

Fabrication of Metal Structures with Nanometer-Scale Lateral Dimensions by Sectioning Using a Microtome

Qiaobing Xu, Byron D. Gates, and George M. Whitesides*

Department of Chemistry and Chemical Biology, Harvard University, 12 Oxford St.,
Cambridge, Massachusetts 02138

Received November 13, 2003; E-mail: gwhitesides@gmwgroup.harvard.edu

This Communication describes the fabrication of planar structures comprising metallic features with nanometer-scale lateral dimensions in a polymer, prepared by sectioning a thin metallic film—embedded in a polymer matrix—in a plane perpendicular to the metallic film using a microtome (Figure 1). This procedure converts a structure that is thin along the *z*-axis into a structure that is thin along the *x*-axis.

There are many ways to grow films with thickness between 1 and 50 nm over large lateral areas: the most important are deposition and adsorption.¹ Developing methods to fabricate structures with in-plane, nanometer-scale features is, however, a continuing problem in nanoscience. Writing using an electron beam or a scanning probe provides the most general access.² Edge lithographic procedures provide more specialized structures.^{3–9} Combinations of multilayer evaporation, fracture, etching, and grinding have generated topographically patterned masters, and fabricated parallel wires;^{8,9} the procedure described in this Communication most closely resembles that of White.⁹

We sandwich a thin metal film (prepared by electron-beam evaporation) between topographically textured polymer slabs and cut the sample perpendicular to the plane of the film using the glass knife of a microtome. This procedure minimizes the delamination between the metal and polymer layer. The width of the exposed metal edge is determined by the thickness of the metal film. This metal edge, contained within an insulating matrix, is electrically conductive. It has the useful feature that it can be addressed electrically on the side of the polymer slab opposite the exposed metal edge; it thus provides an entry into an array of nanoelectrodes.

Figure 1 summarizes the procedure. We prepared an epoxy substrate (Araldite)¹⁰ by casting against a poly(dimethyl siloxane) (PDMS) master comprising $2\ \mu\text{m} \times 2\ \mu\text{m}$ square features. We choose epoxy polymers as the matrix in which to encapsulate the metal film because these polymers (i) cut easily and generate relatively smooth surfaces, (ii) can be electrically insulating and optically transparent, and (iii) adhere well to many surfaces and when polymerized are generally chemically resistant. We deposited a gold film (50-nm thick) onto this patterned epoxy substrate by electron-beam evaporation. The gold-coated epoxy substrate was treated with an oxygen plasma for ~ 20 s (250 mTorr, 100 W barrel etcher). This treatment improved the adhesion of a second layer of epoxy to the metal surface and minimized delamination when sectioning the sandwich structure. This treated substrate was subsequently embedded in the same epoxy. After thermal curing, this process generated a metal film encapsulated in an epoxy matrix. We sectioned the sample perpendicular to the gold film using a microtome equipped with a glass knife operating at $-120\ ^\circ\text{C}$;¹¹ this sectioning exposed a cross-section of the sandwich structure. The resulting surface had an rms roughness of ~ 10 nm, as determined by atomic force microscopy (AFM). Low-temperature sectioning minimized delamination between the polymer and metal

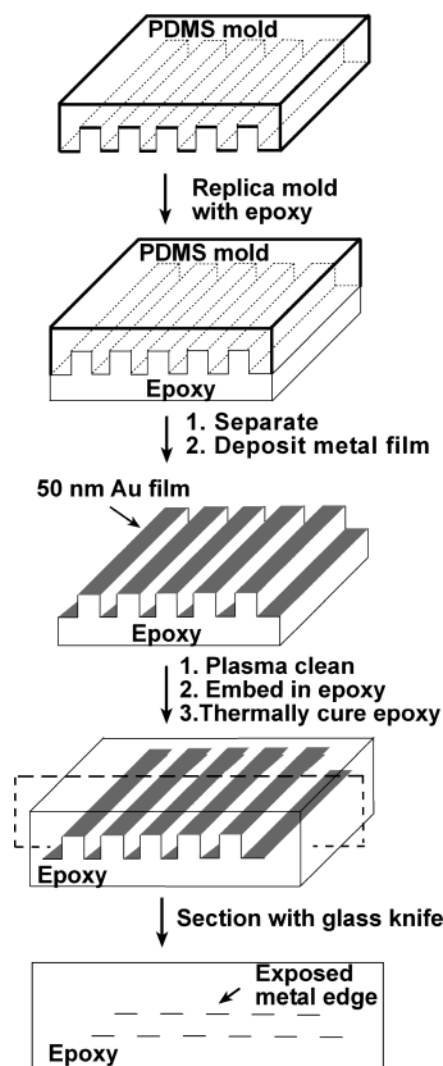


Figure 1. Schematic diagram showing the fabrication of a soft sandwich structure encapsulating a thin metal film. Direction-dependent electron-beam evaporation prevented metal deposition on the side-walls of the epoxy substrate. We exposed the cross-section of the final epoxy-embedded metal film by cutting the substrate with the glass knife of a microtome.

film. The orientation of cutting is also important in maintaining the adhesion between the layers of the sandwich structure; cutting the sample in a direction parallel to the plane of the encapsulated metal film gave the best results.

Figures 2A and 2B show scanning electron microscopy (SEM) images of the cross-section for a 50-nm gold film embedded in the epoxy substrate and exposed by sectioning. The bright lines in the image are due to electrons scattered by the metal. We attribute the roughness of the cross-section to nonuniform cutting of the sample

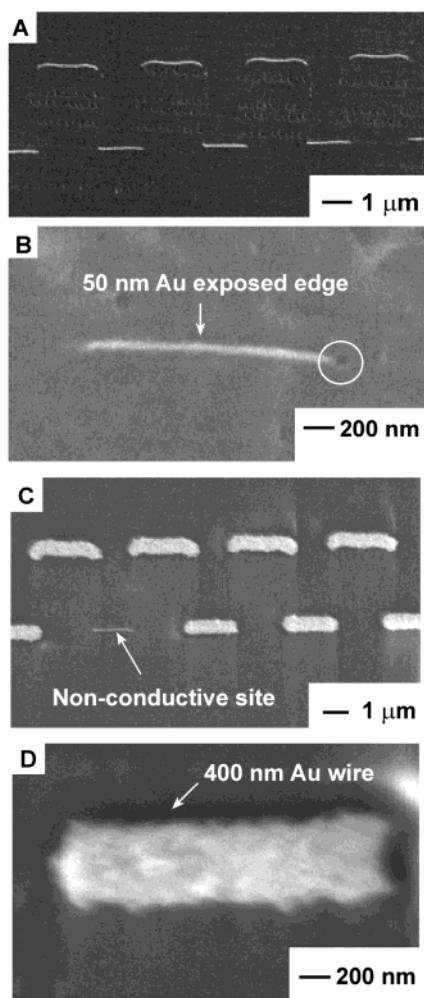


Figure 2. Patterned metal edge of gold exposed in a matrix of epoxy. (A) SEM shows the embedded 50-nm wide, structured, gold film and the continuous epoxy matrix. (B) Close-up for one of the exposed edges shows partial delamination (indicated by the circle) between the metal and the encapsulant. (C, D) Metal edge could be used as an electrode for electrochemical deposition of another metal; gold, electrodeposited onto the gold edge, identified the conductive regions.

by the glass knife. The difference in mechanical modulus between the metal and polymer can result in delamination in regions of high stress (for example, the small hole shown in Figure 2B). We believe more uniform edges can be obtained by using a harder microtome knife (e.g., tungsten carbide or diamond).

SEM demonstrates that a thin metal edge can be exposed by sectioning but does not establish whether the encapsulated thin film is electrically continuous. Electrochemistry accomplished this demonstration. We connected the opposite edge of the encapsulated 50-nm thick gold features to a voltage source and electrodeposited gold.¹⁰ Figures 2C and 2D show SEM image of gold (~500-nm wide) electrodeposited on the exposed 50-nm wide gold edge.

We also sectioned a flat epoxy substrate supporting 12 alternating layers of Ni and SiO₂. Each layer had a thickness of 20 to 100 nm; this thickness was controlled by electron-beam deposition (Figure 3).

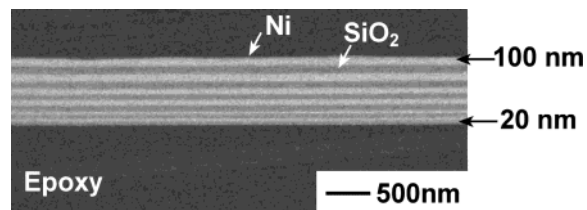


Figure 3. Cross-section of a multilayer Ni/SiO₂ structure, embedded in the epoxy matrix, and exposed by cutting. The thickness of the deposited film was tuned from 20 to 100 nm.

This work demonstrates a simple and inexpensive procedure that generates nanoscale structures in the *x*, *y* plane by a three-step process: (i) forming topographically contoured metal sheets by deposition in the *z*-direction, (ii) embedding these sheets in epoxy, and (iii) sectioning in the *x*, *z* plane. The composition of the exposed nanoedge can be selected by changing the composition of the thin film. Our approach substitutes “sectioning by microtome” for the “writing” commonly done by electron-beam or scanning probe lithography. This fabrication technique needs only readily available facilities—for soft lithography, metal evaporation, spin coating for polymers, and sectioning—but is limited to simple nanostructures (albeit ones that are electrically addressable). Since soft lithography can generate complex 3D structures, and stacking multiple layers is straightforward, it should be adaptable to generate substantially more complex features than those shown in Figure 2.

Acknowledgment. This research was supported by DARPA and used the MRSEC Shared Facilities supported by the NSF under Award No. DMR-9809363. We thank Dr. David Bell and Dr. Richard Schalek of CIMS for assistance with the ultramicrotome and Dr. Yuan Lu for assistance with SEM.

Supporting Information Available: Experimental protocols for the fabrication of the polymer test structures, and the microtome sectioning process. This material is free of charge via the Internet at <http://pubs.acs.org>.

References

- (1) Xia, Y.; Rogers, J. A.; Paul, K. E.; Whitesides, G. M. *Chem. Rev.* **1999**, *99*, 1823.
- (2) Ito, T.; Okazaki, S. *Nature* **2000**, *406*, 1027. Vettiger, P.; Cross, G.; Despont, M.; Drechsler, U.; Gotsmann, B.; Haberle, W.; Lantz, M. A.; Rothuizen, H. E.; Binnig, G. K. *IEEE Tran. Nanotech.* **2002**, *1*, 39.
- (3) Love, J. C.; Paul, K. E.; Whitesides, G. M. *Adv. Mater.* **2001**, *13*, 604.
- (4) Black, A. J.; Paul, K. E.; Aizenberg, J.; Whitesides, G. M. *J. Am. Chem. Soc.* **1999**, *121*, 8356.
- (5) Odom T. W.; Thalladi, V. R.; Love, J. C.; Whitesides, G. M. *J. Am. Chem. Soc.* **2002**, *124*, 12112.
- (6) Yang, H.; Love, J. C.; Arias, F.; Whitesides, G. M. *Chem. Mater.* **2002**, *14*, 1385.
- (7) Cherniavskaya, O.; Adzic, A.; Knutson, C.; Gross, B. J.; Zang, L.; Liu, R.; Adams, D. M. *Langmuir* **2002**, *18*, 7029.
- (8) Stormer, H. L.; Baldwin, K. W.; Pfeiffer, L. N.; West, K. W. *Appl. Phys. Lett.* **1991**, *59*, 1111. (b) Fasol, G. *Science* **1998**, *280*, 545. (c) Melosh, N. A.; Boukai, A.; Diana, F.; Gerardot, B.; Badolato, A.; Petroff, P. M.; Heath, J. R. *Science* **2003**, *300*, 112.
- (9) Morris, R. B.; Franta, D. J.; White, H. S. *J. Phys. Chem.* **1987**, *91*, 3559.
- (10) See Supporting Information for further details.
- (11) Glauert, A. M. *Practical methods in electron microscopy*; American Elsevier Publishing Co. Inc.: New York, 1974; pp 213–329.

JA0395893