Fabrication and Characterization of Microscale Sensors for Bone Surface Strain Measurement

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Abstract

We present micro-fabrication and device characterization results towards the realization of a flexible, implantable sensor array for measuring surface strain on live bone. Thin-film metal strain gauges were embedded in a poly-dimethyl-siloxane (PDMS) membrane. Various adhesives used to adhere the device to both imitation bone materials and cow bones were tested. The measured strain level and resolution indicates that the device is suitable for bone surface strain measurement. Ultimately this gauge will be part of an implantable, wireless array for real-time monitoring of bone disease and bone remodeling.

Keywords

Micromechanics, MEMS, piezoresistivity, strain gauge, bone

INTRODUCTION

Measuring bone surface strain *in vivo* would be invaluable for studying the structural effects of osteoporosis, tumors in bone, and prosthetic implants. However, currently available strain sensors are too large to provide measurements with suitable resolution and difficult to mount because of the irregular surface topology of bones. These limitations of off-the-shelf devices hinder important bone research.

By employing microfabrication technology, hundreds of microscale strain gauges could be fabricated in the space otherwise occupied by an off-the-shelf device and could potentially offer orders of magnitude higher spatial and temporal resolutions. Designs and simulations using ANSYS® finite-element modeling tool of the thin-film metal strain gauge embedded in a flexible and biocompatible PDMS membrane have already been presented [1]. In this paper, we demonstrate the fabrication and characterization of a single microscale prototype strain gauge. Metal films for electrical interconnection encapsulated in PDMS have been previously demonstrated [2]. In this work, we report the first demonstration of PDMS encapsulation of metal films that function as strain sensors as well as the adhesion characteristics of the sensors onto both imitation and live bone samples, paving the way for future research in implantable flexible strain sensor arrays for orthopaedic research.

DEVICE FABRICATION

The PDMS-based strain gauge with piezoresistive readout was fabricated on a silicon substrate, which functioned as a carrier for subsequent processing steps. The PDMS membrane thickness was first characterized to facilitate encapsulation designs.

PDMS Membrane Thickness Characterization

A thin layer of PDMS was spun onto glass slides with various spin rates from 200 to 2,000 rpm for 30, 60 and 90 seconds. The membrane thickness was then measured by varying the focal depths of an optical microscope while focusing on the top and the bottom surfaces of the PDMS. The measurement results are shown in Figure 1. Longer spin time also produced a more uniform layer of PDMS than shorter ones.

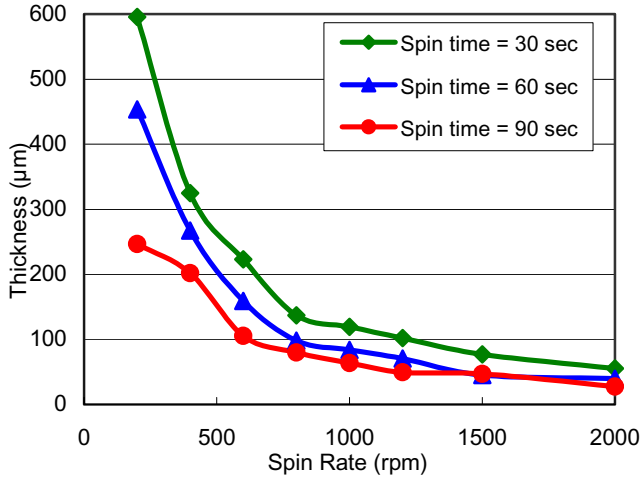

Figure 1. Characterization of PDMS thickness as a function of spin rate at 30, 60 and 90 seconds.

Fabrication Steps for Encapsulating Strain Gauge in PDMS

1. *Silanization:* The silicon wafer was first silanized for 24 hours to make the surface hydrophobic and to reduce the adhesion strength between PDMS and the silicon surface. This step was necessary to facilitate easy separation of the finished device from silicon dur-

ing the last step in the fabrication process. This silanization was done with trichlorosilane in a gas phase reaction, which produced a very thin silane coating on the silicon surface.

2. *PDMS Preparation:* PDMS prepolymer was mixed with its curing agent (Sylgard 184® elastomer, Dow Corning) at a ratio of 10:1 by weight. The mixture was degassed in vacuum to remove air bubbles.
3. *First PDMS Layer:* The PDMS in its liquid precursor state was spun onto the silanized silicon wafer at 1,200 rpm for 90 seconds, producing a uniform membrane thickness of 50 μm. The PDMS membrane was then cured at 70°C for 2 hours.
4. *Photolithography:* Prior to photoresist application, the PDMS was treated with oxygen plasma for 40 seconds at an RF power of 100 Watts under 100 mTorr of pressure. This step improved the adhesion between the photoresist and the polymer surface, resulting in a smooth and uniform coat of photoresist. The positive photoresist (AZ5214, Clariant) was then spun onto the PDMS surface and pre-baked. The photoresist features were then exposed and developed using the image reversal technique to generate a negative photoresist profile. The undercut being created in the photoresist profile would assist the ease of lift-off process in the later stage.
5. *PDMS Metallization:* A 100 nm gold film was deposited onto the photoresist patterns with e-beam evaporation following a 30 nm of chromium as the adhesion layer. Acetone was introduced to remove the photoresist covered by excess metal, leaving the metal in developed regions.
6. *Wire bonding and Second Layer of PDMS:* The bond wires were attached to the bond pads by applying conductive silver epoxy to the electrode pads. After the silver epoxy was cured at 70°C for 2 hours, the wafer was treated with air plasma, followed by a spin coating process of the second layer of PDMS to encapsulate the metal gauges.
7. *Device Separation:* The final step in the fabrication process is to cut individual devices followed by peeling from the silicon wafer.

Improved Device Separation Approach

Although silanization of the silicon wafer surface reduced adhesion of PDMS onto silicon, it was found that the weakened adhesion was still strong enough to cause damage to the devices during peeling, reducing the yield of working devices. We derived an alternative with the use of Kapton HN500 (DuPont) 5-mil-thick polyimide film as the interlayer between silicon and PDMS, resulting in a substantial reduction in adhesion beyond what we could achieve with silanization, while still maintain sufficient adhesion for mechanical supports for subsequent process steps.

The modified process to include Kapton was illustrated in Figure 2. A layer of PDMS was first spun onto a blank silicon wafer, followed by placement of the Kapton film on top of the PDMS, which serves as the adhesion layer between Kapton and silicon surface. The top Kapton surface was silanized for 30 minutes to further reduce the adhesion force between it and a subsequent PDMS layer. Then a structural PDMS layer was spun in its liquid precursor state at 1200 rpm for 90 seconds to create a membrane thickness of 50μm. The PDMS was then cured, followed by the steps similar to the original one.

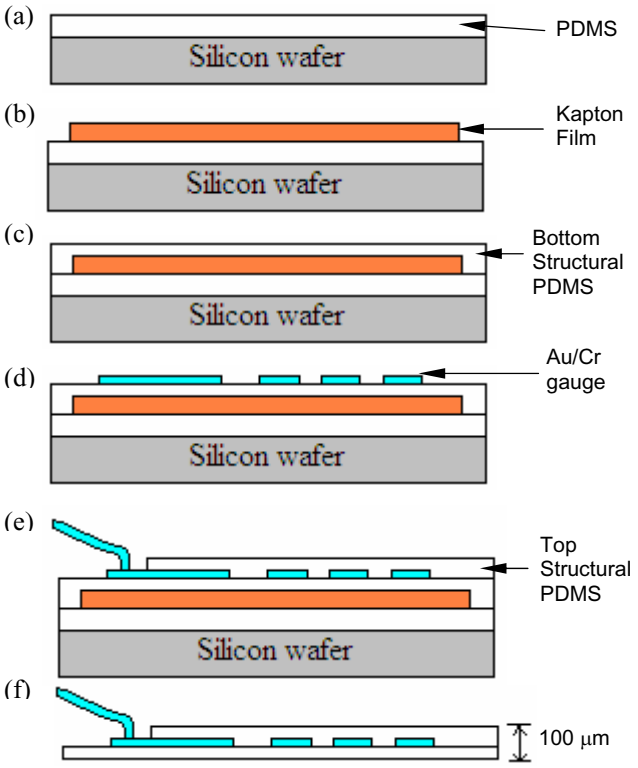

Figure 2. Process sequence for the fabrication of the flexible micro-sensor. (a) Spin a layer of PDMS onto the wafer. (b) Place a Kapton film on the cured PDMS and silanize. (c) Spin a 50-μm-thick layer of PDMS onto the Kapton surface as the bottom PDMS layer. (d) Photo pattern and E-beam evaporate 30 nm Cr and 100 nm Au, then lift-off PR to form the metal traces. (e) Bond wires to electrode pads with silver epoxy and spin on the top layer of PDMS (50 μm) to encapsulate the metal gauge. (f) Cut and peel off the device from the wafer.

CHARACTERIZATION

Several experiments were performed to measure the mechanical properties of the device and the electrical performance of the strain gauge.

Mechanical testing

A tensile tester was used to evaluate the mechanical properties and adhesion of the device to a rigid polyurethane foam block that mimics the surface of real bone (Figure 3).

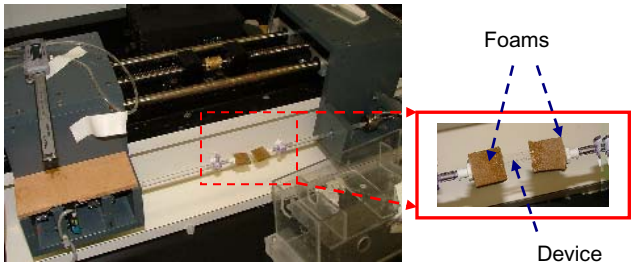

Figure 3. Sample under test in the tensile tester. Inset shows the device under tension.

The tensile tester imposed a controlled force and elongation of the device was measured. Strain was then calculated using LABVIEW®. Force was increased until the device yielded and broke apart in the center. The maximum strain that the device could sustain before breaking was 110%. The measured results were then plotted in Figure 4 showing the force/strain characteristics of the device. The plot clearly illustrates the elastic to plastic transition. From the slope of the curve, the effective Young's modulus of the device was calculated to be 17.7KPa. The stiffness was found to be 147N/m within the elastic region.

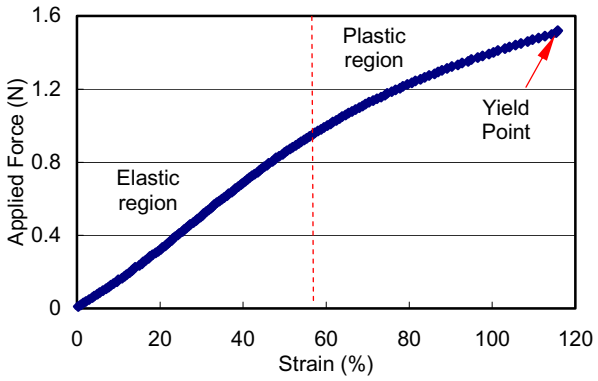

Figure 4. Force/strain curve for the device adhered to foam blocks with silicone sealant.

Electromechanical testing

The sensitivity of the piezoresistive strain sensor was measured by observing the change in resistance as a function of varying strain. The device was placed flat onto a Mitutoyo Absolute Coolant Proof Caliper, with the turns of metal gauges suspended over the 3.64-mm gap (Figure 5). The caliper resolution of 10 µm enabled strain of the device to be read with a precision of 0.27%. The different resistance values of the gauge were measured with an HP 34401A Digital Multimeter, at 10 µm increments of tensile displacement. The direction in which strain was measured in this caliper test was at 90° to the measurement direction in Figure 3.

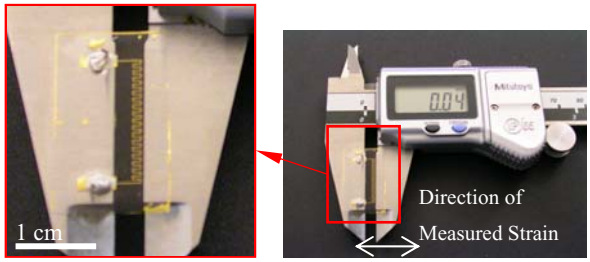

Figure 5. Stress-strain measurement. Inset shows the individual fabricated device and the direction in which strain was measured.

The sensitivity of the piezoresistive strain sensor had been measured by observing the relative change in resistance as a function of strain in the gauge. The measured result was plotted in Figure 6. It was found that the change in resistance was proportional to the change in length. Based on previous results shown in Figures 4 and 6, it could be concluded that the metal gauges encapsulated inside PDMS were operating in the elastic region.

The piezoresistive effect of the strain gauge can be quantitatively expressed by a gauge factor, G, which is defined as the change in resistance per unit strain:

$$G = \frac{\Delta R}{R\varepsilon}$$

, where R is the resistance of the gauge, ΔR is the change in resistance, and ε is the strain. Hence, from the slope of the plot, the gauge factor of the device was found to be 6.86. This value was higher than the normal gauge factor of 2 for metal gauges [3]. This was probably due to differences between thin film and bulk characteristics of gold.

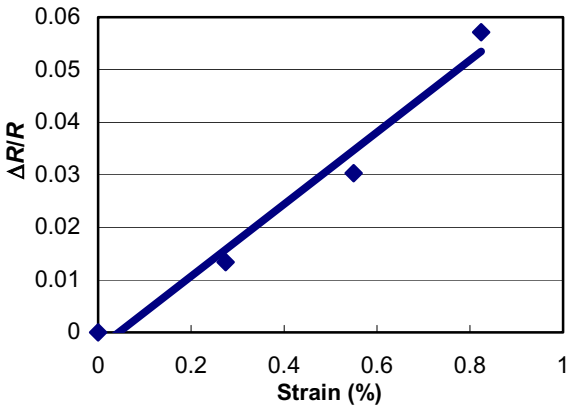

Figure 6. Strain sensitivity of the measured strain gauge.

It was also observed that when the tensile displacement exceeded 0.04 mm (1.09% of strain), the gauge was open circuited. Upon release, the gauge readings returned to normal. We concluded that 40 µm stretching caused the metal

traces to yield. The crack in the metal line was closed upon release due to the elastic behavior of PDMS.

For measuring strain in cortical bone or in an individual trabecula, a maximum of 0.5% strain would be used for nondestructive testing. As the device would eventually be used *in vivo* to measure the surface strain of the bone, the measured result showed that the device was capable of measuring strain in the required strain range, i.e. 0 to 0.5%.

Adhesion testing

A set of tests was conducted to compare the effectiveness of different adhesives. Several adhesives were evaluated both on high-density polyurethane foam and on bovine bone. The adhesives that were evaluated included Polymethyl methacrylate (PMMA), polycyanoacrylate, silicone gel, double-sided silicone based tape, and epoxy. With a simple peeling test, it was found that only polycyanoacrylate, silicone gel and double-sided silicone-based tape had firm attachment of the device to the bone surface. Further tensile testing was performed on these three adhesives on foam with an MTS servo-hydraulic testing machine. The results were summarized in Table 1.

Table 1: Maximum force to break the adhesion

Maximum Force for adhesive failure (N)			
Curing time	30 Min.	45 Min.	60 Min.
Silicone gel	103	145	204
Double-sided tape	60	56	20
Poly-cyanoacrylate	216	934	1091

Polycayanoacrylate was found to be the strongest, and the strength of the adhesives depended greatly on curing time (Table 1). It was noted that polycyanoacrylate and silicone gel exhibited increased adhesion forces as the curing time increased. However, the opposite was true for double-sided silicone-based tape.

The maximum force required to break the device was 1.5N (Figure 4). Hence, all three of the tested adhesives listed in Table 1 would be suitable for attaching the device to the surface of the bone.

In the future, we will reduce the device size to form a two-dimensional array of strain sensors with high resolution. The fabrication process will need to be further optimized to achieve the desired reduction in size.

CONCLUSIONS

We have fabricated a prototype of a microscale strain sensor encapsulated in PDMS for measuring strain on a bone surface. We have also electromechanically tested the devices and shown that is suitable for monitoring strain on a bone surface.

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REFERENCES

[1] G. Y. Yang, V.J. Bailey, G. Lin, W. C. Tang, and J. H. Keyak, "Design of microfabricated strain gauge array to monitor bone deformation *in vitro* and *in vivo*," *Proc. IEEE 4th Symp. Bioinformatics Bioeng. (BIBE 2004)*, Taichung, Taiwan, May 19 – 21, 2004, pp. 30 – 37.

[2] M. Maghribi, J. Hamilton, D. Polla, K. Rose, T. Wilson, and P. Krulevitch, "Stretchable micro-electrode array," *Proc. 1st IEEE Eng. Medicine Biology Soc. Summer School on Medical Devices & Biosensors*, Madison, WI, Jul. 29 – Aug. 02, 2002, pp.80 – 83.

[3] Kovacs, G., *Micromachined Transducers Sourcebook*, first edition, WCB/McGraw-Hill: Boston, 1998.