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MEMS based Low Cost Piezoresistive Microcantilever Force Sensor and Sensor Module

H. J. Pandya^{1,*}, Hyun Tae Kim¹, Rajarshi Roy¹, and Jaydev P. Desai¹

¹Department of Mechanical Engineering, Maryland Robotics Center, Institute for Systems Research, University of Maryland, College Park, MD 20742, USA

Abstract

In the present work, we report fabrication and characterization of a low-cost MEMS based piezoresistive micro-force sensor with SU-8 tip using laboratory made silicon-on-insulator (SOI) substrate. To prepare SOI wafer, silicon film (0.8 μm thick) was deposited on an oxidized silicon wafer using RF magnetron sputtering technique. The films were deposited in Argon (Ar) ambient without external substrate heating. The material characteristics of the sputtered deposited silicon film and silicon film annealed at different temperatures (400–1050°C) were studied using atomic force microscopy (AFM) and X-ray diffraction (XRD) techniques. The residual stress of the films was measured as a function of annealing temperature. The stress of the as-deposited films was observed to be compressive and annealing the film above 1050°C resulted in a tensile stress. The stress of the film decreased gradually with increase in annealing temperature. The fabricated cantilevers were 130 μm in length, 40 μm wide and 1.0 μm thick. A series of force-displacement curves were obtained using fabricated microcantilever with commercial AFM setup and the data were analyzed to get the spring constant and the sensitivity of the fabricated microcantilever. The measured spring constant and sensitivity of the sensor was 0.1488N/m and 2.7mV/N. The microcantilever force sensor was integrated with an electronic module that detects the change in resistance of the sensor with respect to the applied force and displays it on the computer screen.

Keywords

MEMS; Piezoresistor; Microcantilever; Force Sensor; Sensor Module; Atomic Force Microscopy

1. Introduction

There has been significant interest in the mechanical characterization of biological materials such as cells and tissues. Mechanical properties such as Young's modulus may be used as a useful label-free biomarker for the diagnosis and treatment of diseases [1–4]. The mechanical properties of the biological materials can be measured by nanoindentation

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hjpandya@umd.edu, Phone: +1-301-405-7012, Fax: +1-301-314-9477.

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technique which requires the ability to measure forces in the micro-to-nano Newton range. Thus, a highly sensitive micro-force sensor can be used for bio-mechanical characterization. The bending of the cantilever is related to the applied force. Thus, the applied force can be detected by measuring the displacement of the reference cantilever sensor. The sensing methods for detection of micro-forces are: piezoresistive, capacitive and optical/laser detection. The optical method, which is mostly used in AFM, is based on focusing a laser spot on the cantilever and then monitoring the motion of the reflected light using a photodiode. A highly accurate optical system is required for measuring the displacement and the tip of the cantilever depends on the laser spot size. This method therefore becomes difficult when the size of the cantilever is smaller [5]. The capacitive method is based on the change in capacitance based on the deformation of the structure. The application of this method of detection is in micro accelerometers and sensors for harsh environments. The capacitive structures, however, need to be completely isolated between two electrodes [6]. The difficulty in fabricating the capacitive micro-force sensor is primarily in controlling the etching area to obtain a completely isolated structure and the measurement requires complex electronic circuits. The piezoresistive transducers translate a force into a change in the value of a resistor. This type of transducer is used as nano-mechanical sensor [7], binding force sensor [8] and bio-chemical mass sensor [9]. A resolution of pN [10] and even fN [11] is possible in case of piezoresistive microcantilevers.

It is known that the single crystal silicon exhibits a strong piezoresistive effect [12]. The piezoresistive cantilever sensor is generally fabricated using silicon-on-insulator (SOI) substrate due to the established fabrication technology and excellent material properties [13–31]. However, the cost of the sensor is an issue. The polysilicon material has been explored for fabricating piezoresistive cantilevers by several groups [32–34]. In our previous work [35], we have presented our preliminary work on the fabrication of the piezoresistive sensor and performed some preliminary experiments. The present work is an attempt to address the problem of high fabrication cost of piezoresistive microcantilever using SOI substrate and fabrication of cylindrical tips by: (a) reducing the cost of the device by using laboratory made SOI rather than conventional SOI substrate - batch fabrication of SOI from standard silicon is possible and thus the cost of piezoresistive microcantilever fabricated using homemade SOI is comparatively lower than commercially available SOI substrate and (b) using SU-8 polymer as the cylindrical tip material. To, understand the material properties of the sputtered silicon used in the microcantilever fabrication, material characterization techniques such as XRD, AFM, SEM and stress measurement are studied in detail. The sensor spring constant and sensitivity of the fabricated microcantilever is measured using AFM [36,37]. A sensor module is developed to integrate the sensor to measure and display the output of the sensor when a nano-to-micro Newton force is applied.

2. Experimental Details

To prepare silicon-on-insulator substrate, the silicon (Si) wafers [n-type, (100), 4-inch diameter] were cleaned by using a standard wafer cleaning process and a thermal oxide (SiO₂) of 1 μm thickness was grown using the Tystar oxidation system. Si films (0.8 μm) were sputtered on oxidized Si wafer by RF magnetron sputtering (ATC 1800, AJA International) without any external substrate heating. The summary of the deposition process

is given in Table 1. The residual stresses of the thin film depends on the microstructure of the film and mechanisms such as crystallographic orientation of the film, grain size, and surface roughness have been identified as possible causes for the residual stress evolution [38–41].

To study the effect of annealing on the quality of the film, a set of four films were post deposition annealed in nitrogen (N_2) ambient medium at 400°C, 600°C, 800°C, and 1050°C for 1h duration at each temperature.

The thickness of the film was measured by reflectance measurement (n&k analyzer from n&k technologies) technique. The crystallographic orientation of the as-deposited and annealed film was studied using XRD measurement technique (Bruker D8 Advance powder diffractometer in glancing angle mode). Surface roughness of the as-deposited and annealed Si films was measured by AFM (Model: MFP-3D-BIOTM, Asylum Research). The residual stress of 0.8 μm thick Si films was determined from the change in the radius of curvature of the wafer, before, and after film deposition, using a temperature controlled film stress measurement system (Flexus F2320). The stress measurements were performed after individual annealing step. Silicon nitride (Si_3N_4) was grown using PECVD on silicon film to compensate the stress in the film [42].

Sensor Fabrication: The schematic diagram of the piezoresistive force sensor with its dimensions is shown in Fig. 1. A seven mask process was used to fabricate the sensor. The process flow for sensor fabrication is shown in Fig. 2.

The process of preparing SOI from standard Si is as described above.

The sensor was fabricated using the following process: (a) SOI was selected as the substrate, (b) 1 μm SiO_2 was thermally grown on SOI, (c) window was opened for diffusing boron in resistor area (mask #1), (d) wafer was coated with spin-on-dopants (SODs) B155 from Filmtronics, Inc. - the diffusion was carried out at 1050°C for 1h in N_2/O_2 (70:30%) environment and after diffusing boron, the boron oxide layer was etched in buffered hydrofluoric acid (BHF). The resistivity of the film was found to be $1.316 \times 10^{-3} \Omega\text{-cm}$, (e) to make ohmic contact with the metal (Cr/Au) used in the subsequent step, the silicon at the piezoresistor contact pads needs to be heavily diffused. SiO_2 was grown after diffusing the piezoresistor area to facilitate opening of contact pads where the silicon needs to be heavily doped, (f) Window was opened in the contact area (mask #2), (g) SOD B154 was used for doping the contact area to diffuse the contact area with high doping concentration. The resistivity of the film was $1.747 \times 10^{-4} \Omega\text{-cm}$ and the optical photograph of the doped resistor with contact area is shown in Fig. 3, (h) Chrome/Gold (Cr/Au: 100/500 nm) was deposited using e-beam evaporator and patterned to form electrical contact pads (mask #3) (Fig. 4), (i) 200 nm thick silicon nitride was deposited using PECVD and window was opened from the contact area using RIE (mask #4), (j) Si was etched from the top to form cantilever structure using deep-reactive-ion-etching (DRIE), (k) To make the sensor tip, a 10 μm thick SU-8 (SU-8 2007, MicroChem. Corp) polymer was spin coated and patterned (mask #5) (Fig. 5), (l) Front-to-Back alignment was used to open the window from backside to etch through silicon (mask #6). EVG 620 system was used for performing front-to-back

alignment lithography. The contact alignment marks were used for back side alignment with respect to front side. The bulk etching of silicon is carried out using dry etching method called deep-reactive-ion-etching (DRIE). The thickness of the wafer is $500 \pm 10 \mu\text{m}$. As the etching of silicon from back window is crucial we have used silicon dioxide (SiO_2) as an etch stop as seen in Fig. 2(j), and (m) SiO_2 layer was etched from the backside of the sensor using RIE to release the sensor as shown in Fig. 6. The photograph of fully released batch fabricated microcantilevers on 4" diameter wafer is shown in Fig. 7. The SEM image of completely released sensor is shown in Fig. 8.

The SEM image of SU-8 tip ($8 \mu\text{m}$) integrated with the cantilever is shown in Fig. 9. The diameter of the SU-8 tip was kept to be $10 \mu\text{m}$. As the process steps require to pattern SU-8 are comparatively easy than to form Si tip it was selected for tip fabrication.

To calibrate the spring constant and sensitivity of the cantilever beam an AFM setup with reference cantilever beam was used. The calibration was performed by applying a known reference force from the reference AFM cantilever and measuring the deflection of the test cantilever.

3. Results and Discussion

3.1 X-ray Diffraction

The XRD patterns of the Si films were investigated to obtain a correlation between the structural and mechanical properties. The XRD measurements were carried out with Cu-K α radiation, 40 kV anodic voltage, and 40 mA current at each stage of the processing. A small scanning step of 0.015° and a fixed time interval of 1 second per step were used. Fan et al. [5] showed that the compressive residual stress in polysilicon films could be greatly reduced by depositing layers with a dominant grain orientation of (111). Therefore, the microstructure of the silicon films is first analyzed by XRD. Figure 10 shows the XRD patterns of silicon films ($0.8 \mu\text{m}$): as-deposited and annealed for 1h at 400°C , 600°C , 800°C , and 1050°C respectively.

It can be seen that the as-deposited film grows in the (220) orientation. As a result of the annealing, the peak intensities of (111) and (311) increase with annealing temperature while the peak intensity of (220) was found to be same. This indeed indicates a change in the microstructure as the film is further crystallized due to annealing. Furthermore, the film is reasonably stable and the crystallization level increases with annealing temperature.

The higher (111) peak may indicate that some defects disappear and recrystallization of the silicon films occurs with increase in the annealing temperature. The crystallite sizes of the as-deposited and annealed Si films at 400°C , 600°C , 800°C , and 1050°C calculated from Scherrer's formula is given by [43]:

$$C_p = \frac{K\lambda}{\beta_{\frac{1}{2}} \cos\theta} \quad (1)$$

where, C_p is the crystallite size, λ is the X-ray wavelength, K in our case is 0.94, $\beta_{1/2}$ is the line broadening at full width half the maximum intensity (FWHM), and θ is the Bragg angle. The calculated crystallite size was 93 nm, 98 nm, 106 nm, and 144 nm respectively.

3.2 Surface Roughness

In Fig. 11, we present the surface roughness results of as-deposited and annealed Si films as obtained from AFM. It was observed that the surface roughness of the Si film increases with increasing annealing temperature.

With the increase of annealing temperature, an increase of surface mobility occurs. By the growth of grains and decrease of grain boundary area, the film lowers its total energy, which leads to the increase of the grain sizes [6]. The roughness of the film surface is represented by the RMS value, which is calculated by using the Igor Pro software (WaveMetrics, Lake Oswego, OR) used with the AFM. The RMS of the as-deposited and annealed Si films at 400°C, 600°C, 800°C and 1050 °C is 1.296 nm, 1.812 nm, 2.509 nm, 11.507 nm, and 3.134 nm respectively. The surface roughness of the film increases in the annealing temperature range of 400–800°C and the average crystallite size increases from 93 nm to 106 nm. At the same time, the film becomes more granular which also results in increased surface roughness. For the thin film annealed at 1050°C, the decrease of the RMS (3.134 nm) is the result of the partial agglomeration of grains.

3.3 Residual Stress

The success ratio of the sensor fabrication depends on the stress of the material from which it is fabricated. Thus, the effect of temperature on stress of the deposited Si film was studied. The substrate curvature method relies on Stoney's formula relating the average stress of the film to the substrate curvature under the assumption that the film is much thinner than the underlying substrate [7]. The stress in a thin film of thickness d_f can be expressed as [44]:

$$\sigma_f = \frac{E_s d_s^2}{6(1 - \nu_s) d_f} \left[\frac{1}{R_f} - \frac{1}{R_s} \right] \quad (2)$$

where, E_s , ν_s and d_s are Young's modulus, Poisson ratio, and the thickness of the substrate, respectively; R_s and R_f are respectively the radii of substrate curvature before and after film deposition. Figure 12 shows the dependence of the stress on the post deposition annealing temperature for 0.8 μm thick Si film. The annealing was performed in N_2 ambient medium with a purity of 99.995% for 1h. The as-deposited Si thin film has a compressive residual stress of about 340 MPa. On increasing the annealing temperature the stress decreases gradually. Annealing Si films at 1050°C for 1h resulted in tensile stress of about 38.3 MPa. To avoid buckling/delamination of the Si film and compensate the stress, a low stress Si_3N_4 film (200 nm) was deposited on top of Si film using PECVD resulting in overall stress of 20.0 MPa. The variation in the residual stress in silicon film with increase in annealing temperature may be attributed to the change in the grain size of the crystallites. The increase in the grain size also means that the grain boundaries are reducing in the Si thin film. Grain boundaries are known to change the stress in Si films [45, 46].

3.4 Spring Constant

The theoretical spring constant for the fabricated sensor can be computed using Eq. (3), as given below [44]:

$$k = \frac{Ewt^3}{4l^3} \quad (3)$$

where, E, w, t and l are the Young's modulus of silicon (169 GPa), width, thickness, and length of the cantilever beam, respectively. However, geometric errors arising out of microfabrication inaccuracies, in particular the thickness of the cantilever, t, necessitate prior calibration of the spring constant of the force sensor against a known standard before they are used for biomechanical characterization. We use the reference cantilever method [47] using a pre-calibrated AFM probe (reference cantilever) to calibrate the spring constant of the microfabricated force sensor (test cantilever). The calibration process is carried out as follows:

1. The spring constant of the reference cantilever, k_{ref} , is first calibrated in air using the thermal method [48]. This is carried out using an AFM (Model: MFP-3D-BIO™, Asylum Research) and the Igor Pro software (WaveMetrics, Lake Oswego, OR).
2. To calibrate the spring constant of the test cantilever, a known force is applied to the end of a test cantilever, as shown in Fig. 13. This is performed by taking an AFM force curve at the end of the test cantilever (Fig. 14). The measured transmitted force is given as:

$$F = k_{ref}(d_1 - d_0) \quad (4)$$

where, d_1 and d_0 are the final and initial deflection of the reference cantilever as sensed by the AFM photodiode respectively (Fig. 13 and Fig. 14).

The deflection in the test cantilever, δ_s , is given by the difference of the total Z-travel and the net deflection of the reference cantilever, as given below [49]:

$$\delta_s = (z_1 - z_0) - (d_1 - d_0) \quad (5)$$

The transmitted force, F, and the deflection δ_s of the test cantilever are plotted in Fig. 15 for increasing force values. The calibrated spring constant of the test cantilever is given by slope of the linear fit of F and δ_s . The slope was found to be 0.1488 N/m, while the theoretical spring constant was 0.32 N/m.

3.5 Sensor Module

The sensor resistance changes with the indentation force. This change in resistance is sensed by converting it to change in voltage by providing bias to the sensor. The sensor module generates bias for the sensor to convert change in resistance to voltage. The sensor output now contains two voltage components; one corresponding to the bias voltage and the other corresponding to the change in resistance due to the force. The signal corresponding to the

force is separated by removing the bias from the buffered signal from the sensor. This signal is then amplified by a variable gain instrumentation amplifier. The analog output voltage of the instrumentation amplifier is then applied to Data Acquisition Card (Sensoray Model 626) and is displayed on the computer screen. The block diagram of the sensor module is shown in Fig. 16.

The detailed circuit diagram of the sensor module is shown in Fig. 17. Figure 18 shows the output voltage (V) of the sensor as a function of time on applying a 120 nN force. The sensitivity S was calculated as:

$$S = \frac{(V_1 - V_2)}{F} \quad (6)$$

Where, the subscript 1 refers to the point of contact between the SU-8 tip and the glass surface and subscript 2 refers to the maximum deformation in the sensor. For a force (F) of 120nN, the sensitivity of the sensor was found to be 2.7mV/N. The force of 120nN is selected in the present study as this amount of force was used to indent the breast tissue using semi-automated positioning system using AFM [50]. The results obtained shows that the fabricated sensors can be used to characterize the mechanical properties of breast tissues.

4. Conclusion

We have successfully demonstrated a low cost fabrication process for piezoresistive microcantilever sensors with SU-8 tip using a SOI substrate developed in the laboratory. The present sensor fabrication technology can be adopted in several application areas in research as well as in industry, including but not limited to standard IC manufacturing, without any major modification. The sensitivity and spring constant of the sensor was measured using AFM technique. The measured value of sensitivity and spring constant from AFM studies were found to be 2.7mV/N and 0.1488 N/m respectively. An electronic circuit consisting of sensor head, signal conditioning unit, and display interface was developed. Sensor output was measured using the electronic module and was displayed on computer screen. The results demonstrated the feasibility of using piezoresistive cantilevers to mechanically characterize biological cells and tissues.

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Fig. 1.
Schematic of piezoresistive micro-force sensor.

Fig. 2.
Process flow for piezoresistive microcantilever sensor.

Fig. 3.
Optical photograph of doped resistor.

Fig. 4.
Optical photograph of piezoresistor with patterned Cr/Au contacts.

Fig. 5.
Optical photograph of front side of un-released piezoresistive cantilever beam with SU-8 tip.

SU-8 tip 10 μm diameter

Fig. 6.
Optical photograph of released sensor with SU-8 tip.

Fig. 7.
Photography of fully released microcantilevers on 4" diameter wafer.

Fig. 8.
SEM image of completely released sensor.

Fig. 9.
SEM image of SU-8 tip.

XRD DATA ANALYSIS OF Si/SiO₂/Si samples: as-deposited, annealed at 400 °C, 600 °C, 800 °C and 1050 °C

Fig. 10.
XRD spectra of as-deposited and annealed silicon films.

Fig. 11.

Effect of annealing temperature on surface roughness of Si film. (a) as-deposited film (1.296 nm), (b) Si film annealed at 400°C (1.812 nm), (c) Si film annealed at 600°C (2.509 nm), (d) Si film annealed at 800°C (11.507 nm), and (e) Si film annealed at 1050°C (3.134 nm).

Fig. 12.
Effect of the sequential annealing process on residual stress for Si film.

Fig. 13.

Schematic diagram of the AFM configuration used for measuring the spring constant of an unknown test cantilever using reference cantilever. (a) AFM reference cantilever in contact with the test cantilever, (b) reference cantilever is pressed against the test cantilever to a preset force value.

Fig. 14. AFM force curve on the test cantilever. The approach curve is used for calibration process.

Fig. 15. Plot of probe deflection versus force of fabricated piezoresistive cantilever sensor. (R-square of the linear fit: 0.9852).

Fig. 16. Block diagram of sensor module incorporating the sensor chip, signal conditioning circuit and the display.

(a)

Fig. 17.

(a) Circuit diagram of sensor module with sensor-head and electronic conditioning circuit,
(b) Photograph of AFM with Sensor Module, (c) photo of electronic conditioning circuit.

Fig. 18.
Plot of sensor module output on applying a force of 120 nN.

Table I

Sputtering parameters used for depositing Si films

Deposition Parameters	Conditions
Si target size	6-mm thick, 3-mm diameter
RF Power	200 W
Sputtering gas	Argon
Sputtering pressure	5 mTorr
Target-to-substrate distance	100 mm
Substrate	Oxidized Silicon (4-inch diameter)
Substrate Temperature	Room Temperature (~30 °C)
Rate of Deposition	25 nm/min