Rectangular bit islands arranged in quasi-rectangular lattices also offer a more natural extension of the disk drive roadmap, which is based on conventional granular media. Outside magnetic recording, dense arrays of rectangular features with full pitch below 30 nm may also prove useful in semiconductor and other nanofabrication applications.

In this paper, we demonstrate a nanofabrication method to achieve highly uniform rectangular patterns combining block copolymer directed assembly with e-beam lithography to achieve highly uniform rectangular patterns with a critical dimension of 16 nm, a full pitch of 27 nm, and arbitrary length/width aspect ratio. Our method preserves both the natural feature uniformity and the natural center-to-center spacing of the original block copolymer. Previous efforts have made significant progress at achieving morphologies in block copolymer thin films that deviate from the common hcp or striped patterns used for lithography. Thin films of triblock copolymers and blends with a similar phase behavior have been demonstrated to form features in square lattices.[25,26] However, the phase diagram limits the application to features with a symmetric aspect ratio in a square lattice. Directed assembly with polymer blends[27−29] offers a more flexible method to achieve a larger variety of shapes such as elongated features, oblique lines, and alternating dashes and dots. However, the use of polymer blends comes at the expense of size uniformity and a shift of the center-to-center spacing toward larger dimensions due to the increased volume in the polymer blend.[30,31] When considering bit patterned media applications with a high bit aspect ratio architecture, specifications call for the high resolution and tight feature size uniformity of the original block copolymer. It is also highly desirable to have the flexibility to generate an arbitray length/width aspect ratio that can be tuned to the write-head design. In this work, we show a fabrication method that generates rectangular features from lamellar phase block copolymers with high feature size uniformity and an arbitrary aspect ratio.

RESULTS AND DISCUSSION

The fabrication process is depicted in Figure 1. A substrate coated with hydroxyl-terminated polystyrene (PS) is prepatterned using e-beam lithography and reactive ion etching (RIE) to form sparse stripes with chemical contrast (Figure 1a) as described in more detail in the experimental section and elsewhere.[1,12] In Figure 1b a lamellar phase poly(styrene-*block*-methyl methacrylate), PS-*b*-PMMA, block copolymer film is applied and thermally annealed. Provided the chemical contrast stripes are commensurate to the spacing between lamellae, L_o = 27 nm, the block copolymer stripes align in registration to the chemical contrast substrate. In Figure 1c, the PMMA block is selectively removed. In Figure 1d, a new film of e-beam resist is applied, and a second set of stripes perpendicular to the remaining PS lines is exposed by e-beam. The e-beam resist is developed as shown in Figure 1e, and oxygen plasma is used to remove the exposed portions of the PS stripes. Finally, in Figure 1f, the remaining e-beam resist is selectively removed leaving an array of rectangular PS features. Further details can be found in the Methods.

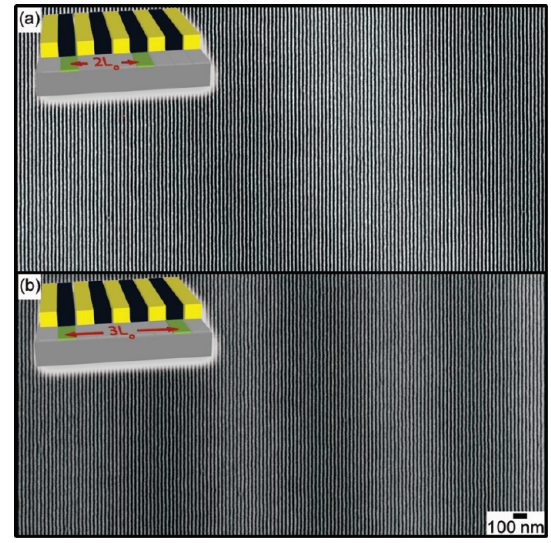

Figure 2. Directed self-assembly of PS-*b*-PMMA lamellae stripes with a full pitch of 27 nm and a density multiplication factor of (a) 2 and (b) 3. Insets are schematic representations of block copolymer assembly on chemical contrast patterns.

In our experiments, the sparse chemical contrast patterns were exposed with a line separation of $L_s = 2L_o$ and $L_s = 3L_o$ where $L_o = 27$ nm is the center-to-center lamellae spacing of the block copolymer in an undirected thin film. Scanning electron microscope (SEM) micrographs of the block copolymer striped patterns assembled on chemical contrast sparse patterns with $L_s = 2L_o$ and $3L_o$ are shown in Figure 2, parts a and b, respectively. The insets are schematic representations of the registration between the e-beam defined chemical contrast patterns and the block copolymer domains. In both cases the block copolymer films form defect-free arrays of stripes with a full pitch equal to L_o multiplying the feature density by a factor of 2 and 3, respectively, when compared with the e-beam written sparse chemical contrast patterns. The resulting striped pattern in both cases is the same except for the amount of residual brush under the PMMA domains. This method for feature density multiplication on chemical contrast patterns is similar to previous examples also reported for lamellar-forming PS-*b*-PMMA block copolymers,[11] except that here we use a hydroxyl-terminated surface modification layer that may be better suited for high-fidelity pattern transfer due to the relative ease of removal.

A dry developing process is more effective in selectively removing the PMMA block from the block copolymer stripes than a wet developing process to avoid pattern collapse. In PS-*b*-PMMA block copolymer films, PMMA is commonly removed by first exposing the film to UV radiation followed by immersion in acetic acid.[32] This developing process is commonly used in cylindrical-phase or large-pitch, low vertical aspect ratio lamellar phase block copolymers. However, in small pitch or large vertical aspect ratio features, capillary forces will tend to collapse the remaining vertical lamellae if the feature widths are too small or their vertical aspect ratio too high. Figure 3a shows an example of pattern collapse during wet developing of a PS-*b*-PMMA film with a full pitch of 27 nm. A low etch rate dry-developing process using oxygen plasma can be used to prevent collapse. Under O_2 plasma RIE, PMMA etches faster than PS. It is possible to find conditions to remove all of the PMMA and still leave sufficiently thick PS stripes. Figure 3b shows an example of PS stripes after PMMA removal. It is possible to observe some residual material every other stripe, which could come from the remaining PS brush layer in those locations that were situated under the PMMA blocks and that had not been exposed by the original e-beam exposure when generating the chemical contrast pattern. Another explanation is a potential domain reconstruction in which the PS domains rearrange in a "U" shape at the locations not exposed by the e-beam. With a slight overetch, however, this residual material can be cleared as shown in Figure 3c.

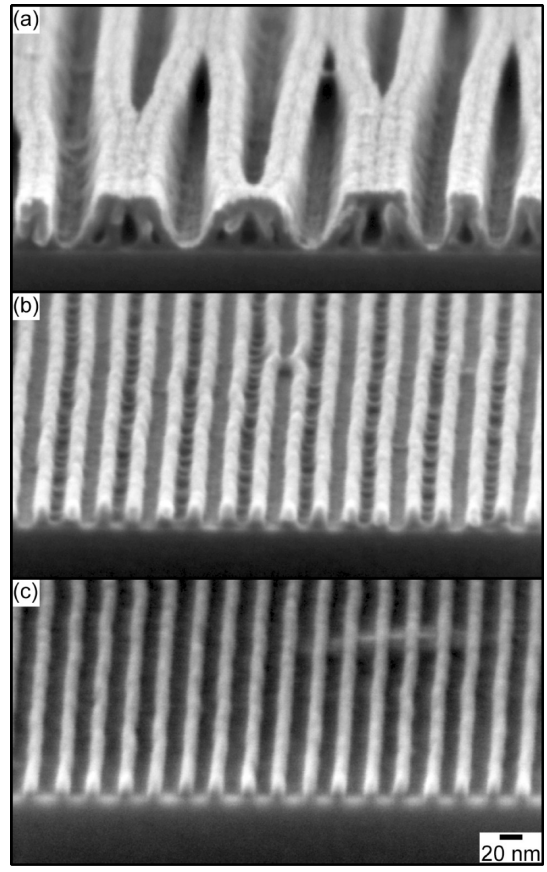

Figure 3. PMMA removal in lamellar phase block copolymers: (a) pattern collapse after PMMA removal by wet developing; (b) PMMA removal by O_2 plasma RIE; PS residue can still be seen every other line; (c) complete PMMA and brush removal after longer O_2 plasma etch.

We use a second lithographic step to cut portions out of the remaining PS stripes to generate rectangular structures. The process used in this work starts with a thin Si coating (~2 nm thick) on top of the PS stripes to prevent the structures from moving or distorting in the following liquid process steps. A ZEP e-beam resist is spin coated on top as in Figure 1d. Using e-beam, we exposed an array of line patterns perpendicular to the PS lines with a full pitch of 54 nm. After developing the ZEP resist, we used reactive ion etching to remove the exposed portions of the PS stripes. We first use a CHF_3 plasma to remove the thin SiO_2 coating followed by O_2 plasma to remove the PS as shown in Figure 1e. Finally as depicted in Figure 1f, an array of rectangular PS features is obtained after removing the remaining e-beam resist. Figure 4 shows a top-down SEM micrograph of the rectangular PS patterns at the end of the process as well as a side view from a 10° tilt angle. In this case, we chose the second lithographic step to define lines with a spacing of 54 nm that when combined to the block copolymer pitch at $L_o = 27$ define a rectangular lattice with an aspect ratio of two for the unit cell; however, any arbitrary value could be chosen for the spacing of the second lithographic step. There is also poten-

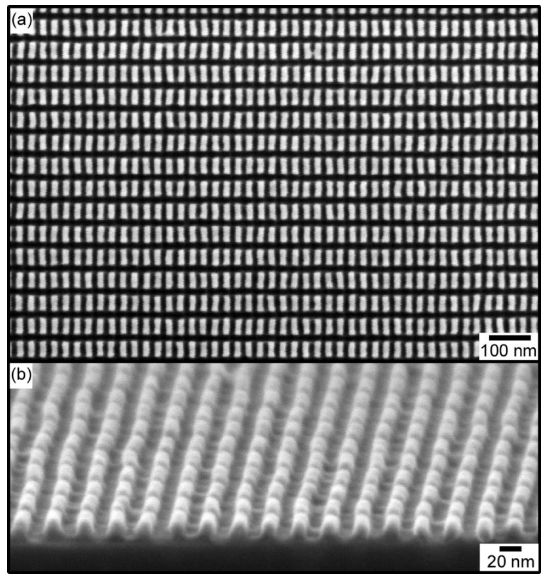

Figure 4. SEM micrographs of PS rectangular patterns at the end of the process. The full pitch in the horizontal and vertical directions is 27 and 54 nm, respectively: (a) top-down view; (b) view from a 10° tilt angle.

tial for other creative periodic or nonperiodic shapes depending on how the second lithographic step is designed and overlaid.

The uniformity afforded by self-assembly in feature size and pitch, combined with the long-range orientational and translational order induced by e-beam lithography, provides superior size uniformity and placement accuracy for these dense features. We performed image analysis on an SEM micrograph from a sample area of 2528 × 2528 nm^2, containing over 4 × 10^3 features. In the analysis, each object is identified and measured to extract the centroid coordinates and the dimensions of each rectangle. The centroids are used to fit the positions of the array to a rectangular lattice. The magnitudes of the lattice vectors in the x and y directions are L_{px} = 27.36 nm and L_{py} = 54.61 nm as measured from the SEM micrographs. Figure 5a shows a small portion of the total area analyzed with a grid of the best-fit lattice superimposed in green.

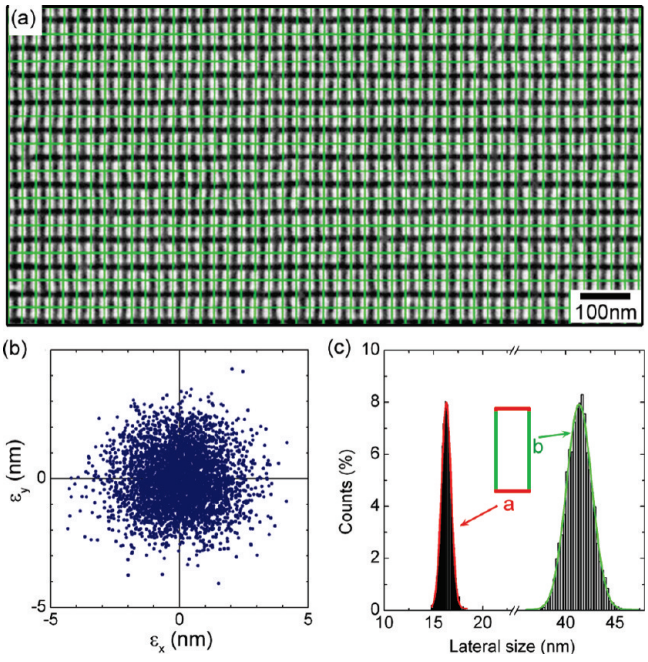

Figure 5. Placement accuracy and size uniformity: (a) SEM image with a superimposed grid of the best-fit rectangular lattice; (b) placement error about the best-fit lattice point for over 4 × 10^3 features; (c) size distribution of the short and long sides of the rectangular features. The mean sizes with standard deviation are a = 16.3 nm, σ_a = 0.49 nm, and b = 41.3 nm, σ_b = 1.3 nm, respectively.

Placement accuracy is quantified by the vector distance between the centroid of a rectangle and its best-fit lattice point. Placement errors for all features in the analyzed image are plotted in Figure 5b. The placement error vector, $\boldsymbol{\varepsilon}$, is the residual of the best fit to the lattice and therefore, by definition, has a mean value of $\boldsymbol{\varepsilon}$ = 0. The standard deviation of placement error in both x and y directions are used here to quantify placement accuracy. The resulting values were σ_x = 1.24 nm and σ_y = 1.16 nm. Both values lie below 5% of L_{px} and L_{py}, respectively, as required by the specifications set for bit patterned media.[16] In this experiment, σ_x is determined by the block copolymer, while σ_y is limited by the e-beam lithography.

The size distributions for both the short and long sides of the rectangle are plotted in Figure 5c. The short side of the rectangle is defined purely by the block copolymer and has a mean value of a = 16.3 nm and a standard deviation of σ_a = 0.49 nm (or 3.0%). The long side, in turn, is defined by e-beam lithography and shows b = 41.3 nm with σ_b = 1.3 nm (3.1%). However, percentage wise, e-beam values tend to increase with smaller dimensions highlighting the advantage of using block copolymer for the critical dimension. We also note here that the mean value of a is larger than the expected $^1/_2L_{px}$ from a symmetric block copolymer due to the thin Si coating deposited after PMMA removal.

SUMMARY

In summary, we have presented a fabrication method to achieve high-density rectangular patterns where the critical dimension (16 nm) is defined by block copolymer directed assembly of a lamellae forming material with a full pitch of 27 nm. A dry-developing process was used to remove the PMMA block from the block copolymer to avoid pattern collapse. The PS stripes formed by the block copolymer are subsequently cut into rectangular shapes using e-beam lithography. The aspect ratio of the rectangular cell can be chosen to any arbitrary value to fit the design of a particular application. We also anticipate that the second lithographic step could be done by a second block copolymer using

directed assembly. The high feature size uniformity, placement accuracy, and density multiplication afforded by the block copolymer materials, makes this a promising candidate to define patterned media templates with rectangular bits where the aspect ratio can be tuned according to design needs.

METHODS

In our experiments, we used a hydroxyl-terminated polystyrene, PS−OH, with a weight-average molecular weight, M_w = 3.7 kg/mol, and a symmetric poly(styrene-*b*-methyl methacrylate), PS-*b*-PMMA, block copolymer with 50% PS content, total M_w = 51 kg/mol. Both polymers were spin-casted from toluene solutions.

Si wafers (with native silicon oxide) were spin coated with PS−OH. The film was vacuum annealed at 210C. A PMMA e-beam resist was then spin coated on the substrate, and a 100 kV e-beam was used to expose arrays of striped patterns with a full pitch of L_s = 54 and 81 nm. L_s was chosen to match twice or three times the natural pitch of the PS-*b*-PMMA block copolymer observed on undirected films (L_o = 27 nm). After exposure and development of the striped patterns on the PMMA resist, the exposed areas were partially removed/oxidized by a brief exposure to oxygen plasma.[1] After the PMMA resist is stripped away, the resulting substrate, as shown in the schematics of Figure 1a consists of a PS−OH coated surface having a chemical contrast with striped patterns on those locations that were first marked by the e-beam exposure.

A 30 nm thick film of the lamellae-forming PS-*b*-PMMA was spin coated onto the chemical contrast patterns and annealed in vacuum for 1 h. The block copolymer registers with the chemical contrast pattern forming lamellae domains perpendicular to the substrate and aligned along the chemical contrast stripes. The PMMA domains register on top of the oxygen plasma exposed stripes. Since the block copolymer forms stripes with a full pitch of 27 nm, that is, with a higher frequency than the chemical contrast pattern, the block copolymer multiplies the feature density by a factor of 2 (for L_s = 54 nm) or 3 (for L_s = 81 nm). Other film thicknesses can also be sustained by DSA, we chose 30 nm as a good compromise between feature quality and clean removal of PMMA during dry developing.

The PMMA of the block copolymer stripes was removed by a dry process using oxygen plasma. An oxygen plasma process that etches PMMA about 2.2 times faster than PS was used to remove PMMA and still keep close to half of the PS thickness after all of the PMMA is removed. A thin (~2 nm) Si coating was deposited by RF sputtering to protect the remaining PS stripes from deformation during the following wet processing steps.

A film of ZEP 520A resist was spin coated onto the substrate, and e-beam was used to expose an array of lines perpendicular to the underlying PS stripes with a full pitch of 54 nm ($2L_o$). After development of the ZEP 520A resist, the sample was exposed to a CF_4 plasma to remove the thin coating layer followed by an oxygen plasma to remove the portions of the exposed PS underneath as represented in Figure 1e. Finally, the remaining ZEP 520A resist was removed in warm *N*-methyl-2-pyrrolidone. The final result is an array of rectangular PS structures on the wafer as depicted in Figure 1f and Figure 4.

Image Processing. SEM micrographs for image analysis were acquired at a resolution of 1.23 nm/pixel with a size of 2528 × 2528 nm^2. The image size contained over 4 × 10^3 features with about 440 pixels per feature. Low- and high-frequency noise was filtered with a band-pass filter.[1,33] We utilized square images so that the analysis comprised the same range of wavelengths in both dimensions. After filtering, the mean gray tones for the background and foreground were identified from the histogram and the midpoint between them was used to apply a threshold filter rendering a black and white image. Each rectangular object was identified by a labeling algorithm that identifies and labels the pixels that form each object. Once the individual objects were labeled, statistical analysis followed. For each feature in the image, we extracted size, centroid, and second moment of area about both centroidal axes (*i.e.*, I_{xx} and I_{yy}).

The centroids of each feature were fitted to a two-dimensional lattice with lattice vectors $\mathbf{L_{px}}$ and $\mathbf{L_{py}}$. The fitting parameters were L_{px}, L_{py}, α, θ, x_o, and y_o, where L_x and L_y are the magnitudes of the lattice vectors and α is the angle between them; θ is the angle between $\mathbf{L_{px}}$ and the *x*-axis. Finally, x_o and y_o represent the offset of the lattice with respect to the origin. In the model, there is one feature (*i.e.*, "atom") per unit cell located at the lattice point. We found L_{px} = 27.36 nm and L_{py} = 54.61 nm as measured from the SEM micrographs. The discrepancy between the e-beam written dimensions (L_{sx} = 27.0 nm and L_{sy} = 54.0 nm) and those measured from SEM micrographs arises from a ~1% discrepancy in the cross-calibration between the tools with the e-beam being the most accurate unit. The residual vector or position error, $\boldsymbol{\varepsilon}$, is defined as the distance between the feature centroid and its best-fit lattice point. The distribution of $\boldsymbol{\varepsilon}$ is shown in Figure 5b. $\boldsymbol{\varepsilon}$ is given by the residual of the least-squares fit, and by definition, $\sum \vec{\varepsilon}_n = 0$. The standard deviation of ε_x and ε_y are σ_x = 1.24 nm and σ_y = 1.16 nm, respectively. Repeated measurements over two other locations yielded similar results with less than 1.5% variability between measurements.

Assuming the features have a rectangular shape, *a* and *b* (the short and long lateral dimensions of the rectangle) are obtained from the second moment of area: $a = 2\sqrt{3}(I_{yy}/A)^{1/2}$ and $b = 2\sqrt{3}(I_{xx}/A)^{1/2}$, where *A* is the area of the rectangular feature. We notice that $(I_{yy}/A)^{1/2}$and $(I_{xx}/A)^{1/2}$are identical to the standard deviation of the mass distribution about the centroidal axes and also identical to the radii of gyration about both centroidal axes. The horizontal or "*x*" axis is chosen to be parallel to $\mathbf{L_x}$, thus I_{xx} and I_{yy} are calculated once θ is known from the lattice best-fit. The distributions of *a* and *b* from all objects are shown in Figure 5c. From an image containing 4103 features, the mean values with their respective standard deviations are a = 16.3 nm, σ_a = 0.49 nm, b = 41.3 nm, and σ_b = 1.3 nm. In this case, *a* is wider than $^1/_2L_{px}$ due to the thin Si coating that was deposited on top of the PS lines after PMMA removal.

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