Fabrication of microcoil/microsprings for novel chemical and biological sensing

Yangqing Lu, Hai-Feng Ji

Institute for Micromanufacturing, Louisiana Tech University, Ruston, LA 71272, USA

Received 23 July 2006; received in revised form 24 October 2006; accepted 30 October 2006

Abstract

This paper reports the fabrication of a novel microsensor structure using inductively coupled plasma source (ICP) dry etching process. The novel sensor is based on a SiO2/Si/SU-8 trilayered microcoil/microspring structure. The diameter of the microcoil was approximately 600 μm. The ICP process and SU-8 exposing time are discussed. The SiO2 layer can be conveniently modified according to typical silica surface modification procedures. The microcoils could expand or contract upon interaction of specific species in the environment with receptors on the SiO2 surface due to the surface stress or energy applied on the SiO2 surface of the microcoil. This microcoil device may be widely used in biomedical and chemical sensing applications.

Keywords: Chemical sensor; Microsensor; Microcoil; Microspring; Silicon plasma dry etching

1. Introduction

The first crude hygrometer was built in the 1400s by Leonardo da Vinci. In early 1700, Robert Hooke invented the first balance spring or coil that improved the meteorological instruments such as the barometer, anemometer, hygrometer, etc. [1]. In a dial hygrometer, a humidity sensitive polymer coated metal coil is attached to a dial to indicate humidity. The coils undergo extension or contraction due to moisture vapor absorption by confining the absorption to one side of the coil. When the relative humidity increases, the polymer swells which extends the coil and rotates the dial hand. At a low relative humidity, the polymer releases the tension on the coil, which allows the dial hand to move in the opposite direction. However, although the bilayer-based dial barometer, thermometer, and hygrometer have been used for several centuries, no other sensors and microsensors have been developed based on this device. Furthermore, although the coil movement can be readily observed by human eyes and it advances as a cost-effective and power-free device, due to the relative large size, the dynamic response time of this coil device cannot compete with many other micro and nanosen-

* Corresponding author. Tel.: +1 318 257 5125; fax: +1 318 257 5104.
E-mail address: hji@chem.latech.edu (H.-F. Ji).

© 2006 Elsevier B.V. All rights reserved.

Keywords: Chemical sensor; Microsensor; Microcoil; Microspring; Silicon plasma dry etching

Fig. 1. The SEM picture of a SiO2/Si/SU-8 trilayered microcoil and array. In the extended form, the dimensions of the microcoil were 9.8 mm in length, 50 μm in width, and 28 μm in thickness. The coil size is 500 μm × 600 μm. The thickness of the Si and SU-8 layer were 8 and 20 μm, respectively. For fabrication simplicity, the coil was made in a structure that the two ends of the coil were attached to two pads outside of the coil. To match with this structure, the specially designed SU-8 layer was kept on the outside surfaces of the coil in order to achieve a uniformed contraction or expansion of the coil, i.e. the SU-8 layer from the bottom pad to the center was not connected with the SU-8 from the top pad to the center.
It is expected that a coil device at micro or nanoscale size will provide a sensor platform with an improved dynamic response, greatly reduced size, and integration of micromechanical components with on-chip electronic circuitry. In this work, we report the fabrication of a SiO₂/Si/SU-8 trilayered microcoil (or called microspring) for sensing validation. The piezoresistive property of the doped Si was used to determine the adsorption/absorption induced microcoil extension.

2. Fabrication process

A fabricated trilayered microcoil/microspring is shown in Fig. 1. The diameter of the microcoil was approximately 600 μm. In the extended form, the dimensions of the microcoil were 9.8 mm in length, 50 μm in width, and 28 μm in thickness.

A commercially available Si wafer was used to fabricate the microcoils. The fabrication process is illustrated in Fig. 2. First, the Si-layer coil was patterned by photolithography and BOE (buffered oxidation etchant, HF:HNO₃ = 1:6) etching; Shipley 1813 positive tone photoresist was spun on the surface of the silicon wafer (Fig. 2A). The Si coil pattern was transferred to the photoresist layer on the front side of the wafer by standard photolithography process (Fig. 2B and C) and a SiO₂ mask was formed by etching using BOE (Fig. 2D). The patterned Si wafer was put into inductively coupled plasma source (ICP) to release...
Si coil (Fig. 2E). After removing S1813 by acetone (Fig. 2F) and the SiO₂ mask layer by BOE etchant (Fig. 2G), two platinum pads (not shown in the figures) were applied on outsides of the two ends of the microcoil by sputtering and lift-off patterning process. The wafer was then put in a furnace for oxidation to obtain an 1 μm-thick of SiO₂ on the sidewalls (Fig. 2G). Depending on the temperature setting of the furnace, this process could last at least 12 h. Then SU-8 negative photoresist was spun on the front side of the wafer by spin-coating and photolithography to form the layer of polymer coil (Fig. 2H). The last step of the fabrication procedure involved backside ICP process to release the microcoil from the substrate. This was realized by photolithography to define the etching pattern followed by ICP process (Fig. 2I–K).

3. Results and discussions

ICP (ionic coupled plasma) etching is a chemical dry etching technique that offers many advantages over wet chemical etching methods, including high etch rate, compatibility with traditional IC processing [2], high Si:SiO₂ etching selectivity (up to 350:1) [3]. The high selectivity is a great advantage in producing the Si coil as well as releasing the coil from bulk Si. Deep dry plasma etching achieves the high etching rate and straight sidewall. In an ICP plasma etching process, the flow rates of SF₆ and C₄F₈ and the SF₆/C₄F₈ ratio are the critical parameters for the quality of the Si microcoils. The etching rate was slow when the SF₆ and C₄F₈ flow rates were slow. On the other hand, undercut occurred when fast SF₆ flow rate and large SF₆/C₄F₈ ratio were used (Fig. 3). Since the sidewall width of the designed microcoil was only 8 μm, ICP recipes for small features would be needed. One recipe that was successfully used in these work was 1800 W power, 300 sccm (standard cubic centimeters per minute) SF₆, 150 sccm C₄F₈, 50 W bias power and 20% pressure.

The processing of SU-8, an epoxy based negative photoresist, was another challenge to produce a thin Su-8 coated microcoil with high uniformity. SU-8 was selected in this work as the negative photoresist for microcoil patterning and then acted as a part of the structure. It has high functionality, high optical transparency and is sensitive to near UV radiation. Cured SU-8 is highly resistant to organic solvents, acids, and bases and has excellent thermal stability, making it well suited for applications in which cured structures are a permanent part of the device [4,5]. In this work, after photolithography, SU-8 was left on the wafer to act as one layer of the desired coil structure.

In these experiments, a SU-8 50 from Microposit was used. For the spin-coating process, a 50 μm-thick SU-8 50 layer was spin-coated on the wafer. The photoresist was initially baked for 6 min at 65 °C, followed by a 20-min baking at 90 °C. The initial baking at lower temperature allowed the solvent to evaporate at a better controlled rate, resulting in better coating fidelity, reduced edge bead and better resist-to-substrate adhesion. After baking, the wafer was exposed to near UV (350 nm) for 50 s to cross-link the patterned area. This exposure time need to be precisely controlled. Fifty-second exposure time would provide the designed Su-8 thickness and shape (Fig. 4, left). Over exposure would result in cross-linking of the photoresist adjacent to the exposed area (Fig. 4, right).
Post-exposure bake was performed for 1 min at 65 °C and 8 min at 95 °C to further cross-link the exposed portions of the film. In the next photoresist developing process, the wafer was put into SU-8 developer for 6 min. Strong agitation such as ultrasonic was used to get better developing result.

4. Sensing behavior of the microcoils

It was anticipated that microcoils could expand or contract upon binding of specific species in the environment depending on the surface stress or energy applied on one surface of the microcoil as shown in Fig. 5.

To demonstrate the concept of microcoil-based sensors, we modified the silicon side of a microcoil with a thin film of amines by treating the coil with aminopropyltriethoxysilane according to a typical silica surface modification procedure [6]. As expected, the resistance of the microcoil changed in an acidic environment as shown in Fig. 6, left. Our results showed that the resistance of the microcoil changed from $5.50 \pm 0.01$ to $5.78 \pm 0.01 \, \text{M}\Omega$ upon exposure to a 2.1% acetic acid vapor in air. It is known that when piezoresistive material such as doped silicon is under stress, its resistance changes. The results suggested that the interaction of acids with amino groups on the silicon surface produced changes in free surface energy [7] that extended the coil and resulted in the subsequent resistance change of the coil. Control experiment showed that a microcoil without amine film did not change its resistance upon exposure to acids, ruling out the possible interaction of acetic acid molecules with silicon surface or SU-8 polymer. Another control experiment was performed on an aminopropyltriethoxysilane modified SiO$_2$/Si strip on a wafer. The dimension of this strip was the same as an extended microcoil, i.e. 9.8 mm in length, 50 μm in width. The results showed that the resistance change of the strip was negligible upon exposure to a 2.1% acetic acid. These experiments confirmed the resistance change was caused from surface stress induced microcoil expansion.

Recently, microcantilevers have been proven to be an attractive platform for sensors with on-chip electronic circuitry and extreme sensitivity [8,9]. The microcoil device functions similar to a microcantilever device. We compared the results from microcoils with those of microcantilevers. Since no microcantilevers have been made at 7.7 mm in length, in this work, we used a piezoresistor-on-SiO$_2$ microcantilever we prepared before [10]. The dimensions of these SiO$_2$ cantilevers were 250 μm in length, 100 μm in width, and 1 μm in thickness. The piezoresistor was 100 μm in length and 20 μm in width. The back side of these cantilevers was covered with a thin film of chromium (3 nm) and followed by a 20-nm layer of gold, both deposited by e-beam evaporation. Microcantilevers at this size had been widely used in sensing applications [11]. The $\Delta R/R$ change of this coil to the acetic acid vapor was 5.1%, which was significantly bigger than 0.03% of the cantilever sensor upon exposure to the same amount of acetic acid vapor (Fig. 6, right). The noises over resistance (noise/$R$) of the microcoil and microcantilever were $8.6 \times 10^{-4}$ and $6.4 \times 10^{-5}$, respectively, as shown from Fig. 6. The signal/noise ($S/N$) ratios at equilibrium of the microcoils and microcantilevers were 60 and 8, respectively, i.e. the $S/N$ ratio of the microcoil was approximately 7.5 times that of the microcantilevers. These results suggest two advantages of the microcoils over the microcantilever sensors: (1) the $\Delta R/R$ of the microcoil is large enough to be measured by a simple circuit without amplifiers. This will lower the cost of the device. (2) The $S/N$ ratio of the microcoil is larger than the microcantilever device used in this study, suggesting that the microcoils are more sensitive than piezoresistive approach of the cantilevers used. Lower detection limit for microcoil than microcantilever devices may be expected.

5. Conclusion

The fabrication process of SiO$_2$/Si/SU-8 trilayered microcoils has been discussed. This microcoil device has a potential to be used as a novel microsensor design for the detection of chemical and biological species both in air and solutions.

Acknowledgement

We thank the support from NSF under SGER ECCS-0643193.
References


Biographies

Yanqing Lu received her BS and MS degrees in Electrical Engineering from University of Science and Technology, Beijing, China, in 1996 and 1999, respectively. She is presently a PhD candidate in Engineering at Louisiana Tech University. Her current research interests include micro/nano device simulation and fabrication.

Dr. Hai-Feng Ji received his PhD degree of chemistry from Chinese Academy of Science, China, in 1996. After his graduation, he spent one year as a postdoctoral associate in University of Florida under supervision of Dr. Kirk Schanze, then worked in Oak Ridge National Laboratory between 1998-2000. In 2000, he joined the faculty in the Institute for Micromanufacturing at LaTech. His research interests focus on MEMS devices, surface modification, and nanoassembly. He is currently a co-author of 70 peer-viewed journal articles and book chapters and is one of the most-active researchers in the microcantilever sensors field.