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Fabrication of Three-Dimensional Microstructures by Electrochemically Welding Structures Formed by Microcontact Printing on Planar and Curved Substrates

Rebecca J. Jackman, Scott T. Brittain, and George M. Whitesides

Abstract—**This paper describes two convenient techniques for the fabrication of three-dimensional (3-D) structures with micron-sized features. The methods use microcontact printing (μCP) to define patterns with feature sizes as small as 20 μm both on planar substrates and on cylinders (diameter ~2 mm). Electrodeposition serves as a micron-scale tool for metal deposition and welding that transforms these patterned surfaces and cylinders into metallic complex 3-D microstructures (e.g., tetrahedra). Final tetrahedra (~2-cm sides) have feature sizes as small as 50 μm. [286]**

Index Terms—**Electrochemical deposition, fabrication, microcontact printing, three-dimensional microstructures.**

I. Introduction

RAPID GROWTH in the area of microelectromechanical systems (MEMS) has created a demand for three-dimensional (3-D) micron-scale components. Most fabrication techniques used to make components for MEMS have their basis in the microelectronics industry [1]: they can be grouped broadly into the categories of silicon micromachining and through-mask plating. Silicon micromachining [2], [3] uses photolithography to define patterns that can be transferred into silicon, and related materials, by standard techniques. These processes can be combined to build 3-D structures layer by layer. Micromachining, however, has several disadvantages: 1) it can pattern only a limited number of materials; 2) it can only form low-aspect-ratio planar structures with geometries that are determined by the crystallinity of the material; and 3) it requires facilities that limit accessibility and are not well suited to rapid prototyping.

Through mask electroplating [4] or LIGA (Lithographie, Galvanoformung, Abformung), [5]–[8] uses lithography to define a mold in photoresist and then uses electroplating to deposit metal (usually, nickel) in the mold. The use of thick resists (<200 μm) exposed by collimated synchrotron radiation makes high-aspect-ratio structures possible, but variations in the third dimension are still difficult. In one case, 3-D structures have been achieved by use of an elaborate lathe that allows exposure on all sides of a resist-coated fiber [8]. Limited access to synchrotron radiation, however, has resulted in the development of alternative schemes for generating high-aspect ratio molds in photoresist and other materials [9]–[15].

Approaches to MEMS fabrication that represent significant departures from conventional lithography have been successful in "writing" 3-D structures in metals and polymers in serial fashion. These techniques include: UV stereolithography [16], laser-assisted chemical-vapor deposition (LCVD) [17], [18], localized electrochemical deposition [19], and laser micromachining [20], [21].

In this paper, we present two convenient alternatives to conventional photolithography for the fabrication of 3-D structures: these methods use microcontact printing (μCP) [22]–[25] to define patterns both on planar substrates and on cylinders. Electrodeposition then serves as a micron-scale tool for metal deposition [26] and welding that transforms these patterned surfaces and cylinders (after release from their substrates) into metallic complex 3-D structures.

II. Fabrication Methods

A. Fabrication of Photomasks and Stamps

Both of the fabrication methods that we have developed for the generation of 3-D microstructures rely on μCP to create patterns with micron-sized features. μCP uses an elastomeric "stamp," having micron scale relief, to transfer an "ink" to a substrate [22], [27], [28]. This ink can either protect the underlying surface against etching [27], [29] or can initiate deposition of material [30]. We typically form the "stamps" by casting and curing polydimethylsiloxane (PDMS) prepolymer (Sylgard 184, Dow Corning, Midland, MI) against a "master" consisting of a pattern defined in photoresist, although other strategies are also possible [31].

We generated the masters using rapid prototyping [32]. In this technique, a pattern is designed using a computer-assisted design (CAD) program. Designs are printed onto transparencies using a commercial laser-assisted image-setting system (Herkules PRO, 3387 dpi, Linotype-Hell Company, Hauppauge, NY). The smallest features that can be formed by this method are 20 μm, a dimension limited by the resolution

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The authors are with the Department of Chemistry and Chemical Biology, Harvard University, Cambridge, MA 02138 USA.

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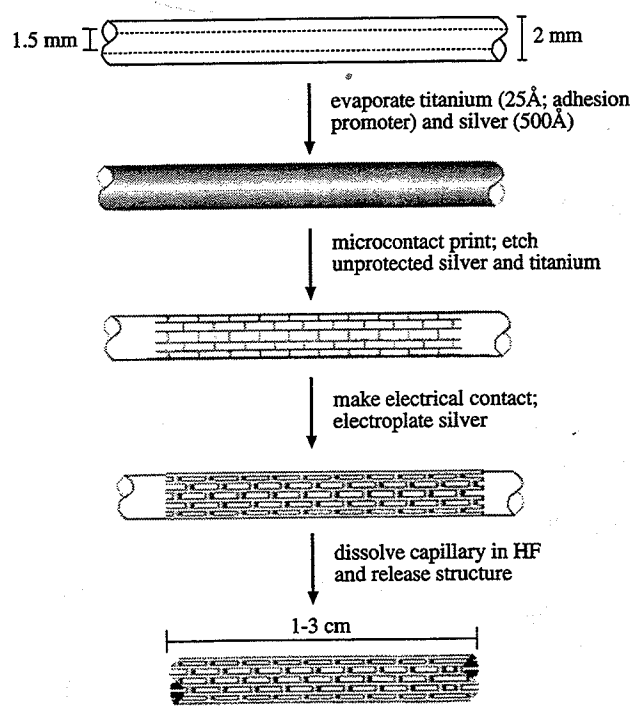

Fig. 1. Scheme for fabricating mesh structures using μCP and electroplating. Glass cylinders were coated with titanium (~25 Å) and silver (~500 Å) using an *e*-beam evaporator. μCP with hexadecanethiol formed a self-assembled monolayer in the pattern of the stamp. Wet chemical etching removed silver not derivatized in the printing step. Immersion in 1% hydrofluoric acid (HF) solution removed exposed titanium. Electroplating of silver formed rigid structures with thicknesses of 50–200 μm. A free-standing structure resulted after dissolving the glass capillary in HF.

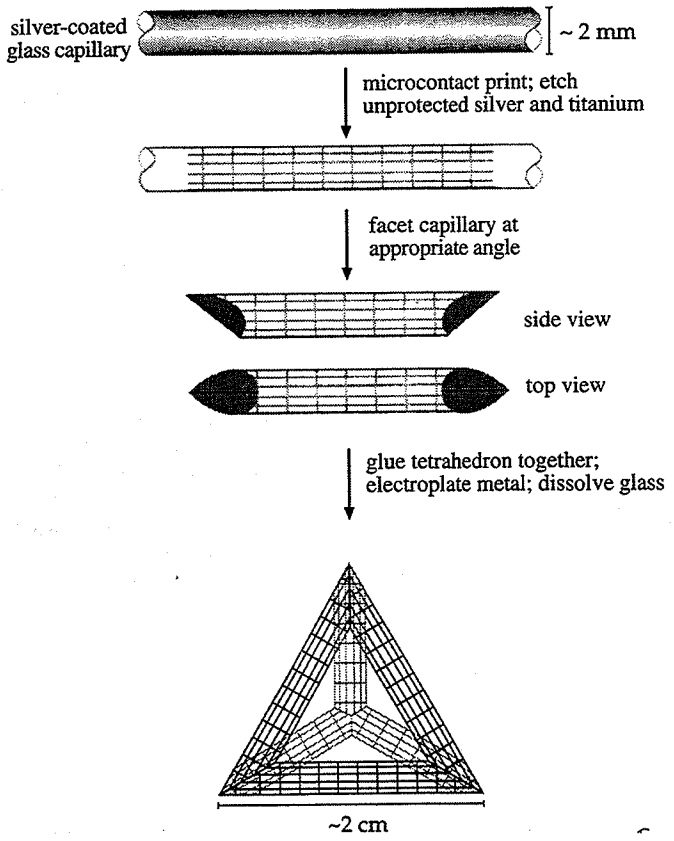

Fig. 2. Scheme for fabricating a tetrahedron and other 3-D structures. μCP, followed by wet chemical etching, formed thin (~500-Å) silver mesh structures on glass capillaries. Polishing the ends of the capillaries to the appropriate faceting and interface angles, using a faceting machine, produced pieces that would fit together to form the vertices of a tetrahedron. While holding the pieces in position using a PDMS mold, we glued them together using epoxy. Spots of silver paint were used to ensure electrical conductivity throughout the structure. Electroplating of silver formed rigid 3-D structures with thicknesses of 50–200 μm. A free-standing structure resulted after dissolving the glass capillaries in HF.

of the printer. Using the transparencies as photomasks, we perform photolithography to produce the masters.

B. Fabrication of Patterned Cylindrical Meshes

Fig. 1 illustrates schematically the fabrication of patterned cylindrical meshes. After preparing the surfaces of glass capillaries (Pyrex, diameter ~1.7 mm) by cleaning in a piranha solution [concentrated H_2SO_4 : 30% H_2O_2 (3 : 1)—caution: piranha solution can react violently with organics and should be handled with care], we coated them with titanium (~25 Å, adhesion promoter) and silver (~500 Å) by *e*-beam evaporation. Mounting the capillaries on two orthogonally rotating stages in the *e*-beam evaporator enabled them to be coated around their entire circumference [33].

Using an elastomeric stamp, we printed patterns of hexadecanethiol around the capillary. A set of translation and rotation stages allowed control over the orientation of the stamp to the capillary, the pressure applied during printing, and the duration of printing [24]. Placing the capillary in an aqueous ferricyanide bath [0.001 M $K_4Fe(CN)_6$, 0.01 M $K_3Fe(CN)_6$, and 0.1 M $Na_2S_2O_3$] for 15–30 s removed silver not derivatized by the μCP step [29]. Immersion of the capillary in a 1% HF solution for ~10 s removed the exposed titanium and left a thin continuous metal structure on the surface of the glass.

To increase the rigidity of the structure, we electroplated silver (Techni-Silver E2, Technic, Inc., Providence, RI) onto it. Conducting epoxy ensured electrical contact between the metal pattern and a clip. Electroplating at a current density of ~20 mA/cm^2 for ~2 h at room temperature formed structures with thicknesses of 50–200 μm. We estimated the area of the pattern by measuring the total area spanned by the pattern and then by calculating, based on the design of the pattern, the percentage of the area covered by metal. Dissolution of the capillary in concentrated HF released a patterned silver cylindrical microstructure (caution: the direct exposure of skin to concentrated HF can result in damage to skin and bones).

C. Fabrication of Fully 3-D Structures

Patterned cylinders formed the basis for the construction of 3-D structures (Fig. 2). μCP, followed by wet chemical etching, resulted in the formation of thin metal patterns on glass capillaries. To form a 3-D structure, we polished the patterned capillaries using a faceting machine (Graves Mark IV Faceting Machine, Graves, Pomano Beach, FL) producing beveled ends that formed a clean joint when brought together. For a tetrahedron, the faceting angle (the angle of polishing relative to the normal of the long axis of the capillary) was set to 54.8°, and the interface angle (the angle between the polished faces) was 120°.

A tetrahedral mold, formed by casting PDMS against a four-sided die, held the faceted patterned capillaries in the appropriate orientation while they were glued together with epoxy. Small drops of conducting epoxy ensured electrical conductivity between all parts of the tetrahedron. Electroplated silver (~100 μm at a current density of 20 mA/cm^2) increased the rigidity of the structure and electrochemically welded the components together. Concentrated HF was used to dissolve the glass capillaries and to release the 3-D microstructure.

D. Fabrication of a 3-D Tetrahedral Cage

By *e*-beam evaporation, we coated silicon wafers with titanium (~25 Å, adhesion promotor) and silver (~500 Å). The silicon wafers had been heated at 1100°C overnight to grow a layer of silicon dioxide (200–400 nm) to ensure that they were insulating. An elastomeric stamp transferred a monolayer of hexadecanethiol onto the silver in the pattern of the two-dimensional (2-D) representation of the tetrahedral cage (Fig. 3). Placing the wafer in an aqueous ferricyanide bath for 30–40 s etched the underivatized silver. We isolated the pattern electrically by removing the exposed titanium in a 1% HF solution for ~10 s.

Conducting epoxy made an electrical connection to the silver pattern at several points along its perimeter for the electroplating step. The rigidity of the structure increased as we electroplated nickel (Techni-Nickel "S," Technic, Inc., Providence, RI) onto the patterned structure. By electroplating at a current density of ~20 mA/cm^2 for ~30 min at ~45°C, we increased the thickness of the structure to 30–50 μm. Dissolution of the underlying silicon dioxide layer in a 12% HF solution freed the nickel structure from the rigid support. We assembled the 2-D components of the structure into a 3-D structure, locking it together along the edges manually with tabs and slots. Electroplating nickel at a current density of ~20 mA/cm^2 for ~2 h welded the edges together electrochemically. The final thicknesses of the features were 100–200 μm.

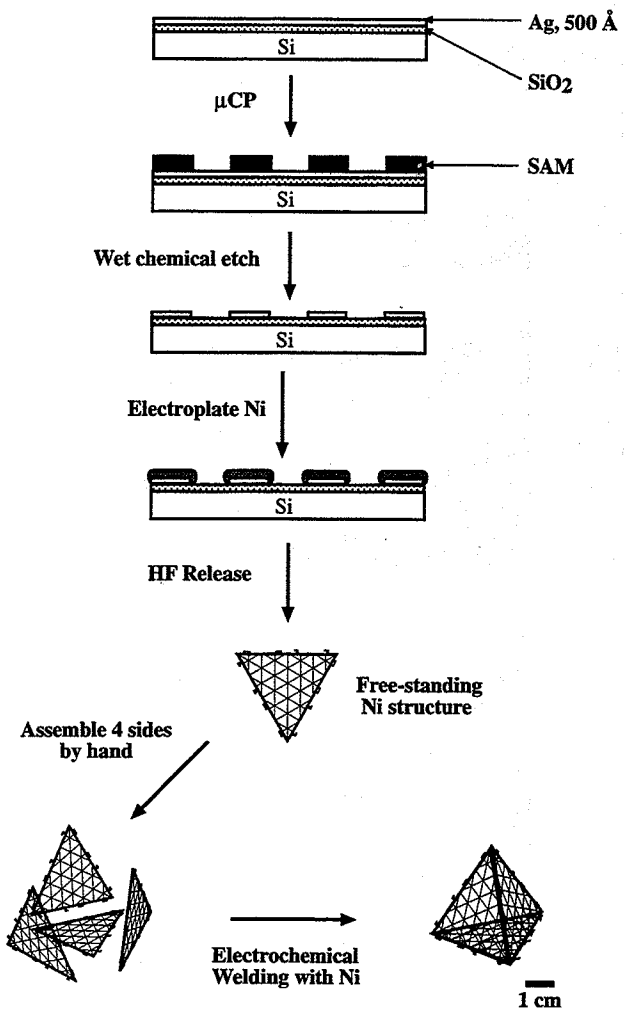

Fig. 3. Scheme for fabricating 3-D tetrahedral cage using μCP, electroplating, and assembly. Silicon wafers having a 200–400-nm thermally grown layer of silicon dioxide were coated with titanium (~25 Å) and silver (~500 Å). μCP with hexadecanethiol formed a self-assembled monolayer in the pattern of the 2-D projection of the tetrahedron. Wet chemical etching removed silver not derivatized in the printing step. Immersion in 1% HF solution removed exposed titanium. Electroplating of nickel formed rigid structures with thicknesses of 50–100 μm. A free-standing structure resulted after dissolving the silicon dioxide layer in 12% aqueous HF. The 2-D components were assembled into a 3-D tetrahedron manually, and the edges were locked together with the tabs and slots. A sturdy structure resulted after electrochemically welding the edges together with a 50–100-μm layer of nickel.

III. Results and Discussion

Examples of lightweight cylindrical mesh structures, with hexagonal and diagonal patterns, formed by electroplating silver onto thin metal patterns formed on capillaries by μCP and then subsequent removal of the underlying support are shown in Fig. 4. This technique for forming cylindrical meshes has the advantage that it can be used to make complex 3-D structures from a wide variety of materials. Any metal or polymer that can be electroplated is appropriate.

These structures can be designed to deform when a stress is applied to them. Stretching a hollow tube patterned with a diagonal mesh [Fig. 4(c)] anisotropically by ~120% resulted in a tube with a diameter ~50% smaller than the original [Fig. 4(d)]. As Fig. 4(e) illustrates, bending along the length of the connecting wires, rather than bending specifically at the joints, gave rise to the contraction. In these systems, even if cracks are introduced by the deformation, they can be repaired by further electroplating.

Fig. 5 shows a tetrahedron formed by electrochemical welding of faceted patterned capillaries. Removal of the supporting glass mandrils released this free-standing 3-D structure. A complex 3-D structure, a nickel tetrahedral cage, assembled from its 2-D components is illustrated in Fig. 6. μCP of a 2-D projection of components of the tetrahedral cage onto a planar substrate followed by electroplating formed a structure that when released from the surface, could be locked together to form the cage. Further electroplating welded the structure together.

IV. Conclusions

Here, we have demonstrated two potentially low-cost methods that rely on μCP for the fabrication of complex 3-D

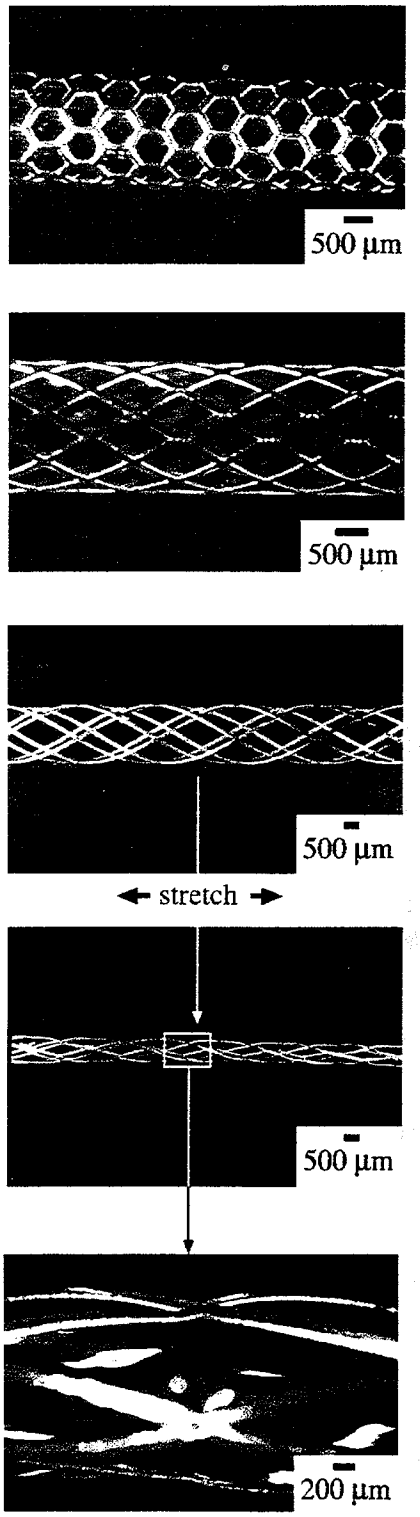

Fig. 4. Optical micrographs of mesh structures formed by electroplating metal onto thin metal patterns formed on glass capillaries ($d \sim 2$ mm) by μCP and subsequent removal of the underlying support. (a) Hexagonal mesh (line width $\sim$100 μm). (b) Diagonal mesh (line width $\sim$100 μm). (c) Unextended open mesh tube (line width $\sim$100 μm). (d) Open mesh tube shown in (c) after anisotropic extension of $\sim$120% along cylindrical axis. Diameter of tube is reduced by $\sim$50% after extension. (e) Detail of extended tube shown in (d). Bending along the length of the connecting wires, rather than bending specifically at the joints, gave rise to the contraction.

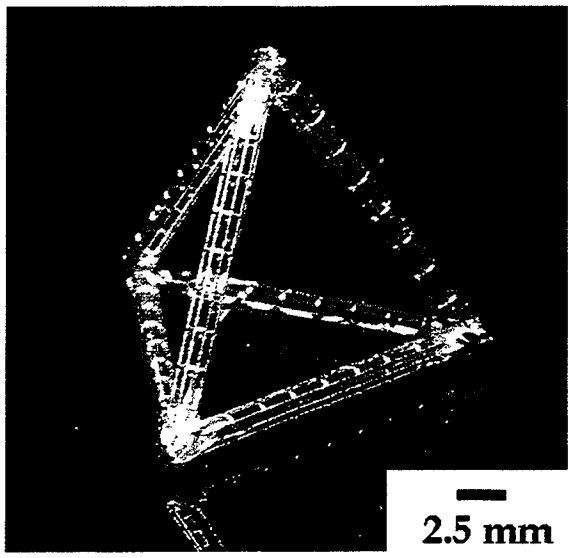

Fig. 5. Tetrahedral structure constructed by electrochemically welding together thin metal patterns formed on capillaries that have been faceted and glued in place. After electroplating, we dissolved the underlying glass supports.

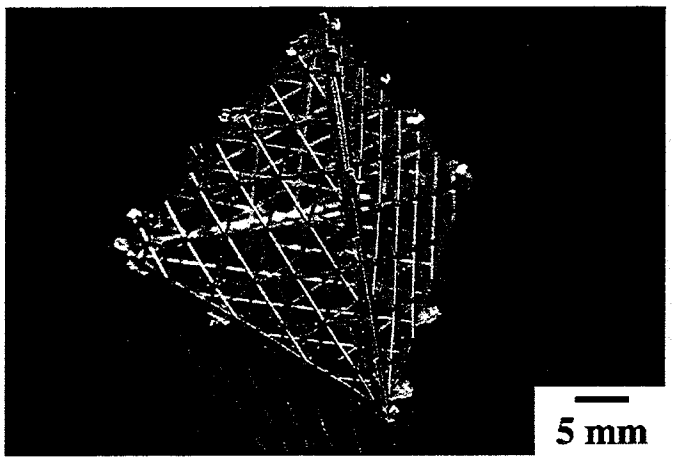

Fig. 6. Photograph of a 3-D tetrahedral cage formed by μCP on a planar substrate followed by electroplating and manual assembly.

structures. The first technique allows the formation of patterned meshes on cylindrical supports, but could potentially be extended to noncylindrical mandrils in a wide variety of materials. The second method can produce 3-D structures either by assembly from its 2-D components or by folding up its planar representation formed in metal on silicon wafers. It should, however, be possible to substitute a metallized polymer film for the silicon wafer. While the PDMS stamps used for printing are compliant, spatial errors in the size of the printed components are small: errors in printed features are typically $\pm$2% of the size of the features on the stamp. We have shown that errors in the registration of patterns produced by μCP can be limited to distortions on the order of $\sim$500 nm over square areas of $\sim$0.25 cm^2 for stamps similar to the ones used here [34]. We have observed that these distortions can be reduced to $\sim$500 nm over square areas of 1 cm^2 by using thin ($\sim$0.1-mm) stamps cast against rigid supports.

The set of rotation and translation stages with which we print should make it possible to align subsequent layers on a

first layer. Using a microscope similar to the one we typically use, it should be possible to align features to within 5 μm.

Both methods for the production of 3-D structures share limitations.

1) The electrodeposition step is not confined by a mold (as in through-mask electroplating or LIGA processes), and the growth of the plated structure is approximately isotropic. The exact dimensions of the final 3-D microstructure compared to the size of the pattern printed using the stamp is dependent on the final thickness of the 3-D structure. For example, a feature with an initial width of 25 μm will have a width on the order of 75 μm after plating this structure to a thickness of $\sim$25 μm. This step is where the primary source of difference in feature sizes between features on the stamp and the final structure arises. The change is, however, predictable and could be compensated for in the design of the stamp for printing. The isotropic growth of structures affords only limited control over the shape of metal features: straight side walls would be difficult to achieve.
2) A sacrificial support (either a mandril or a wafer) is required and is typically removed with HF (although other release layers or etchants may be used).
3) A lower limit on the size of the structures that can be formed is imposed by manual assembly.
4) The assembly of the final 3-D structure can be problematic—for the tetrahedron assembled from cylinders, imperfections in the faceting require small drops of conducting epoxy to ensure electrical conductivity. For the cage, holding the structure in place requires tabs and slots to keep the sides together. The folding method also requires that the final 3-D structure can be projected into 2-D without overlap of features. This problem can, however, be solved by using separate pieces to form the structure as we have used here.

Currently, line widths (and overall dimensions necessary to produce an open mesh) are limited by the dimensions of the masks that we have used. We have shown previously that submicron-sized features can be formed by μCP [22], [23]. There is then no reason to believe that it would not be possible to make these structures with finer features and smaller dimensions if a different mask, with features less than 20 μm, were used to generate stamps.

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Rebecca J. Jackman was born in Farnham, Surrey, U.K., in 1971. She received the B.Sc. degree in chemistry from McGill University, Montreal, Canada, in 1993. She is currently working toward the Ph.D. degree in chemistry at Harvard University, Cambridge, MA.

Her research interests include the use of self-assembly in the fabrication of 3-D microstructures and nanostructures for use in microelectromechanical, microoptomechanical, and microfluidic systems.

Scott T. Brittain was born in Gadsden, AL, in 1969. He received the B.S. degree in chemistry from the University of Alabama, Tuscaloosa, in 1995. He is currently working toward the Ph.D. degree in chemistry at Harvard University, Cambridge, MA.

His research combines organic materials with nontraditional methods of fabrication for the prototyping of components for use in microelectromechanical systems.

George M. Whitesides was born on August 3, 1939 in Louisville, KY. He received the A.B. degree from Harvard University, Cambridge, MA, in 1960 and the Ph.D. degree from the California Institute of Technology, Pasadena, in 1964.

He was a Member of the Faculty of the Massachusetts Institute of Technology, Cambridge, from 1963 to 1982. He joined the Department of Chemistry, Harvard University, in 1982 and was Department Chairman from 1986 to 1989. He is now Mallinckrodt Professor of Chemistry at Harvard University. His present research interests include biochemistry, surface chemistry, materials science, molecular virology, optics, self-assembly, and organic synthesis.