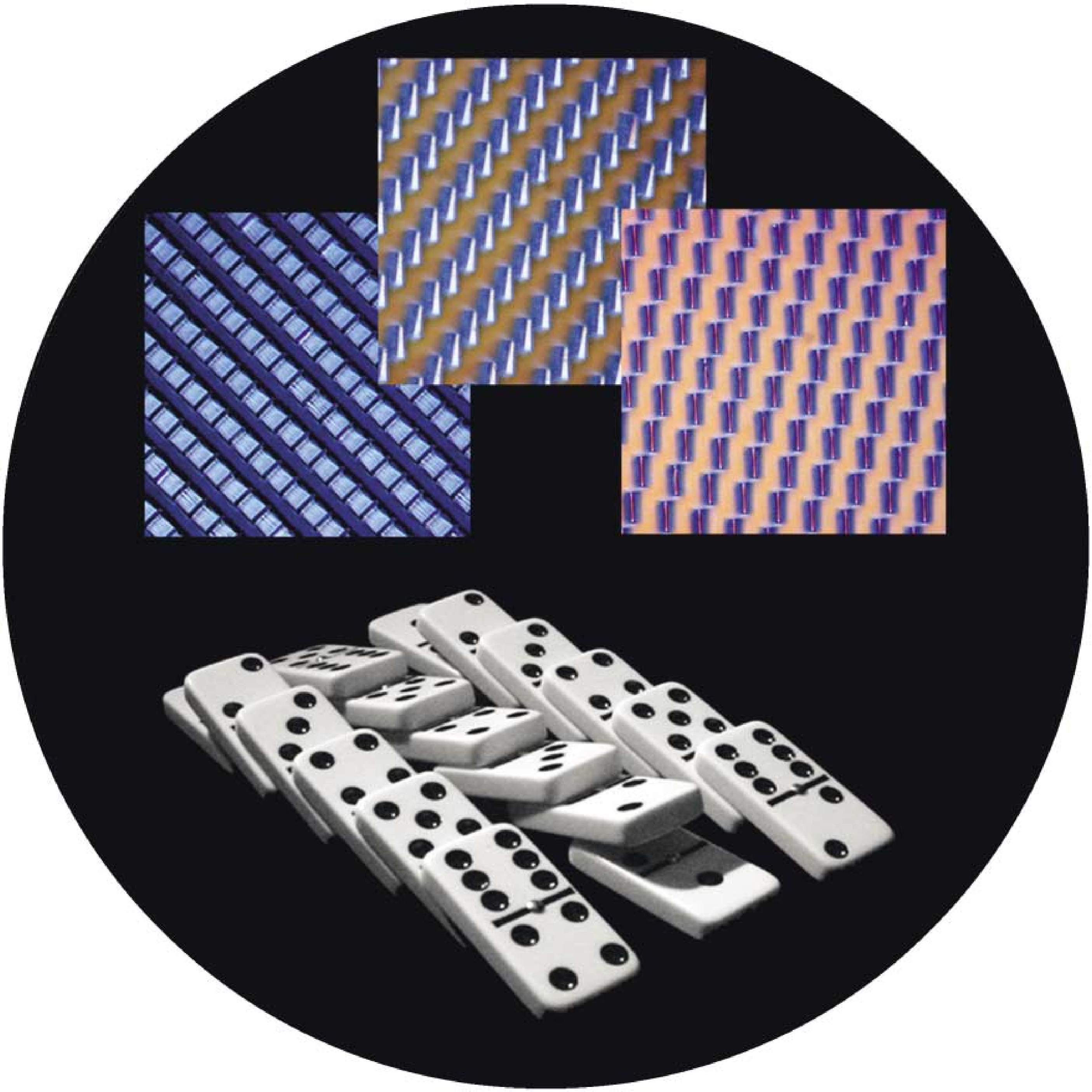
Communications



Tipping large arrays of micrometer-scale dominos onto their side generates complex 3D topographies and arrays of nanoscale electrodes. These microdominos collapse in parallel when a shearing force is applied uniformly to an array of free-standing pillars. For more information, see the Communication by G. M. Whitesides and coworkers on the following pages.

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Nanotechnology

Shear Patterning of Microdominos: A New Class of Procedures for Making Micro- and Nanostructures**

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This paper describes the preparation of fields of micrometer-scale pillars ("microdominos"), and the tipping of these pillars onto their sides by shearing. Previously, the repositioning of micrometer-scale features has focused on the use of surface tension forces to bring features out-of-plane (e.g., mirrors in microelectromechanical systems, or MEMS),[1] or to align in-plane features.[2] Figure 1 illustrates our approach schematically, and gives a representative result showing the uniformity of the process. This process provides a route to repetitive 3D microstructures that would be difficult or impossible to prepare by conventional micro/nanolithography. Since the top surfaces of these pillars can be coated with nanometer-thick films by evaporation, tipping also provides a method of generating regular arrays of features with nanometer-scale lateral dimensions in-plane. Although the use of thin-film deposition techniques to generate features with functional, nanoscale edges is well established,[3] this method provides a simple route to arrays of nanoscale edges that can be addressed electrically.

We fabricated arrays of microdominos on a silicon support by photolithography by using SU-8 photoresist.[4] This silicon substrate was positioned on a 2 mm thick glass substrate by using a poly(dimethyl siloxane) (PDMS) slab as an adhesion layer. We mounted these substrates on a horizontal translation stage equipped with a stepper that had 1 µm resolution. A second PDMS slab was placed on another glass substrate positioned parallel to, and above, the silicon support. Each PDMS block was ≈3 mm thick. We moved the top PDMS slab into contact with the microdominos by using a vertical translation stage that also had 1 µm resolution. The transparency of the top substrates allowed us to use optical microscopy to observe the contact between the PDMS slab and the microdominos. Horizontal translation of the bottom substrate created a shear force between the array of microdominos and the upper elastomeric block. The applied shear induced cohesive failure of the SU-8 at the base of each polymer structure, and each microdomino in the area was tipped onto one of its sides. When we separated the two substrates, the fractured microdominos transferred from the silicon wafer to the top PDMS slab. The pattern of the collapsed structures was determined by the geometry of the microdominos and the direction of the horizontal component of the shear.

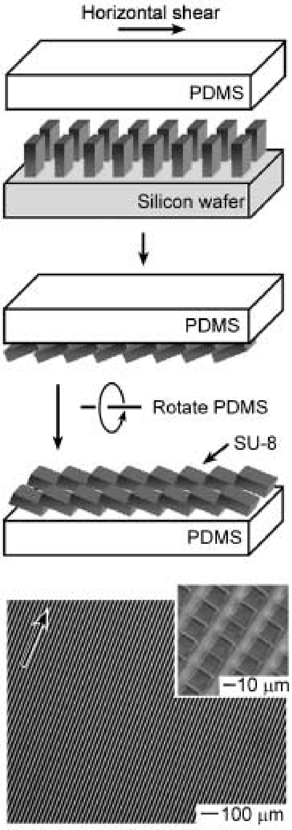

Figure 1. Schematic of the procedure used to reorient microdominos. The sheared polymer structures transferred to the PDMS surface when the PDMS block was separated from the silicon wafer. The optical microscopy image shows overlapping 20 µm tall rectangles of SU-8 supported on PDMS. We fabricated this topography by shearing an array of rectangles (with a 5 µm×15 µm cross section) in the direction indicated by the arrow—perpendicular to the 15 µm edge. The inset shows a scanning electron microscopy (SEM) image of these overlapping microdominos.

To be able to tip the dominos reproducibly, they must fracture from the substrate under shear. Slight modifications were made to the manufacturer's recommended conditions for photolithography by using SU-8 when fabricating the microdominos: we used a shorter (3 min) pre-exposure bake at 95 °C than that recommended, followed by a lower integrated intensity ($150\,\mathrm{mJ\,cm^{-2}}$) UV exposure.[5] These altered parameters decreased the cross-linking density in the microdominos; when sheared, the resulting polymer posts fractured at the interface with the silicon substrate.

By shearing the microdominos with a PDMS slab, we achieved uniform pattern generation and transfer. In our hands, other materials (e.g., poly(methyl methacrylate) (PMMA) and polyvinyl chloride) when coated onto a silicon support or a PDMS slab, generated irregular patterns of

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collapsed microdominos on shearing; many of these structures remained on the silicon wafer. PDMS has two important roles in obtaining uniform patterns of sheared microdominos: 1) this polymer adheres to the SU-8 microstructures noncovalently; and 2) the elastomeric properties of PDMS permit it to deform upon contact with the polymer posts, thus absorbing some of the force upon contact.

This procedure will generate uniform patterns of tipped microdominos covering up to about 1 cm^2.[6] A uniform applied force is important to obtain homogeneous patterns over this large area. To address this concern, we cured the PDMS substrates on a flat surface and used a level to align the substrates prior to applying the shear. Uniform collapse of the microdominos can be prevented by dust particles on either substrate; conveniently, the elastomeric slab conforms to surface contaminants and minimizes the effect of these abnormal topographies.[6] To limit surface contamination from airborne particles, we cured the PDMS against a flat substrate without exposing this PDMS surface until it was used for shearing the microdominos.

Figure 2 shows some of the topographies that can be formed by shearing rectangular, triangular, and circular posts. Figures 2 A and B show a ribbed structure that was created by shearing 20 µm tall rectangles in a direction perpendicular to their longest lateral edge. The height and spacing between the microdominos determines the amount of overlap between the neighboring structures. We patterned different topographies by applying a shear to the rectangles at various angles (Figures 2 C–E). The rectangles in Figures 2 C and D were tipped onto the narrowest edge (5 µm wide). In Figures 2 E and F the structures were also tipped onto the narrowest edge; this arrangement was, however, unstable and these microdominos subsequently collapsed onto their widest (15 µm) face.[6] Collapse in this second direction, perpendicular to the applied horizontal shear, is difficult to control as the intermediate stage (microdominos tipped onto the 5 µm edge) is unstable towards the slightest horizontal translation. Figure 2 G–J show microdominos of different shapes tipped onto one side. The sheared triangles in Figures 2 G and H and the cylindrical rods in Figures 2 I and J each provide a topography that is impossible to obtain by conventional photolithography.[7]

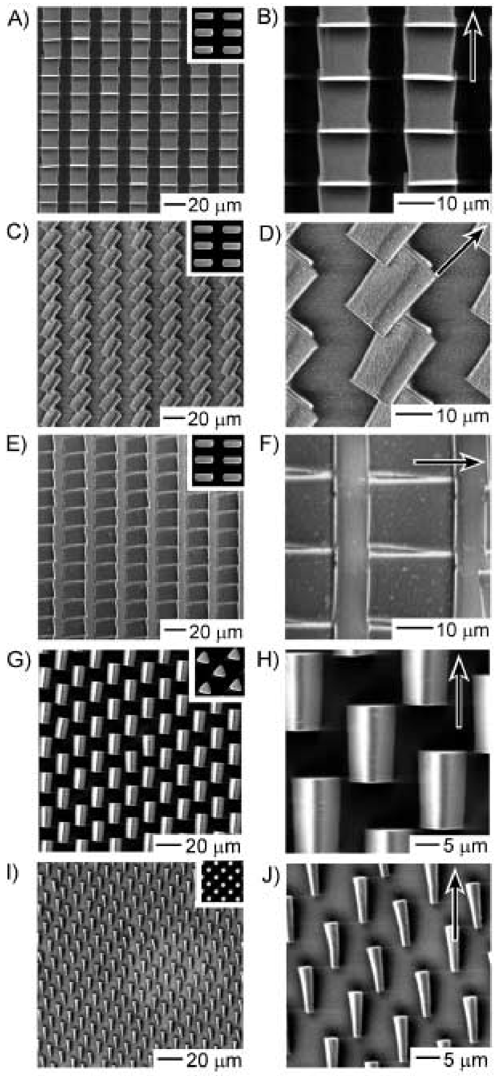

Figure 2. SEM images of rectangles, triangles, and cylinders of SU-8 that were transferred to a PDMS block after applying a shear. The insets show a top view of the original, uncollapsed pillars and the arrows indicate the direction of the shear. (A, B) Stacked microdominos (LxWxH: 15 µm × 5 µm × 20 µm) oriented by a horizontal force applied in a direction perpendicular to the 15 µm edge of the rectangles. The structure of the stacked rectangles can be changed by controlling the direction of shear relative to the 15 µm edge: (A, B) 90°; (C, D) 45°; (E, F) 0°. Distinct patterns were obtained from a square lattice of equilateral triangles (5 µm on a side) sheared at about 45° relative to the orientation of the lattice (G, H). Shearing 10 µm tall cylindrical posts reorients the vertical array into a planar array of parallel, approximately cylindrical rods (I, J).

Electrets are dielectric materials with the ability to store an electric field—either charge or polarization induced.[8] We have previously patterned charge in electrets by using electrodes with dimensions down to about 150 nm, patterned by conventional lithography.[9] Here we used an array of narrow electrodes obtained by shearing microdominos to print, in parallel, a pattern of charge into a film of PMMA (Figure 3). A 15 nm metal film, deposited by electron-beam deposition at a 20° angle relative to the plane of the silicon substrate, coated the top and part of one of the 15 µm faces of the original microdominos (Figure 3 B). We deposited Pd onto the microdominos because of the small grain size (15–20 nm) of this metal when deposited by electron-beam evaporation and its resistance to oxidation.[10] The microdominos were sheared by using a substrate of conducting polyaniline (PANI) grafted onto a PDMS slab, which also served as an electrode.[6,11] The array of 15 µm tall rectangles, capped with Pd, were tipped onto this conducting polymer surface (Figures 3 C and D) with one of the metal-coated surfaces in contact with the conducting polymer electrode. We imaged the exposed Pd features by SEM after etching the SU-8 with reactive ion etching, thus confirming an edge thickness of about 15 nm.[6] Figures 3 D and E show the configuration of the metal-capped microdominos, after tipping, for printing charge into PMMA.

Printing charge from the (now electrically connected) array of edges is straightforward. The dimensions and shape

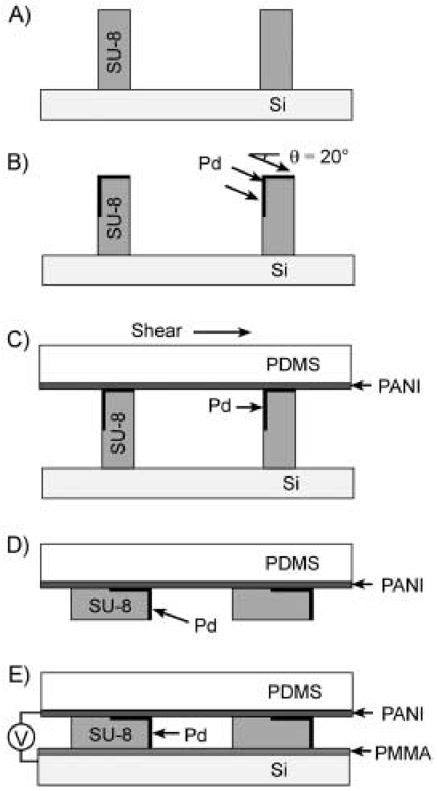

Figure 3. Schematic for preparing arrays electrodes with narrow lateral dimensions by using microdominos as templates. The microdominos of SU-8 (A) defined by photolithography were coated with a Pd film (B) at an angle of 20° relative to the silicon substrate. C) A slab of PDMS with PANI grafted onto the surface was used to apply a horizontal shear to the microdominos. D) The collapsed microdominos transferred to the PANI/PDMS substrate, and were brought into contact with a PMMA film supported on a silicon wafer. We used the Pd films as electrodes with narrow lateral dimensions (E) by applying a potential between the PANI and the silicon substrate.

of the microdominos determined the pattern. Figures 4 A and B show SEM images of an array of Pd capped 15 μm tall rectangles transferred onto the polyaniline surface. After bringing a thin film of PMMA into contact with this array of electrodes, we applied a voltage of +50 V for 30 s (current density of ca. 20 nA nm^{-2}, or ca. $2 \cdot 10^6$ electrons per Pd atom) between the conducting polymer and the silicon wafer (Figure 3 E). We separated the patterned electrode from the PMMA, and imaged the surface potential of the PMMA by Kelvin probe force microscopy (KFM). Figures 4 C and D show the topography of the PMMA surface (ca. 0.5 nm rms roughness), and an array of 600 nm wide regions of charge. These lines of charge show a peak surface potential of about 400 mV.

In summary, we have demonstrated that a vertical structure defined with photolithography can be easily repositioned onto one side face by shear. The procedure requires tuning the conditions of conventional photolithography to acquire an array of polymer posts that can easily collapse upon applying the shear. This patterning technique has been applied to, but is not limited to, arrays of rectangular, triangular, and cylindrical posts. The reorientation of microdominos offers routes to 3D micrometer-scale structures that are now difficult or impossible to prepare. This tunable fabrication approach to 3D topographies could find several applications in microfabrication and materials science. For example, simple arrays of Pd structures with narrow lateral dimensions were prepared by tipping over Pd capped micrometer-scale structures, which could be electrically addressed to pattern charge in electrets.

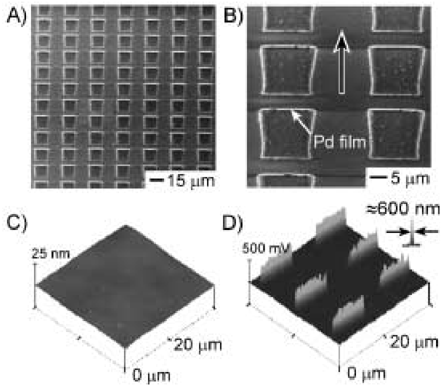

Figure 4. Printing charged regions in electrets from arrays of nanometer scale conducting features: (A, B) SEM images of 15 μm tall rectangles capped with a 15 nm Pd film and sheared onto a polyaniline coated PDMS substrate. The arrow indicates the direction of the applied shear. (C, D) Topography (25 nm vertical scale) and surface potential (500 mV vertical scale) of the 100 nm thick PMMA surface after patterning regions of charge (ca. 600 nm wide, ca. 400 mV peak surface potential). Each 3D plot is shown at a 45° angle for clarity. The inset in (D) shows the side profile for one region of charge.

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[1] See, for example: a) K. F. Harsh, V. M. Bright, Y. C. Lee, *Sens. Actuators A* **1999**, *77*, 237–244; b) R. A. Syms, E. M. Yeatman, V. M. Bright, G. M. Whitesides, *J. Microelectromech. Syst.* **2003**, *12*, 387–417.

[2] See, for example: a) U. Srinivasan, D. Liepmann, R. T. Howe, *J. Microelectromech. Syst.* **2001**, *10*, 17–24; b) H. O. Jacobs, A. R. Tao, A. Schwartz, D. H. Gracias, G. M. Whitesides, *Science* **2002**, *296*, 323–325.

[3] These edge structures have been explored for applications as vertical cavity lasers, magnetic storage media, nanoband electrodes, and templates to grow nanowires to be incorporated into nanoelectronic devices. See, for example: a) N. A. Melosh, A. Boukai, F. Diana, B. Gerardot, A. Badolato, P. M. Petroff, J. R. Heath, *Science* **2003**, *300*, 112–115; b) S. L. Caston, R. L. McCarley, *J. Electroanal. Chem.* **2002**, *529*, 124–134.

[4] Y. Xia, G. M. Whitesides, *Angew. Chem.* **1998**, *110*, 568–594; *Angew. Chem. Int. Ed.* **1998**, *37*, 550–575.

[5] MicroChem, Inc. (Newton, MA), see www.microchem.com.

[6] See Supporting Information for further details and examples.

[7] Each cylinder has a tapered structure because of the short UV-exposure used when originally defining the arrays of cylindrical posts.

[8] *Electrets, Vol. 1–2* (Ed.: G. M. Sessler), 3rd ed., Laplacian Press, Morgan Hill, CA, **1998**.

[9] H. Jacobs, G. M. Whitesides, *Science* **2001**, *291*, 1763 – 1766.
[10] J. C. Love, D. B. Wolfe, M. L. Chabinyc, K. E. Paul, G. M. Whitesides, *J. Am. Chem. Soc.* **2002**, *124*, 1576 – 1577.
[11] To our knowledge this is the first demonstration of grafting PANI onto a PDMS substrate. A similar procedure has been used to graft PANI onto glass substrates (e.g., Z. F. Li, E. Ruckenstein, *Synth. Met.* **2002**, *129*, 73 – 83).