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## REVIEW

# Microfabricating Conjugated Polymer Actuators

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Conjugated polymer actuators can be operated in aqueous media, which makes them attractive for laboratories-on-a-chip and applications under physiological conditions. One of the most stable conjugated polymers under these conditions is polypyrrole, which can be patterned by means of standard photolithography. Polypyrrole-gold bilayer actuators that bend out of the plane of the wafer have been microfabricated in our laboratory. These can be used to move and position other microcomponents. Here we review the current status of these microactuators, outlining the methods used to fabricate them. We describe the devices that have been demonstrated as well as some potential future applications.

The miniaturization of electronic and optical devices has fueled the information technology revolution. During the past decade, a similar miniaturization has been going on for sensors and actuators for mechanical, chemical, and biological applications. The integrated gas chromatograph was an early example (1); today, an integrated analysis system for sample handling for biological characterization has recently been developed (2).

Microstructures promise to be of great importance for the coming biotechnology revolution. There is currently a tremendous increase in both academic and corporate research on micro-machined laboratories-on-a-chip, or micro-total analysis systems (3). These devices will find applications in areas such as genomic and proteomic studies, which will require extensive parallelism to allow many small simultaneous experiments. The integration of multiple experiments on a single carrier requires a miniature format. To minimize the chance of cross contamination when handling biological samples, a single-use device is preferred. Therefore, these devices should be disposable and thus be produced with inexpensive materials and patterning techniques. Polymers might be an option.

Polymers can be patterned by inexpensive

methods such as hot embossing and imprinting and therefore make attractive carrier materials. Imprint methods allow submicrometer patterning with dimensions smaller than 100 nm, as has been demonstrated with hard (4) and soft (5) imprint materials.

Polymers can also deliver active functions. Polymer surfaces can be chemically modified in a variety of ways, and this property is important in microstructures, which have a high surface-to-volume ratio. For example, surface-bound processes may be used to alter biomolecular function (6). Some polymers even allow the formation of electronic devices. Field effect transistors with useful carrier mobility have been made (7). Because thin polymer films may be easily prepared by spin coating, they can be integrated into functional systems. This capability may be important in the development of inexpensive, disposable chemical detectors for genomics and proteomics.

Handling, transport, separation, and detection of most biological species are done in liquids. Cells, organelles, and biomolecules are kept in buffer solutions or blood. The rapid handling of small sample volumes will require microfluidic technology. The movement of samples requires some driving force. One approach being pursued for fabricating small laboratories is to emboss compact discs (CDs) with microfluidic systems (8, 9). These devices use the inertial forces on the rotating CD to force liquids through channels. Another approach is to use microactuators to pump or redirect the samples (10). Microactuators

that are part of lab-on-a-chip systems may need to operate in biological environments and under liquid flow. The response time of these actuators should preferably be in the range of seconds or faster, depending on the specific application. Such actuators, when included in the chip, should preferably also be simple and inexpensive to produce and should be easily integrated with detector systems. Polymeric microactuators may be able to meet this need.

## Conjugated Polymer Actuators

We are currently developing polymer actuators for use in biomedicine and biotechnology. These devices are all based on conjugated polymers, which undergo volume changes during oxidation and reduction. The use of conjugated polymers as volume-changing materials began in the 1980s, primarily in bilayer devices (11–16). One layer of the bilayer was typically a passive substrate onto which the active conjugated polymer layer was applied.

Bilayers were initially used to determine the amount of volume change and to identify the volume change mechanism (16, 17). Bilayers provided a simple way to study classical conjugated polymers, such as polypyrrole, polyaniline (18, 19), and polythiophenes (20, 21). The active polymer layer was deposited onto a substrate, usually by electrochemical synthesis. Under electrochemical oxidation and reduction, volume change in the active layer forced the assembly to bend, and the direction and magnitude of volume change could be deduced from the direction and degree of bending. Such bilayer structures were typically a few centimeters long and a few millimeters wide. These studies showed that the volume change in the conjugated polymer is dominated by ionic movement into and out of the polymer.

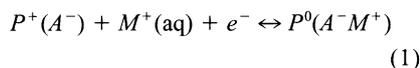
Other studies were done on single conducting polymer fibers submerged in a liquid electrolyte and connected to force- and elongation-measuring equipment. Speed of actuation, stress, and strain were measured, veri-

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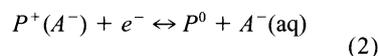
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fying the results from earlier bilayers studies (14, 15, 22, 23).

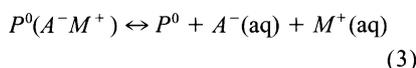
As mentioned above, the volume change of conjugated polymers is controlled by electrochemical processes, which cause ion insertion and deinsertion. There are two possibilities for ion flow. For a polymer ( $P$ ) doped with a large immobile anion  $A^-$  in contact with an electrolyte containing a small mobile cation  $M^+$



that is, cations are inserted and de-inserted. For a polymer doped with a small mobile anion in contact with an electrolyte containing both mobile cations and anions



that is, anions are inserted and de-inserted. In the former case, the volume expands in the reduced state of the polymer (a negative potential), and in the latter case, the volume typically expands in the oxidized state (a positive potential). In the latter case, there may be two moving species because not only reaction 2 occurs but reaction 1 can also occur, which can lead to a "twitching" behavior (17). For monotonic motion, it is preferred to have only one moving species. Another detrimental effect that can occur with mobile cations and anions is salt draining over extended times in the reduced state (16). Both types of ions leave the polymer



For our microactuators, we normally use polypyrrole (PPy) doped with the large immobile anion dodecylbenzene sulfonate (DBS) and an aqueous electrolyte of 0.1 M NaDBS.

The volume change is induced by changing the potential and thus altering the oxidation or reduction state of the conjugated polymer. Not only can we switch from completely oxidized or reduced states but we can also use intermediate states and thus achieve intermediate bending angles, both statically and dynamically.

To drive the actuators electrochemically, an ion source/sink is needed. Aqueous electrolytes have mostly been used, but polymer electrolytes have been demonstrated in macroactuators (24, 25). In these devices, the polymer electrolyte is sandwiched between two conjugated polymer layers, which generate the bending force. The use of polymer electrolytes enables operation in a normal laboratory environment instead of in a liquid electrolyte. Another way to achieve operation of electroactive polymer actuators in air is to encapsulate the complete device: the electroactive polymer layer, a hydrogel electrolyte, and the counter electrode (26).

Because ion transport is controlled by diffusion, we thought that the classical conjugated polymers would be unsuitable for macroscale actuators; thick layers would require too much time for operation. It may be possible to engineer the materials so that they contain pathways for ionic transport, but this approach would require novel materials. We attempted to use cross-linked conjugated polymer gel electroelastomers as actuators (27, 28); however, volume change in those materials was too large for use in bilayer structures, and their elastic modulus was too low. If thin layers of classic conjugated polymers were to be used, the speeds would be acceptable, but the forces would be small on the macroscopic scale.

On the microscopic scale, however, the forces would be large, so microactuators would be practical. We started with devices with millimeter dimensions and made Au/PPy bilayers on a silicon wafer that curled under electrochemical control (29). This work demonstrated that standard methods of photolithography could be used to prepare mini-actuators. It was possible to actuate the polymer and Au bilayers even if the polymer thickness was  $<1 \mu\text{m}$ , which is much thinner than previous actuators (150 to 200  $\mu\text{m}$ ) (30). The road to microstructures was thus open. In microsystems, such microactuators can be used as hinges from which can be built more complex systems such as self-assembling boxes (31), cell clinics (32), and microrobots (33).

Another approach to obtaining reasonable actuation speeds is to bundle thin fibers of conducting polymer in a solid electrolyte and operate many of these simultaneously to achieve macroscopically acceptable forces (34).

### Deposition and Patterning of Conjugated Polymers

To produce conjugated polymer microsystems, we use standard microfabrication procedures, including surface and bulk micromachining methods, which involve sequential deposition and removal (etching) steps. A number of review articles have been written about microfabricating these materials (35–37).

There are several methods for depositing films of conjugated polymers, including casting, dip coating, or spin coating the polymer from a solution. For many conducting polymers, this approach is limited by the insolubility of the polymer. A common route to synthesizing such polymers is through electropolymerization from a liquid electrolyte containing the monomer. Electropolymerization and deposition of conducting polymers occurs via monomer oxidation, monomer addition or dimerization, and the subsequent growth of oli-

gomers and polymers of the oxidized monomer (38). The chain precipitates out of solution and forms a deposit on the electrode surface where the initial oxidation occurred. The deposits are mainly localized to the site of polymerization, and this technique may therefore be used to prepare patterned layers of polymers on patterned electrodes. The polymer is deposited in the oxidized form, with compensating counter ions included from the electrolyte.

The lateral dimensions of the conjugated polymer components are determined by patterning. Standard photolithography is based on applying polymeric films—photoresist—on a surface to protect parts of this surface from etchants. The photoresist is patterned by ultraviolet (UV) light, which turns parts of the film soluble and parts insoluble. The soluble parts are then removed by appropriate solvents. However, some steps for photolithographic patterning may be damaging to conjugated polymers. Solvents can dissolve and swell the conjugated polymer films, some chemicals may attack their structure, and UV light may cause degradation. However, damage can often be avoided by an appropriate process sequence.

Methods that do not require photolithography and that combine deposition and patterning steps have recently been developed. One is soft lithography (5), which includes microinjection molding in capillaries (MIM-IC) and soft lithographic printing such as microcontact printing ( $\mu\text{CP}$ ) (39, 40). Deposition methods based on soft lithography can be categorized as subtractive or additive. Both  $\mu\text{CP}$  and MIMIC are additive methods and lead to in situ patterning, whereas "lift off" is a subtractive method in which the unwanted parts are lifted away (40). Other methods are deposition via ink jet printing (41, 42) and electrochemical atomic force microscopy (AFM) (43). For more complex devices, microfabrication methods can in principle be combined.

The lower limits of the lateral dimensions of our microactuators are currently enforced by our photolithographic equipment. The smallest actuators we have made were 10  $\mu\text{m}$  wide and 40  $\mu\text{m}$  long. Other patterning methods may reduce these dimensions; however, the scaling properties of the bending bilayers may become important and create other limitations.

The patterning of actuators must also take into account the fact that it is necessary to prepare the devices in a manner that will allow them to be operated at a later time. When micropatterned by photolithographic methods, the definition of features requires the layer to be immobile and planar. Upon actuation, this confinement must be broken to set the actuator free. It is sometimes possible to let the microactuators release themselves.

### Fabrication of Microactuators

For our microactuators, we deposit PPy (DBS) electrochemically onto Au-coated Si wafers from an aqueous electrolyte containing 0.1 M pyrrole and 0.1 M NaDBS (35). To initiate growth, a potential between 0.5 and

0.6 V versus Ag/AgCl is applied to the electrode. A higher potential results in faster deposition but also in nonuniform thickness. As a counter electrode, we use another Au-coated Si wafer, and as a reference electrode, an Ag/AgCl electrode. In situ patterning is pos-

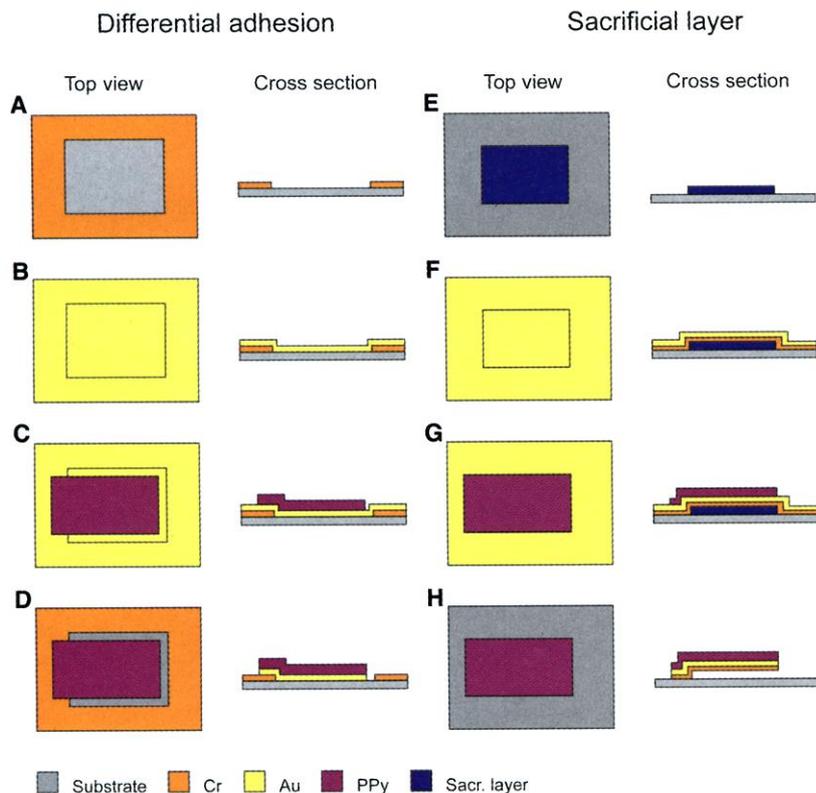
sible. The Au layer can be covered with a patterned photoresist layer that allows PPy to be deposited only on the exposed Au surfaces. After the PPy has been grown, the photoresist layer can be removed with ethanol, leaving a patterned PPy layer. Conjugated polymer layers can also be patterned after deposition by means of reactive ion etching in an  $O_2/CF_4$  plasma. The intended polymer structures are protected with a layer of photoresist.

For the release of PPy microactuators, we use two methods: differential adhesion or sacrificial layer (Fig. 1). The differential adhesion method (44) is based on the poor adhesion between Au and Si. A Cr layer on a Si surface is patterned, resulting in adhesive and nonadhesive areas. Over this surface, a Au layer is deposited, and PPy is electrochemically deposited as described above. The PPy is patterned so that a minor part of the Au/PPy bilayer is in contact with the adhesive Cr layer, and this part functions as an anchor holding the actuator to the surface. The major part of the bilayer is in contact with the Si, to which the Au does not adhere. Activating the bilayer causes it to pull itself free.

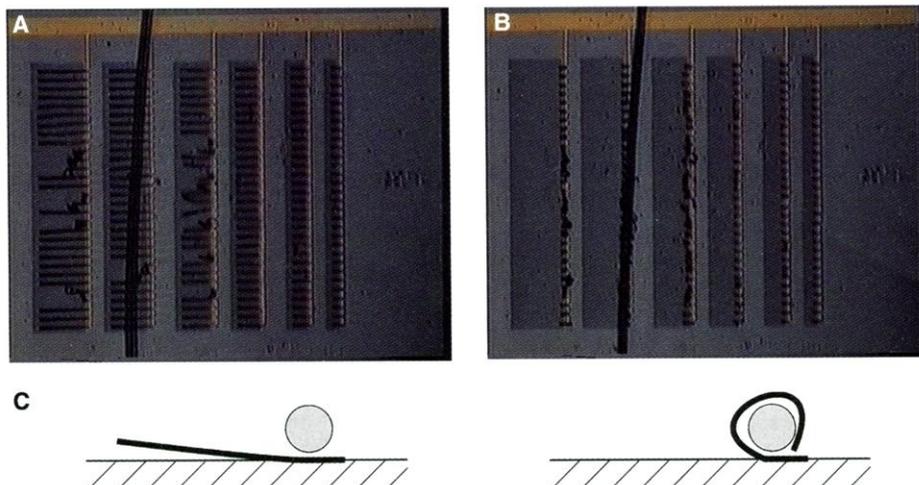
Although the differential adhesion method is easy and fast, it is not suited for all designs. For example, it cannot be used to fabricate some individually controlled microactuators. To make individually controlled actuators, we pattern the metallic layer of the microactuators into segments, one for each actuator. We then mechanically connect these segments by a resin layer [benzocyclobutene (BCB), a rigid, transparent conventional polymer similar to polyimide, which is often used in micromachining applications]. This step causes the resin layer to be in contact with the Si substrate, and because this resin is adhesive, the microactuators cannot release. Therefore, to fabricate micro-robot arms (33), we used the well-known sacrificial layer technique. On a  $SiO_2$ -covered wafer, a support layer of Ti (the sacrificial layer) was patterned. Next, we deposited Cr, Au, and PPy layers. The microactuators were patterned with a small part overlapping the Si as the anchor point and the rest overlapping the sacrificial layer. Removing the sacrificial layer by underetching resulted in a free-hanging bilayer that could be activated. The advantage of the sacrificial layer method is that one can build more complex microactuator devices. Disadvantages include a long underetching time and potential damage to the PPy, depending on the etchant.

### Operation of Conjugated Polymer Microactuators

To actuate the devices on the wafer, we typically use a Au-coated Si wafer counter electrode and a commercial Ag/AgCl reference



**Fig. 1.** A sketch of the fabrication schemes for the differential adhesion method (A to D) and the sacrificial layer method (E to H). (A) Deposition and patterning of the  $\sim 5$ -nm Cr adhesion layer. (B) Deposition of the structural Au layer ( $\sim 100$  nm). (C) Electrodeposition and patterning of the PPy layer ( $\sim 1$   $\mu\text{m}$ ). (D) Etching of the final microactuator structure by removal of the excess Au. (E) Deposition and patterning of the sacrificial layer ( $\sim 50$  nm). (F) Deposition of the Cr adhesion ( $\sim 5$  nm) and structural Au layer ( $\sim 100$  nm). (G) Electrodeposition and patterning of the PPy layer ( $\sim 1$   $\mu\text{m}$ ). (H) Etching of the final microactuator structure and underetching of the sacrificial layer.



**Fig. 2.** A photograph of arrays of six microactuators or "microfingers" with decreasing lengths. The microactuators were 20  $\mu\text{m}$  wide and 300, 250, 200, 150, 100, or 50  $\mu\text{m}$  long. (A) The actuators are stretched; in (B), they are curled up. We grabbed a 30- $\mu\text{m}$  fiber (black vertical line) using the second row of actuators, which were 250  $\mu\text{m}$  long. (C) A sketch of the experiment.

electrode (Bioanalytical Systems, West Lafayette, Indiana). For further miniaturization, we have fabricated on-chip microelectrodes (45). We made a 100  $\mu\text{m}$ -by-150  $\mu\text{m}$  Au counter electrode and a 50  $\mu\text{m}$ -by-100  $\mu\text{m}$  Ag/AgCl quasi-reference electrode. (It is a quasi-reference electrode because there are no  $\text{Cl}^-$  ions in the solution.) Actuation is typically done in aqueous 0.1 M NaDBS, although the actuators will work in virtually any aqueous salt solution, and we have operated them in blood plasma, urine, and cell culture medium. The pH of the solution should, however, be kept above  $\sim 3$  (46).

The performance of microfabricated bilayer actuators has been characterized (47). Force measurements on the PPy microactuators were performed by measuring the mass that the microactuators could reversibly lift. A set of standard weights was produced by cutting glass coverslips into pieces measuring 1  $\text{mm}^2$ . A weight was considered to be successfully lifted if the actuator could rotate

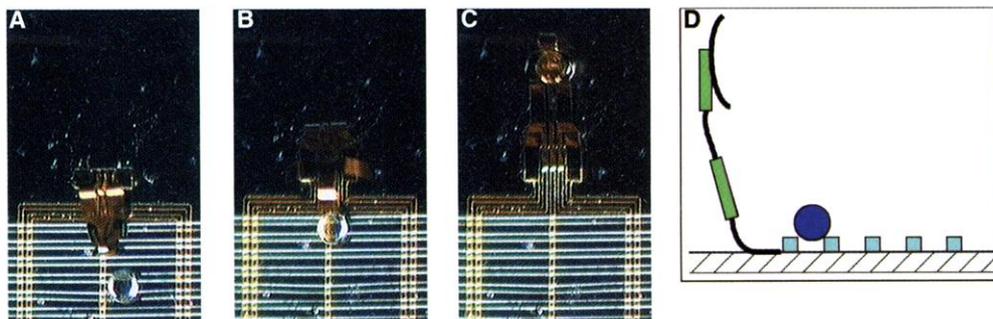
through 180° and back again with the load. For a device with a PPy thickness of 0.9  $\mu\text{m}$ , the ratio of the mass lifted to the mass of the hinge was 43,000:1. The reproducibility in open loop was quite good: The average standard error in bending angle for a given potential was only 0.6°. The radius of curvature was 5.6  $\mu\text{m}$ , and it took 2 s to bend or straighten a bilayer 1  $\mu\text{m}$  thick. Energy efficiencies were  $\sim 0.2\%$ .

The bilayers can be cycled for several hours (a few thousand cycles) before the Au and PPy layers delaminate, which is the primary cause of device failure. Very adhesive PPy films on Ti have been reported that use a different growth technique (48). Roughening the Au surface can also improve adhesion. Modification of the Au surface with self-assembled monolayers is ineffective for applications in which the PPy is electrochemically cycled (49).

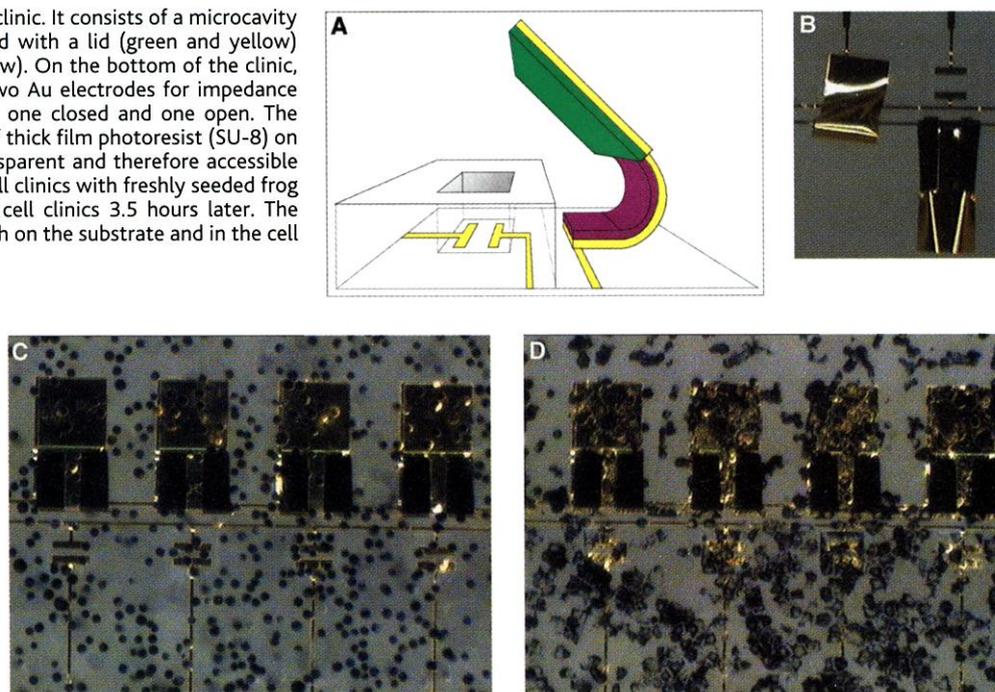
Macroscopic bilayer devices were initially used to measure volume change in conjugat-

ed polymers. The strain was calculated by Pei *et al.* (16, 50) to be 0.45 to 3.4% in PPy doped with various anions. However, bending is a response only to in-plane volume change. We have recently examined the out-of-plane volume change directly, in situ, using AFM and microfabrication (51). We patterned rectangles of PPy(DBS) on a Au-coated Si wafer and immersed the AFM head into the electrolyte during actuation. During each scan, the AFM tip went from the Au surface onto the PPy and back onto the Au, giving an absolute height reference. The  $y$  axis of the scan was disabled so that the tip traced the same path during each scan. The height of the PPy rectangle was measured as a function of applied potential. We found an out-of-plane expansion of  $>35\%$  in the reduced state, which shows that the PPy films are very anisotropic. This volume change is sufficiently large that it can in principle be exploited directly in microactuators that do not need to be released from the surface. Such devices

**Fig. 3.** A microrobot arm made of PPy microactuators. The microrobot arm consisted of an "elbow," a "wrist," and a "hand" with two to four "fingers," and was only 670  $\mu\text{m}$  long (measured from its base to the end of the fingers). Using the microrobot, we could grab, lift, and move a 100- $\mu\text{m}$  glass bead over a system of polyurethane tracks. (A) Bead is lying on track 4. (B) Bead has been moved to track 1. (C) Retracted arm holding the glass bead in its fingers. (D) Schematic picture of the setup. The distance between two adjacent tracks was 60  $\mu\text{m}$ , and the total displacement of the glass bead that could be achieved with the robot in this setup was  $\sim 270$   $\mu\text{m}$ .



**Fig. 4.** (A) A schematic picture of the cell clinic. It consists of a microcavity (100  $\mu\text{m}$  by 100  $\mu\text{m}$ ) that can be closed with a lid (green and yellow) activated by PPy hinges (purple and yellow). On the bottom of the clinic, sensors have been placed; in this case, two Au electrodes for impedance studies. (B) A picture of two cell clinics, one closed and one open. The cavity is defined in a 20- $\mu\text{m}$ -thick layer of thick film photoresist (SU-8) on a glass substrate, making the device transparent and therefore accessible for microscopy. (C) An array of opened cell clinics with freshly seeded frog (*X. laevis*) melanophores. (D) The same cell clinics 3.5 hours later. The melanophores have spread out nicely, both on the substrate and in the cell clinics.



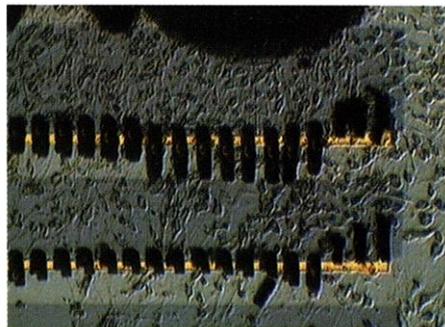
could be of great use as micropumps and valves.

### Devices

The simplest devices are bilayer strips of PPy/Au. These can be used to grab small objects that have a geometry suitable for establishing mechanical contact. They may also be used to establish electrical contact with the object seized, if properly designed (Fig. 2). For example, the cylindrical object in Fig. 2 could be a nerve or nerve fiber.

Adding more micromachined elements leads to more complex and interesting structures. Plates can be added to the ends of the bilayers so that they act as hinges. We have lifted BCB/Au plates (31). Because these can be rotated by 180°, they can potentially be used as lids for microvials or to change the nature of the surface by exposing one or the other side of a flap. Combined in series, self-opening and -closing boxes have been realized (31).

Recently we have fabricated and operated microrobots (33). We linked together microactuators with stiff elements formed from BCB to form a "robot arm" consisting of an "elbow," a "wrist," and a "hand" with two to four "fingers." The arm is only 670  $\mu\text{m}$  long, measured from its base to the end of the fingers. Using MIMIC, we built a track system on the  $\text{SiO}_2$  substrate. With this robot arm, we could pick up, lift, and move a 100- $\mu\text{m}$  glass bead over the surface from track to track, as if on a conveyor belt (Fig. 3). Instead of inanimate glass beads, we envision these arms working in parallel to transfer cells in a lab-on-a-chip. In another design, we have placed two connected hinges perpendicular to each other. We used the first hinge to rotate the second actuator perpendicular to the surface, and then the second was activated to move in plane, perpendicular to the surface (52). By intelligently combining hinge elements, new activation schemes in three-dimensional space are possible.



**Fig. 5.** The microactuators can be used in cell culture medium. A photograph of nerve cells (rat dorsal root ganglia) that were cultured for 1 week with arrays of curled-up microactuators. The microactuators were 20  $\mu\text{m}$  wide and 100  $\mu\text{m}$  (bottom row) or 150  $\mu\text{m}$  (top row) long.

dimensional space are possible.

It is possible to use the microactuators as hinges to move plates (31). These plates could then be used to close and reopen microcavities that contained drugs or cells (32). These sealable microcavities might also be used as small vessels for nano- and picoliter chemistry. We are now building such devices with microfabricated sensors and actuators inside these cavities to extend the functionality. We load these devices with cells and study the cell behavior. We call them cell clinics, and they should be suitable for use in studies of single cells. Frog (*Xenopus laevis*) melanophores were added to a cell clinic in this study; cells then spread on the surface and covered the electrodes at the cell clinic bottom (Fig. 4). We are currently evaluating impedance measurement data.

The microactuators can also be used as tools for fundamental cell biology studies. One such tool under development in collaboration with cell biologists is what we call a cell tapper. This is a microactuator designed to tap on a single cell. We want to use this cell tapper to study the effects of mechanical stimulation on single cells. Microactuators for mechanical stimulation to study single-cell properties have been demonstrated by others. For instance, a microactuator can be used to hold a single myocyte and measure the forces that the cell exerts when chemically stimulated (53). In addition, micromachined springs were attached to single endothelial cells, and the focal points of adhesion to the substrate were studied (54). [We have already shown that it is possible to culture nerve cells (such as rat dorsal root ganglia) on devices containing PPy microactuators (Fig. 5).]

The movement of actuators may be used to block the flow of liquids (55). Inside a channel, an actuator is operated to open and close the entrance to microchannels in microfluidic devices operating with an aqueous electrolyte. These microfluidic devices were built from elastomer slabs assembled on the Si substrate that carried the microvalves. These systems may also come to include micromachined sensors for analysis, located downstream in the microfluidic system. Another way to block liquid flow with polymer-based valves is to use pH-sensitive polyelectrolyte gels fabricated in the form of posts at the entrance to a microfluidic channel; the swelling of the post blocks the channel entry (56).

We have also used bilayer hinges to lift Si plates bulk-micromachined from the wafer using reactive ion etching (47). More functionality can be added on the silicon plates, such as electrochromic elements (57), because it is possible to deliver power to devices on the moving plate via an electrode. This example shows that one may potentially move

and position anything that can be fabricated on silicon, including circuitry, micromachined silicon devices, sensors, and light sources. Videos of the above-mentioned devices in action can be seen at our home page (58).

### Outlook for the Future

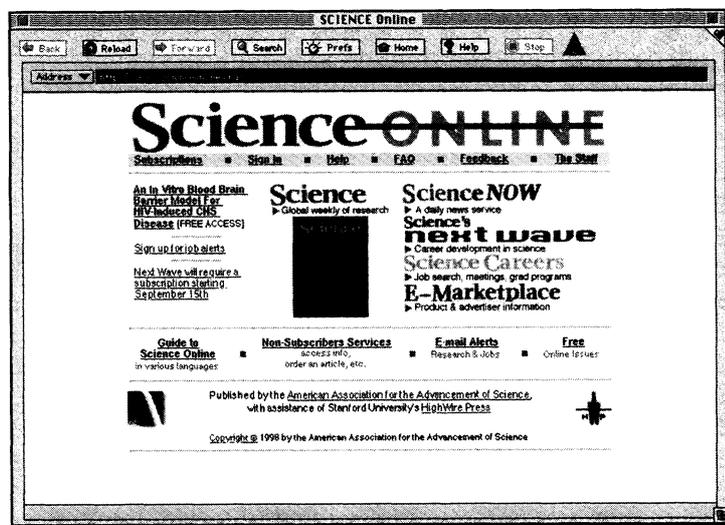
PPy-based microactuators are an exciting technology with many possibilities for applications, particularly in cell biology and biomedicine because these actuators can work in physiological media. They have been shown to work in various salt solutions, blood plasma, urine, and cell culture medium. Contacts with scientists of many disciplines, ranging from microbiology to combinatorial analysis, unravel possible new applications. We believe that the uses of these microactuators are manifold, and many are currently unsuspected.

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