Letter

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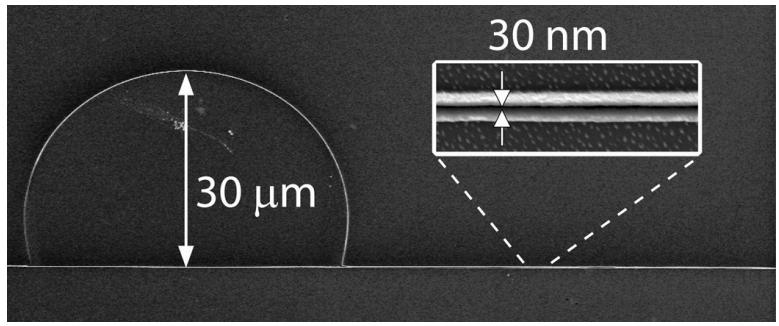

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Electrically Addressable Parallel Nanowires with 30 nm Spacing from Micromolding and Nanoskiving

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ABSTRACT

This paper describes the fabrication of arrays of parallel, electrically addressable metallic nanowires by depositing alternating layers of thin films of metal and polymer—both planar and topographically patterned—and sectioning the laminated structures with an ultramicrotome (nanoskiving). The structures that resulted from this process had two distinct regions: one in which parallel Au nanowires were separated by a minimum distance of 30 nm, and one in which the nanowires diverged such that the distal ends were individually addressable by low-resolution (≥10 μm) photolithography. Conductive polyaniline (PANI) was electrochemically deposited across the nanowire electrodes to demonstrate their electrical addressability, continuity, and physical separation. Before deposition, the wires were electrically isolated; with the PANI, they were electrically connected. After dry etching to remove the polymer, the gap between the nanowire electrodes returned to an insulating state. This procedure provides a method for making wires with dimensions and separations of <50 nm without the use of e-beam or focused-ion-beam "writing" and opens applications in organic and molecular electronics, chemical and biological sensing, and other fields where nanoscale distances between parallel conductive electrodes are desirable.

This paper describes the use of nanoskiving,[1−4] a technique that uses an ultramicrotome to section thin films of metal embedded in a polymer matrix, to fabricate parallel nanowires with controlled spacing (gaps as narrow as 30 nm) whose distal ends diverge in a way that makes it possible to address each wire electrically using low resolution (≥10 μm) photolithography. The process can be used to fabricate two or three (or more) nanowires that are parallel over distances of ∼50 μm and comprises five basic steps: (i) deposition of a thin (<100 nm) metal film on a flat polymeric substrate; (ii) deposition of a thin polymer film onto the metal by spin-coating; (iii) molding microscale parallel lines of polymer on top of the composite structure; (iv) shadow evaporation of a thin metal film on the composite structure; and (v) nanoskiving (see Figure 1 for an overview of the process; a detailed description of it follows). The geometries of the structures accessible by this technique resemble those that are ordinarily made using significantly more sophisticated, expensive, and slower techniques (such as electron- and focused-ion-beam (FIB) lithography) than those used in this study. The technique is particularly useful for manufacturing indistinguishable copies (thousands per hour, in principle) of addressable electrodes for the characterization of electronic materials and the fabrication of devices that rely on charge transport over nanoscale dimensions (e.g., sensors, capacitors, resistors, photovoltaics, transistors, diodes, and molecular junctions). The use of low-resolution photolithography makes the method accessible to general users who do not have access to high-resolution, direct-write techniques, or who wish to use materials that are not allowed in a cleanroom dedicated to solid-state electronics.

Background. Nanowire electrodes that are separated by small (<100 nm) gaps and are electrically addressable individually are useful in sensors,[5,6] as electrodes for dielectrophoresis (used to entrap nanostructures and molecules[7,8]) and electrochemistry,[9,10] in molecular junctions,[11,12] and as test-bed structures for studying nanoscale phenomena or new nanoscale architectures.[13]

Electrical characterization of nanostructures and their incorporation into functional devices depends on the formation of stable electrical contacts between the nanostructures and external electrical circuits; most applications require a minimum of two electrodes for simple electronic function. It is challenging to address multiple individual nanowires electrically that are in close proximity (e.g., in parallel) and separated by a nanoscale gap (<100 nm) without inadvertently contacting multiple wires with a single contact pad.

There are only a small number of techniques capable of generating nanowire electrodes with nanoscale separation that are easy to address electrically. Of these techniques, few combine useful levels of generality and simplicity. "Direct-write" approaches to nanofabrication, most prominently,

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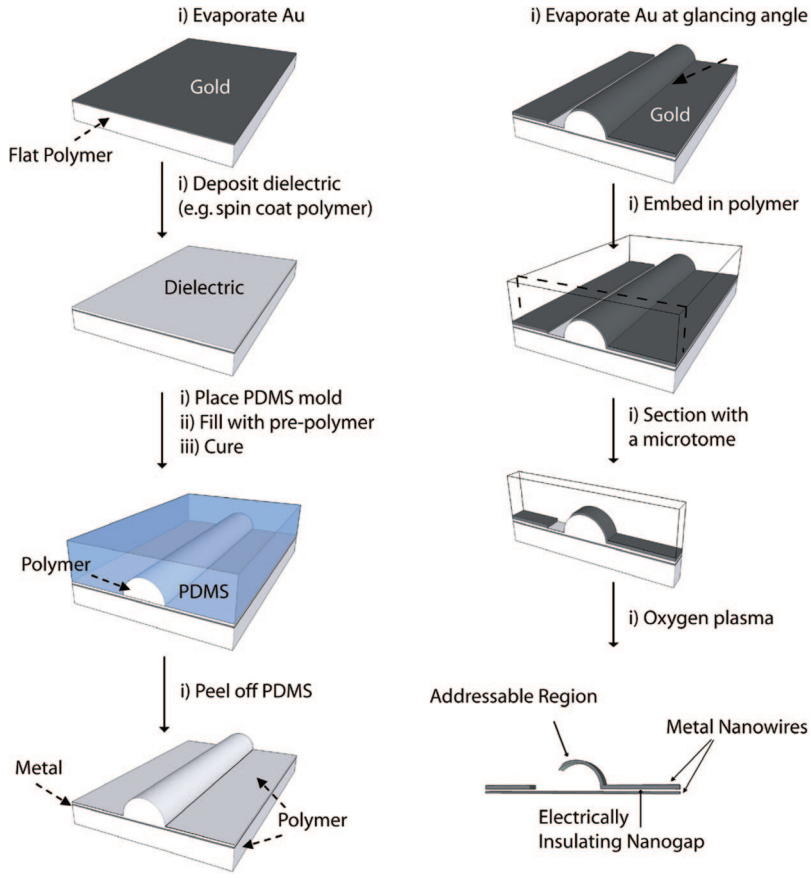

Figure 1. Schematic representation of the procedure used for the fabrication of electrically addressable nanowires. Spin-coating produces a thin-film of polymer on top of a flat polymer substrate coated with gold. A microchannel defined in PDMS is placed onto the substrate and is filled with prepolymer that is then cured with UV light. The PDMS is removed and a thin layer of gold is deposited onto the substrate by evaporation. Embedding the substrate in polymer and slicing it with an ultramicrotome creates parallel nanowires with addressable regions. The polymer can be removed by an oxygen plasma.

e-beam and FIB lithography, are capable of forming addressable nanowires but are expensive, technically challenging, damaging to organic materials, and limited to rigid planar substrates.[14,15] Addressing parallel nanowires using these techniques becomes increasingly difficult as the spacing between the wires decreases, and it is physically impossible when the spacing is smaller than the resolution of the writer (~10 nm).

Photolithography is, by far, the most common method of pattern replication in the semiconductor industry and in research laboratories. State-of-the-art tools for photolithography are capable of patterning sub-100 nm contacts with precise alignment, but most research laboratories are limited by equipment that has relatively low resolution (~1 μm) and alignment precision. In practice, it is nearly impossible in an academic laboratory to align a contact pad using photolithography such that the pad only contacts one structure that is in close proximity (<100 nm) to a neighboring one.

Nonlithographic methods that have been developed to make addressable, narrow junctions between electrodes include shadow mask evaporation,[16] mechanical break junction techniques,[17] local oxidative cutting of carbon nanotubes,[18] electromigration,[19] and on-wire lithography.[20] In general, these methods are restricted to two electrodes with limited geometrical configurations (typically, nanoscale breaks in a single wire), and in some cases the techniques are challenging experimentally. Nonlithographic methods generally are not practical for creating addressable parallel nanowires with separations close to the thickness of the wires.

Nanoskiving. The goal of this work was to develop a simple method to produce nanowires in close proximity that can be addressed individually without the use of direct-write techniques. "Nanoskiving" is the use of an ultramicrotome to generate nanostructures from planar or topographically patterned thin films.[2–4] The technique is attractive as an approach for nanofabrication because of its simplicity—nanoskiving converts features that are thin in the vertical dimension (thin films) to features that are thin in the lateral dimension ("edge lithography").[21] Our laboratory has used nanoskiving to fabricate arrays of metallic nanostructures for optical applications, as well as nanowires of conjugated polymers for electronic and optoelectronic applications.[22–24] We chose to use nanoskiving because it is capable of forming nanowires without the use of sophisticated lithographic techniques; nanoskiving offers a means of producing uniform nanostructures reproducibly and simply. It is easily capable of producing multiple indistinguishable copies (that is, consecutively cross-sectioned slabs) of a parent structure. We sought to produce parallel nanowires whose lateral arrangement with respect to each other would have two distinct

regions: one in which parallel nanowires were separated by a small (<100 nm wide), defined gap, and another in which the wires diverged until they were separated by a large enough spacing (>10 μm) to be addressed individually by contact photolithography.

Choice of Materials. Nanoskiving takes advantage of the precise control of thickness provided by techniques used for thin-film deposition. We utilized e-beam evaporation to deposit the Au that would ultimately become the wires, and spin-coating to define the dielectric spacer layer between the wires. We chose Au because it is easy to deposit, sections well, and does not oxidize. For the spacer layer (in the parallel region), we utilized a photocurable prepolymer whose thickness was defined by spin-coating.

Micromolding in Capillaries. To create the addressable region of the structure, we used micromolding in capillaries (MIMIC).[25] MIMIC is a process that forms structures on a substrate by drawing a fluid into an elastomeric mold. We filled a mold with optical adhesive prepolymer, cured the fluid, and removed the mold to produce topographic features that were an inverse replica of the mold. The use of polydimethylsiloxane (PDMS) ensured conformal contact of the mold with the substrate during the filling process and facile removal of the mold from the substrate due to the low surface energy of PDMS. We fabricated the molds using soft lithographic techniques.[26,27] We used a commercially available optical adhesive (Norland Optical Adhesive 61) as the embedding material for nanoskiving because it contains thiol groups that promote adhesion to the Au, it cures rapidly (<5 min) by exposure to UV radiation, it is inexpensive, and it sections well in the ultramicrotome (it displays minimal compression or other distortion during cutting).

Fabrication. Figure 1 summarizes the process used for fabrication. We evaporated 40−70 nm of Au onto a flat piece of cured optical adhesive; this layer of gold ultimately became one of the nanowires. Onto the gold, we spin-coated a thin film of a prepolymer solution containing 2.5 wt % of a 1:1 (w/w) mixture of a multifunctional thiol (pentaerythritol tetrakis(3-mercaptopropionate)) and an acrylate (di(trimethylolpropane)tetraacrylate) diluted in solvent (propylene glycol methyl ether acetate, PGMEA). This layer could be removed (as a sacrificial material) after sectioning by etching in an oxygen plasma or could serve as a functional material (as a dielectric) between the two nanowires. We used this material because optical adhesive, which serves as the embedding material for microtoming, does not form smooth films at <100 nm thickness.

We filled microfluidic channels in a PDMS mold with optical adhesive and cured the adhesive with UV light. The cross-sectional shape of the channels would ultimately define the shape of the addressable region of the wires. We found that smooth, semicylindrical features sectioned better than those with sharp, rectangular features, presumably because of the way in which mechanical stress is distributed in rounded features during sectioning. We fabricated the PDMS channels by two methods: (i) replica molding lines of positive photoresist (50 μm wide, 30 μm tall lines of AZ P4903 on a 100 μm pitch), then reflowing the resist at 130 °C for 1 h to round the tops of the features, and (ii) replica molding lines of negative photoresist (50 μm wide, 30 μm tall lines of SU-8 resist on a 100 μm pitch), then spin-coating and curing an additional 10 μm thick layer of SU-8 over the substrate to round the features. After removing the PDMS mold, we evaporated 40−70 nm of Au onto the substrate at a glancing angle (~60° angle between the beam and the substrate) to cover the region of the substrate between the molded features and one of the two sets of sidewalls of the molded features.

We cut the substrate with a razor blade into small strips (~2 mm wide) parallel to the long axes of the ridges defined by the MIMIC process. We placed the strips into polyethylene molds (Electron Microscopy Sciences), covered them in optical adhesive, and cured the adhesive with a mercury lamp. We trimmed the face of the resulting block (the face is perpendicular to both the gold film and the lines defined by MIMIC, see Figure 1) with a razor blade to an area of ~1 mm × 1 mm and then sliced thin sections of the sample using an ultramicrotome (Leica Ultracut UCT) fitted with a diamond knife (Diatome™ Ultra 35°). The sectioning speed (i.e., the speed of the knife through the sample block) was 1 mm·s^{-1} for a selected section thickness (typically 70 nm). The sectioned slices of polymer containing the embedded nanostructures floated on the surface of the water in the reservoir of the knife. We transferred the sections (using the Perfect Loop tool, Electron Microscopy Sciences) to the surfaces of Si wafers bearing ~600 nm layers of thermally grown oxide that prevented electrical shorting of the nanowires through the substrate.

We placed the samples on a hot plate at 150 °C to improve the adhesion of the section to the substrate. A 10 min exposure to oxygen plasma (0.2 Torr, 150 W barrel etcher) removed the embedding material from the sections, while the metal nanowires remained on the insulating substrate. We addressed the wires by contact lithography and a typical lift-off process. Briefly, we spin-coated a thin (~3 μm) layer of Shipley 1822 onto the substrate, and baked the photoresist for 3 min at 110 °C. After cooling the substrate to room temperature, we aligned the contact pads on the photomask with the addressable regions of the wire and irradiated the photoresist (ABM mask aligner, ~70 W). Developing (in CD-30) for 30 s produced a pattern onto which we deposited 4 nm of Ti and 40 nm of Au. Acetone removed the resist and extra metal (without sonication), leaving behind Au electrodes on the individual nanowires.

Before electrical characterization, we secured Cu wires to the Au contact pads with drops of graphite ink (Ercon 3456). After drying the ink, we covered the graphite and exposed regions of the Cu wire in five-minute epoxy. We characterized the wires electronically using a Keithley 6430 subfemtoammeter. We deposited polyaniline electrochemically from an aqueous solution of 0.1 M aniline (freshly distilled at 90 °C under reduced pressure), 0.5 M Na_2SO_4, and 0.1 M H_2SO_4 (pH ~ 1). We used a bipotentiostat (Pine, AFCBP1) to electropolymerize the aniline at +0.8 V versus Ag/AgCl with platinum foil serving as the counterelectrode.

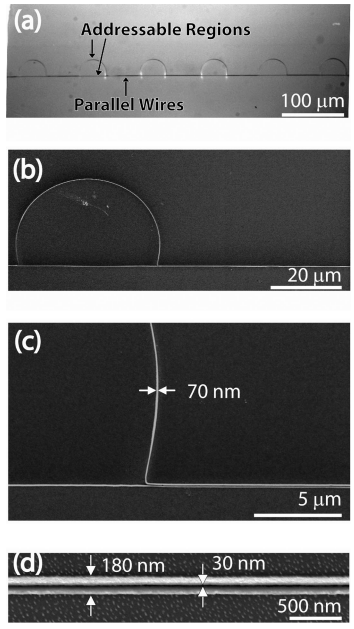

Figure 2. Images of electrically addressable Au nanowires. (a) Optical micrograph of a slab of polymer containing six addressable structures. (b) SEM overview of a single addressable structure. (c) Close-up of structure reveals two parallel nanowires that diverge. The point of divergence corresponds to the semicylinder of optical adhesive formed using MIMIC. (d) Close-up image of two parallel gold nanowires separated by a narrow gap (~30 nm).

Results and Discussion. Figure 2 is a series of images of electrically addressable gold nanowires on a Si/SiO_2 substrate. Figure 2a is an optical micrograph of a polymeric slab that contains six embedded addressable structures. Figure 2b is a scanning electron micrograph (SEM) of a single addressable structure after removal of the polymer slab by oxygen plasma. The structure consists of a parallel region where two wires (~70 nm wide) are separated by ~30 nm, and a diverging, addressable region where the wires are spaced by ~30 μm (i.e., approximately 3 orders of magnitude greater spacing than the parallel region). The parallel region in Figure 2c is ~50 μm long. We produced parallel regions as long as ~150 μm, but in principle the parallel region could be as long as the width of the slab (~1 mm).

The cross-sectional profile of the microfluidic channel used during the MIMIC step defines the 2D geometry of the addressable region of the nanowire electrodes. We found that rectangular addressable regions tended to have discontinuities (breaks) in the wires primarily along portions of the wires perpendicular to the edge of the diamond knife (i.e., parallel to the direction of cutting). We believe that a wire is subject to compression and breakage when it is aligned parallel to the direction of cutting because of differences in the mechanical properties of the gold and the embedding polymer. We tested this hypothesis by observing the effect of the orientation of a sample (consisting of a thin gold film embedded in polymer) during cutting; wires oriented parallel to the direction of the sectioning had significantly more damage and breaks than those formed by cutting perpendicular to the wires. Sectioning at an intermediate angle (e.g., a 45° angle between the wire and the blade) also reduced the amount of total damage to the wires, but we still observed some breakage in the longer portions of the wires. On the basis of these observations, we aligned the knife perpendicular to the parallel region of the wires and chose to use rounded addressable regions (the addressable region in Figure 2 is effectively a semicircle) to minimize the portion of the wires aligned parallel to the direction of cutting. Other designs of the addressable region are discussed in the Supporting Information.

The yield of a representative batch of 49 pairs of addressable, parallel nanowires was 73%; this value included only those structures that were both (i) spatially isolated (individually) and (ii) physically continuous (independently) along the entire length of either nanowire (~100 μm). Independent experiments indicated that nanowires that appeared physically continuous by SEM analysis were also electrically continuous. (See Supporting Information for a demonstration of electrical continuity along single nanowires.) The remaining 27% of structures contained defects such as shorts (where the two wires touched) and breaks. We attribute the defects to damage to the nanostructures by defects in the knife blade, delamination of the Au films from the polymeric matrix during the sectioning process, and other stresses of cutting (see Supporting Information for descriptions of the defects).[28] An individual epoxy section is typically ~1 mm wide, and an individual structure (Figure 2a) is approximately 100 μm long; we can therefore produce 6−10 addressable samples (of the type shown in Figure 2a) per section.

Electrical Characterization. We electrically addressed the parallel wires by standard contact lithographic and lift-off procedures. A simple experiment demonstrated the connectivity of our lithographically defined contact pads to the nanowires, the continuity of the nanowires, and the physical separation of the nanowires. We potentiostatically deposited a conductive polymer, polyaniline (PANI), in the gap between the nanowires (Figure 3a). Electrodeposition of conductive polymers in the trenches of hard substrates (defined by e-beam lithography) has been used to fabricate polymeric nanowires; these nanowires behave as high-surface-area chemical and biological sensors.[5,6,29−31] The gold nanowires (~100 nm wide with ~40 nm gap) served as the working electrodes; the polymer deposited on the wires and between them met in the center. Traces of current versus voltage ($I-V$) for the nanowires joined with PANI yielded linear functions without noticeable hysteresis (Figure 3c). After we exposed the substrate to an oxygen plasma to remove the polyaniline (Figure 3b), a subsequent $I-V$ trace displayed much lower conductivity and hysteresis; we attribute the residual conductivity to the insulating substrate. The conductivity after removing the polyaniline was similar to that obtained for samples that were not exposed to polyaniline or oxygen plasma.

Multiple Nanowires. An advantage of using nanoskiving, compared to other methods of making nanoscale gaps between electrodes, is that the technique is not limited to a single geometry. As a proof of principle, we modified the procedure in Figure 1 to include two additional steps

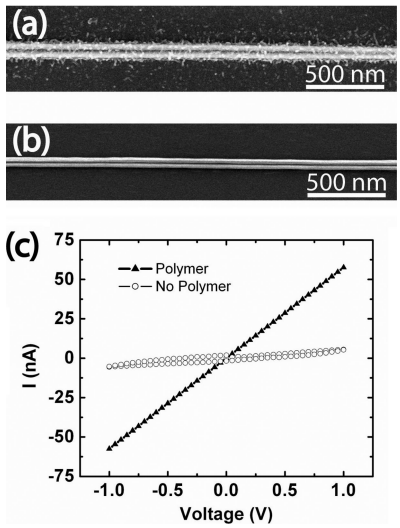

Figure 3. SEM images of electrically addressable Au nanowires. (a) After polyaniline was grown electrochemically across the wires. (b) Oxygen plasma removed the polymer, restoring pristine wires. (c) Electrical characterization of the wires. Inclusion of the polymer between the gaps increases the conductivity of the junctions. In the absence of polymer, the current decreases significantly.

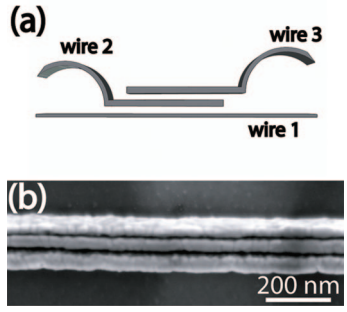

Figure 4. Three electrically addressable nanowires. (a) Schematic depicting the architecture of the wires. (b) Close up image of three parallel wires.

(summarized in Figure 4). After the second deposition of gold (Figure 1), we (i) spin-coated a thin film of polymer to serve as an additional spacer layer and (ii) deposited a third layer of gold (which ultimately became the third wire) while orienting the sample such that gold would deposit on the parallel region and the other side of the protruding addressable region. Figure 4 includes an SEM of this three-wire structure. The ability to fabricate three addressable terminals is useful for incorporating additional functionality into an electrical device, such as a transistor with a source, drain, and gate.

Conclusions. This paper describes a simple method to fabricate individually addressable nanowires without using direct-write lithographic tools. The technique combines nanoskiving and MIMIC to form nanowires that are parallel in one region of the structure, but diverge to create a region in which the wires can be addressed individually using low-resolution lithography.

We believe the technique will be of greatest interest to researchers who want to create nanoscale test structures without the use of sophisticated equipment or techniques. The ability to create many (hundreds to thousands of) easily manipulated polymer slabs containing these nanoscale structures (by successive sectioning) makes nanoskiving a very practical procedure. We fabricated parallel wires with a sacrificial polymeric spacer between the wires, but in principle, active materials (e.g., dielectrics, conducting polymers, and oxides) could be incorporated into that region during preparation of the sample. With further development, it may be possible to adapt this process to form narrower gaps, which could be used, for example, for molecular junctions. The nanowires may also be useful to create large local electric fields with the application of small voltages (e.g., ~2 V applied across a 30 nm gap would exceed the dielectric breakdown of air). In addition, the structures featuring three-terminal (or more) electrodes may be adapted to form sophisticated test-bed devices, such as nanoscale transistors (source, gate, drain).

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Supporting Information Available: More images of wires using different spacer geometries (Figure S1), additional experimental details, an image of the contact pads (Figure S2), a demonstration of electrical continuity along a single wire (Figure S3), and images of damaged wires (Figure S4). This material is available free of charge via the Internet at http://pubs.acs.org.

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