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Single-Nanowire strain sensors fabricated by nanoskiving

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ABSTRACT

This article describes the fabrication of single-nanowire strain sensors by thin sectioning of gold films with an ultramicrotome—i.e., "nanoskiving." The nanowire sensors are transferred to various substrates from the water bath on which they float after sectioning. The electrical response of these single nanowires to mechanical strain is investigated, with the lowest detectable strain determined to be 1.6×10^{-5} with a repeatable response to strains as high as 7×10^{-4}. The sensors are shown to have an enhanced sensitivity with a gauge factor of 3.1 on average, but as high as 9.5 in the low strain regime ($\varepsilon \sim 1 \times 10^{-5}$). Conventional thin films of gold of the same height as the nanowires are used as controls, and exhibit inferior sensitivity. The practicality of this sensor is investigated by transferring a single nanowire to polyimide tape, and placing the sensor on the wrist to monitor the pulse pressure waveform from the radial artery. The nanowires are fabricated with simple tools and require no lithography. Moreover, the sensors can be "manufactured" efficiently, as each consecutive section of the film is a quasi copy of the previous nanowire. The simple fabrication of these nanowires, along with the compatibility with flexible substrates, offers possibilities in developing new kinds of devices for biomedical applications and structural health monitoring.

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1. Introduction

Strain gauges are ubiquitous components of microelectromechanical systems, wearable sensors, and structural health monitors. Miniaturization of such systems requires techniques that can manufacture strain gauges with micro- or nanoscale dimensions. Moreover, strain gauges with reduced dimensions-e.g., nanowires-have the potential for increased sensitivity to strain compared to thin films [1] owing to increased scattering events in structures with a high ratio of surface area to volume. The simplest type of strain gauge uses a piezoresistive mechanism. That is, a change in resistance-generally an increase—with increasing strain. Typical materials used are metals and metallic alloys, along with semiconductors, which have a sensitivity to strain up to an order of magnitude higher than piezoresistors made from thick (~5 μm) metallic foils [2]. Semiconductor strain gauges are, however, planar and require multiple lithographic steps, and both metallic and semiconductor gauges require adhesives such as epoxies to bond them to a substrate of interest [1,3]. Nanoscale materials, in contrast, can bond to substrates by capillary action and stay in place by van der Waals forces. This paper describes the use of a single gold nanowire fabricated by nanoskiving (thin sectioning with an ultramicrotome)[4–10] as a highly sensitive transducer of mechanical strain.

The sensitivity of a strain gauge is described by its gauge factor (GF) which is defined as: $GF = \frac{\frac{\Delta R}{R}}{\varepsilon}$, where $\Delta R/R$ is the normalized change in resistance at strain ε [11]. The gauge factor for isotropic conductors which experience no change in bulk resistivity upon strain is $GF = 1 + 2\nu$, where ν is the Poisson ratio [11]. The change in resistance is due to lengthening of the material along axes parallel to the strain and shortening of the material along axes perpendicular to the strain and implies a maximum gauge factor of 2. Higher performance can be obtained by taking advantage of the effects of size confinement. Chunshien et al. demonstrated that as gold films become very thin—i.e., nominal thicknesses of ≤10 nm-they exhibit a microstructure of disconnected islands with gauge factors of 24–48 [12]. Our laboratory has demonstrated a similar effect for metallic nanoislands deposited on graphene, and attributed the high sensitivity to conductivity that was mediated partially by tunneling of electrons from nanoisland to nanoisland [13]. Contiguous metallic objects can also exhibit high gauge factors due to size confinement, caused by an increase in frequency of scattering events. Scattering events are collisions that can occur at surfaces and grain boundaries. In a scattering process, the momentum of the electron is deflected and the current is attenuated. For example, as each of the dimensions of the metal shrinks towards the electronic mean

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free path (approximately 34 nm for bulk gold, limited by acoustic phonon and grain boundary scattering [14]), the probability of scattering events on the metal surface increases [15]. In turn the mean free path of electrons in a nanowire is likely significantly smaller, on the order of several nanometers [16]. We therefore hypothesized that for nanowires under even minute tensile strains, the frequency of scattering events would increase significantly. That is, the strain should reversibly increase the resistivity of the nanowire more than anticipated by a simple change in geometry as observed in bulk samples. This effect has also been demonstrated in films consisting of many nanowires [17–20]. It seems that the gauge factor of a single nanowire quickly approaches continuum levels as the diameter increases. For example, at a diameter of approximately 300 nm, Chang et al. fabricated wires with gauge factor of approximately 2 (the value expected for a bulk sample of an isotropic conductor) [21].

Nanoskiving—nanofabrication by thin sectioning with an ultramicrotome equipped with a diamond knife—is a technique that relies on mechanical sectioning of an embedded structure in a thermosetting epoxy resin (the "block"). In this technique, the offcuts (the "slabs") contain consecutive cross sections (quasi copies) of the parent structure. This process is highly amenable to the reproducible fabrication of long, but thin nanowires (h, w < 150 nm), i.e., by sectioning thin films [4–6]. After sectioning the block, the slabs float on a water bath where they can be harvested in a thin film of water suspended in a loop tool, or lifted out from underneath the surface of the water using a flat substrate. Nanoskiving has the useful characteristic that it converts films, which are thin in the vertical dimension, into wires and other structures that are thin in the lateral dimension [7]. Moreover, it is possible to place the fabricated objects on any surface that can be wet by water [6]. The ease of use (no lithography) and length of the nanowires make nanoskiving favorable compared chemical synthesis [22], synthesis in templates [23], and lithographic techniques [24]. Fig. 1 summarizes the procedure we used to make nanowires for use as strain sensors.

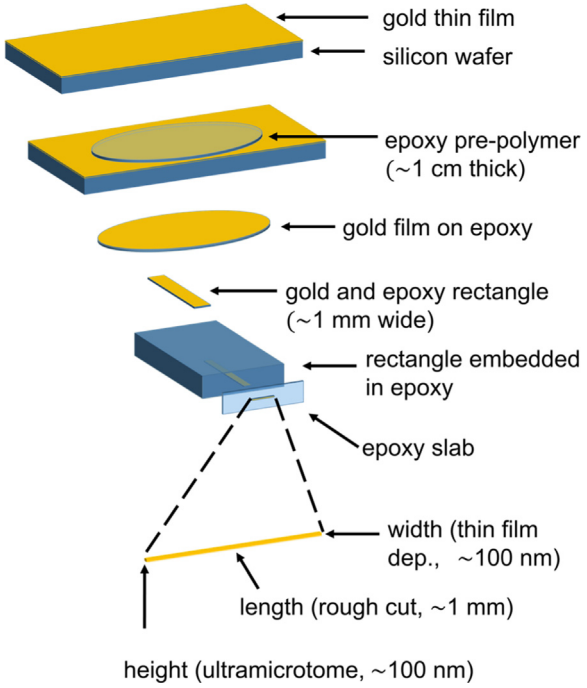

Fig. 1. Schematic diagram showing step-by-step fabrication of gold nanowires by nanoskiving. Gold is first evaporated onto silicon. Then, thermosetting epoxy resin is drop cast onto the gold. After curing, the epoxy is stripped from the silicon wafer, and strips the gold from the silicon as well. The gold film on epoxy is rough cut into a rigid rectangular shape, which is then embedded in more epoxy. Upon taking cross sections, or "nanoskiving," a thin slab of epoxy with an embedded nanowire is formed.

2. Experimental/Methods

2.1. Nanowire fabrication

We began by depositing 100 nm of gold onto a silicon wafer at an approximate deposition rate of 3 Å/s by electron-beam evaporation (Temescal BJD 1800-1). The gold was stripped from the silicon wafer by puddle-casting epoxy (Epo-fix, Electron Microscopy Sciences) on the gold surface. Upon curing at 60 °C for 6 h, we detached the epoxy, which stripped the gold from the silicon wafer. We then cut out long rectangular pieces (w = 1 mm, l = 5 mm) from the gold on epoxy using a razor blade and hammer. Finally we embedded these pieces in additional epoxy using a block mold made from PDMS on a petri dish, and sectioned the block using an ultramicrotome (Reichert-Jung Ultracut E) fitted with a 2.5 mm diamond knife (Diatome Ultra 35°) [4]. The diamond knife was fixed to a water bath, onto which the slabs slid as they were produced. We transferred the slabs using the Perfect Loop tool to silicon for microscopic characterization, thin glass slides (Corning Willow glass) for characterization of the electrical properties, and polyimide tape (Kapton) for human pulse monitoring.

2.2. Electrical addressing

A stripped optical fiber (d ≈ 120 μm) was placed perpendicular to the length of the nanowire to define a conductive span near the center of the nanowire. We then deposited gold contact pads approximately 100 nm thick on the surface, and when we removed the optical fiber, large gold contact pads were available for electrically addressing the wires. The widths of the nanowires (100 nm) were determined by evaporation of the gold film that was ultimately sectioned. The thicknesses of the slabs, and therefore the heights, were determined by the ultramicrotome, which was set to a nominal thickness of 90 nm. However, the set thickness of an ultramicrotome is unreliable, and we therefore measured thicknesses independently by using profilometry (Dektak). Profilometry revealed a range from 70 nm to 110 nm from section to section. The height of the nanowires that we used in for electrical characterization had heights between 80 nm and 90 nm.

2.3. Electrical characterization

We investigated the electrical characteristics of the nanowires by sourcing voltages from −10 to 10 mV. The gauge factors of the nanowires were evaluated by applying small bending strains as follows. First, the nanowires were deposited on glass coverslips. Then, they were placed partially suspended over step edges (10 μm–130 μm) made of photoresist (SU8 Microchem) on a glass slide. The glass coverslips were positioned such that the middle of the nanowire was sitting directly over the step edge. The coverslip was clamped to the supporting glass slide a small distance from the step edge itself, depicted in Fig. 3a. The free end was deflected to the point where it touched the glass underneath the step edge, applying a bending moment on the glass slide (and corresponding tension along the nanowires). Resistance was measured (Keithley 2400) by applying eutectic gallium-indium (EGaIn) drops to the evaporated contact pads, and placing copper wires inside. A continuous change in strain with time was also induced by placing the nanowire on thin 100 μm Willow glass (Corning) as a cantilever and deflecting it using a motorized test stand (Mark-10).

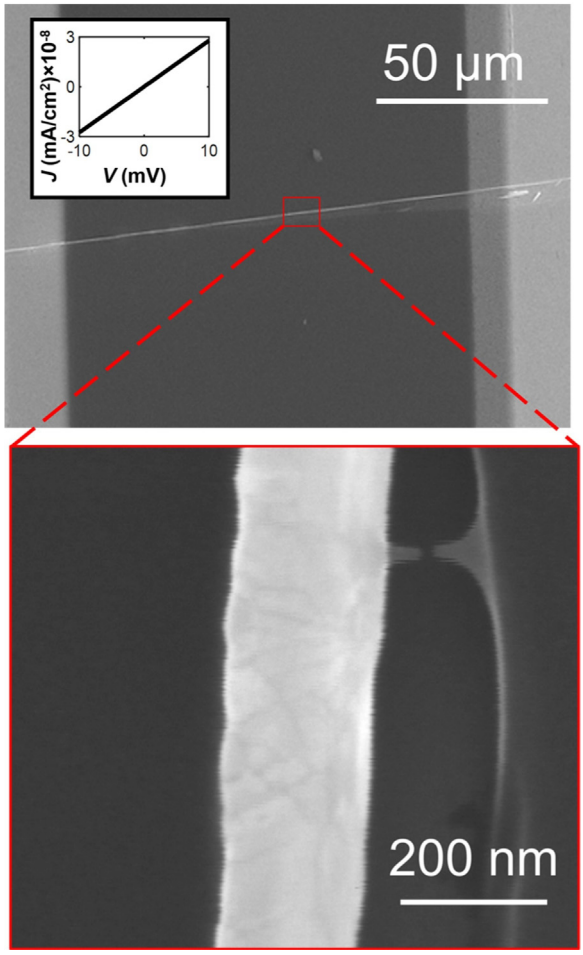

Fig. 2. SEM image of a gold nanowire positioned between gold electrodes. The inset is a typical plot of current density (J) vs. voltage (V). The lower image is a close-up showing the microstructure of the gold.

2.4. Electron microscopy

Scanning-electron microscope (SEM) images were collected using an FEI XL30 SFEG. The accelerating voltage for the inset of Fig. 2 was 10 kV, with a 40 μm spot size, and a through-lens detector, compared to a 15 kV accelerating voltage for the lower magnification image. The sample for imaging was prepared by transfer of the gold nanowire in its epoxy slab from the water bath of the ultramicrotome to a silicon wafer.

2.5. Evaluation of strain on substrate surfaces

The strain applied to the nanowire was calculated using a simple continuum mechanics model, assuming point forces are applied, and solving for the tensile deformation of the glass substrate. The strain on the nanowire was assumed to be the same as that at the top surface of the glass. The clamped end of the cantilever was separate from the step edge, providing a separate reactive force (and a reactive moment at the clamped end). The displacement of the free end was easier to measure than the applied force, and so the equations of elasticity are used to define the bending moment, then the strain on the glass surface [25]. The axial strain is modeled analytically by the equation $\varepsilon_{zz} = \frac{6bh}{4L^2-5aL+a^2}$, where b is the step edge height, h is the height of the substrate upon which the nanowire is affixed (typically glass), a is the distance from the clamped end to the nanowire, and L is the total length between clamped end and displaced end. The model assumes that material mechanical properties are uniform, and that the deformation occurs in the elastic domain of the stress-strain curve.

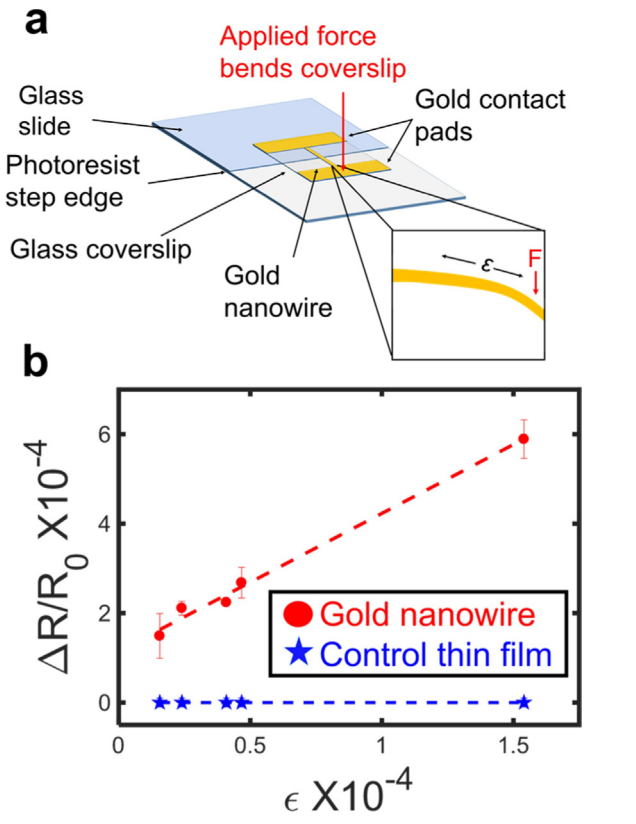

Fig. 3. Electromechanical measurements. (a) Schematic diagram showing experimental setup. A gold nanowire was deposited on a glass coverslip, and the coverslip was bent as a cantilever over a photoresist step edge. (b) Normalized resistance vs. strain ($\Delta R/R$ vs. ε) for which the strain was tuned by varying the thickness of the photoresist step edge. From this experiment, it was possible to measure a gauge factor of 3.1 ± 0.6, all at strains undetectable to the control thin film.

3. Results and discussion

Sectioning the gold thin film yielded 90 nm thick gold nanowires surrounded on all sides by the epoxy slab. These nanowires were clearly visible under electron microscopy (Fig. 2), and their widths confirmed to be approximately 100 nm, and uniform. The resistivity was calculated to be 3.2×10^{-8} Ω m, by SEM measurement of the width, and profilometry measurement of the height. This agrees with several accounts theoretical and experimental on the increased resistivity of nanowires compared to bulk gold; the resistivity of bulk gold is 2.2×10^{-8} Ω m, whereas literature results predict a 90 nm diameter nanowire to have a resistivity of around 3.9×10^{-8} Ω m [16,26].

We then investigated the strain sensing properties of these nanowires. The nanowires were placed on glass coverslips and electrically addressed. The glass coverslip was deflected, and the strain on the top surface of the coverslip produced a change in resistance. Based on the electromechanical measurements as depicted in Fig. 3a the relationship between relative resistance change $\frac{\Delta R}{R_0}$ and ε was determined. The figure demonstrates that there were no competing mechanisms of strain sensing depending on strain regime. The average gauge factor, calculated as the slope of the curve in Fig. 3b was calculated to be 3.1 ± 0.6. The specific gauge factor at the lowest strain measured, 0.0016%, however, was 9.5 ± 3.2. Thus, the gauge factor decreased as a function of strain, identical to the case of a thin film [12]. None of the strains depicted in Fig. 3b were detected by a control gold thin film (100 nm) deposited on

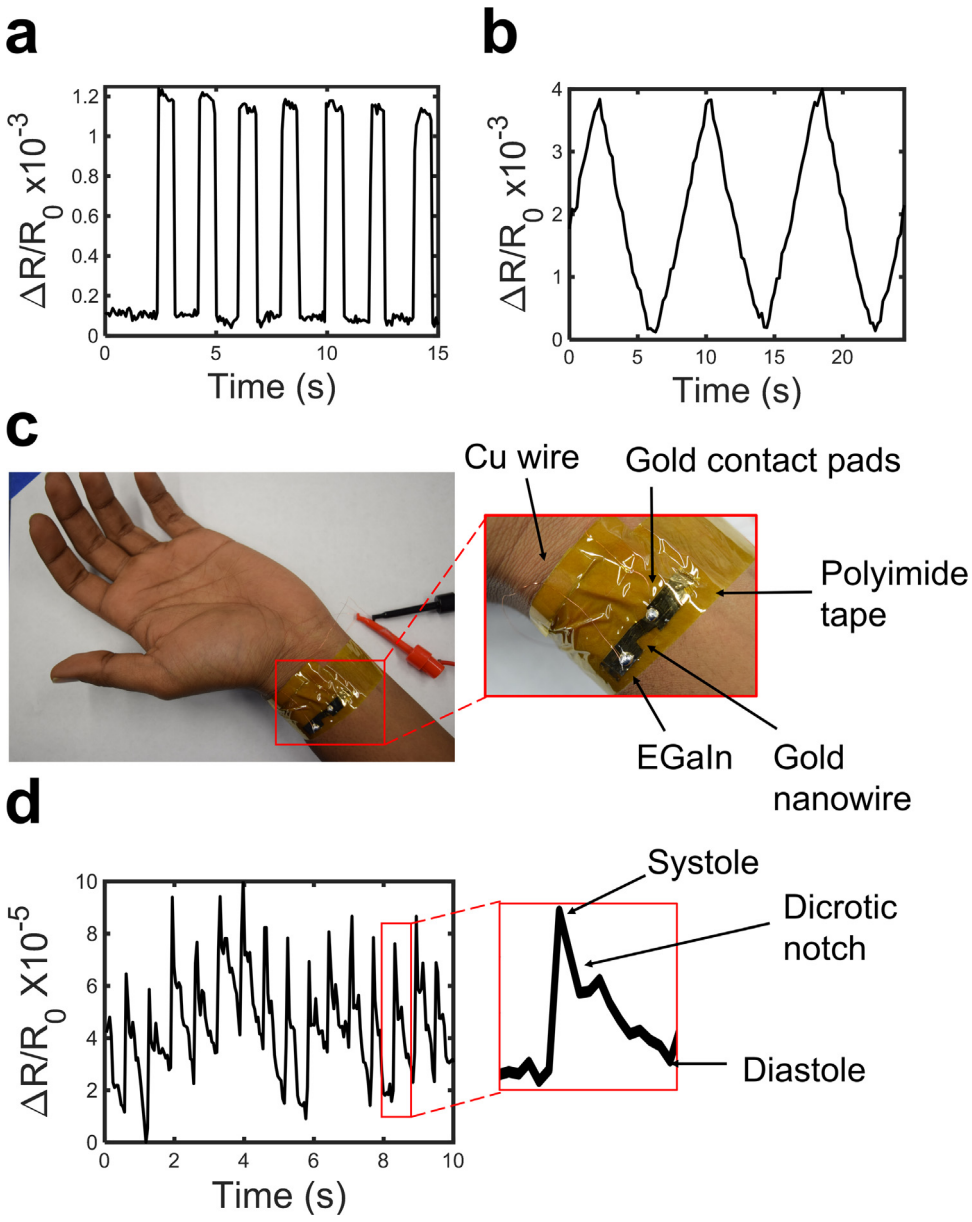

Fig. 4. Dynamic strain response of the gold nanowires. (a) Square waveform for strain of 0.011% applied by manual displacement of free cantilever end. (b) Continuous, cyclical waveform for strain of 0.07%. (c) Detection of human pulse pressure waveform using a nanowire on polyimide tape. (d) Plot of normalized resistance vs. time demonstrating transduction of the heartbeat signal.

a glass coverslip. Previously reported experimental findings have confirmed that these strains would not be detectable for a film of this thickness [27]. As the two cross-sectional dimensions of the nanowire were approximately 100 nm each, compared to the gold thin film being 100 nm thick, the difference in gauge factor can be understood as arising from confining electrons in two dimensions, as opposed to one.

After establishing that the gold nanowires could be used to detect small strains, the nanowires were tested for measurement of various strain signals. A square wave was applied by displacing the free end of a coverslip with the nanowire on top, holding it there briefly, then removing the strain. Fig. 4a shows transduction of the signal with minimal hysteresis at 0.011% strain. Resistance in the nanowire was also measured for a continuous change in strain as a function of time. A nanowire was placed on a sample of willow glass, which was in this case clamped to a test bed, with half of the glass hanging over the edge of the bed. The nanowire was positioned to be directly above the edge of the test bed. The free end was displaced, this time by a motorized test stand, which deformed the glass into an arc shape in a cyclic fashion. The free end was then relaxed, again in a slow continuous manner, forming an oscillating strain signal (although the rate of deformation in this case was actually constant). Fig. 4**b** displays accurate transduction of this signal, in this case with a maximum of 0.07% strain. To illustrate a biomedical application, nanowires were fabricated and transferred to Kapton polyimide tape, then were electrically addressed as discussed in Sections 4.1–4.3. Upon placing the now completely flexible sensor onto the radial artery, it detected the human pulse pressure waveform, as depicted in Fig. 4c.

4. Conclusions

Gold nanowires fabricated by nanoskiving can be used as piezoresistive strain sensors capable of measuring small strains on various substrates. The nanowires detected strains as low as

0.0015%, with a significantly higher gauge factor than that of bulk gold for this strain, or a gold thin film of similar characteristic length. Given the simplicity of fabricating and detecting strains using this device, there are attractive applications where costly lithographic techniques are excessive, such as lab-scale strain measurements. Further, the choice of form factor for the strain gauge is virtually unlimited, as the wires adhere by simply drying on the chosen surface, as long as it is sufficiently hydrophilic for water to wet the surface. This versatility makes the choice of substrate limited only to surfaces which are amenable to physical vapor deposition for the formation of contact pads, although other methods for electrical addressing may also be applicable. Finally, the small sizes of these strain sensors hold promise for possible applications in extremely lightweight wearable devices.

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Aliaksandr Zaretski is a PhD graduate of the Department of NanoEngineering at UC San Diego. Under Prof. Darren Lipomi, his research focused on the development of low-cost methods for the production of graphene and the use of graphene-nanoparticle strain sensors for wearable health and structural health monitoring. He is now the founder and CTO of GrollTex, Inc.

Darren J. Lipomi earned his undergraduate degree in chemistry from Boston University in 2005, and his Ph.D. at Harvard University in 2010, with Prof. George M. Whitesides. From 2010–2012, he was a postdoctoral fellow in the laboratory of Prof. Zhenan Bao at Stanford University. He began his independent position at UC San Diego in 2012 and is now an associate professor in the Department of NanoEngineering. He is the recipient of the AFOSR Young Investigator Award and the NIH Director's New Innovator Award.