

A Planar Vibration Sensor for Mechanical Characterization of Nanowires

L. Zhang*, J. Lu, H. Takagi and R. Maeda

Research Center for Ubiquitous MEMS and Micro Engineering (UMEMSME), National Institute of Advanced Industrial Science and Technology (AIST), Tsukuba, Ibaraki, Japan

Abstract

Nanowires have attracted considerable interest as the nanoscale interconnects and as the moving elements of both electronic and electromechanical devices. The evaluation of nanomechanical property plays a significant role in the development of new nanowire-based devices. Recently, we are engaged in developing an easy method for nanomechanical measurement by using an in-plane-mode piezoresistive vibration sensor fabricated with less process cost and package difficulties. Theoretical analysis and simulation results suggested that the device is capable of high-sensitive and variety, and it is expected to evaluate mechanical properties of metallic or metallic oxide nanowires.

Key Words: Nanowires, Mechanical Property, MEMS, Piezoresistive Sensor

1. Introduction

Metallic and metallic oxide nanowires have attracted widespread research interests because of their special properties and broad applications in the fields of micro-electromechanical systems and nanoelectromechanical systems. Due to the nanowires can provide a good system to investigate the dependence of mechanical, electrical or thermal properties on dimensionality and size reduction [1,2], they may play very important roles as interconnects and functional units in electronic, optoelectronic, electrochemical, and electro mechanical devices [3–5].

Because of the size effect, the mechanical behaviors of nanoscale materials can be much different with those of their bulk. Physical properties of different nanowires are quite also diverse, by their different characteristics of atomic scale structure, size, and chemistry [6]. Thus, to fully utilize the basic and technological advantages offered by their small length scale and to design and fabricate reliable nanowire-based nanodevices, it is essential to investigate their unique characteristics, like mechani-

cal and electromechanical properties of single nanowire and cluster nanowires buildings. Such knowledge, a clear understanding of the behaviors and mechanical properties of nanowires, will not only help in design and fabrication of next generation sensors and actuators, but also advance our understanding of nanoscale mechanics, coupling of electrical and mechanical properties at the small scale and multi-physics modeling.

Characterizing the mechanical properties of nanowires (including some other nanostructure, such as nanobelts and nanotubes) is a challenge to almost all the measuring and testing devices on this planet. The reason could be elucidated very easily. The nanowires are very small but they can exhibit high yield strengths up to 0.4 GPa and elastoplasticity without working hardening [7]. It is rigorous for measuring the nanowires using the conventional method. For tensile and creep testing, the measured sample should have a relative large size which could be clamped with holder tightly to avoid the slide occurring.

Normally, the mechanical property of nanowire was studied by the atomic force microscope (AFM) [8]. The AFM probe can be used to carry the target single nanowire and measure it. However, due to the operating prin-

*Corresponding author. E-mail: chou-ran@aist.go.jp

ciple, the AFM just can measure the compound character between the nanowire and its substrate, but not the intrinsic properties of nanowire itself. Moreover, for cluster nanowires, because the normal bonding strength and shear bonding strength are up to the value of 10 Ncm^{-2} [9,10], which is much larger than the AFM probe measuring scale. Thus, the testing issue for cluster nanowires is difficult for the AFM functional power.

Recently, we are developing a planar-mode piezoresistive vibration sensor fabricated with less process cost and package difficulties. The vibration sensor is holding the quality of variety and sensitivity, which means the device could be designed and fabricated with different dimension, for covering the testing scale of nanowires' mechanical property. In this paper, we are investigating the planar vibration sensor works an easy method for evaluating the nanomechanical properties of metallic and metallic oxide nanowires, not only the single but also the cluster bonding ones.

2. Mechanical Properties of Nanowire Measurement Principle

Figure 1 shows schematic view of our proposed planar vibration sensor with surface micromachined piezoresistive gauge and the idea for nanomechanical measurement. Using focus ion beam (FIB) the single nanowire (the single nanowire can be fabricated by using the electrode-position method) could be bonded above the boundary slit between the proof mass and Si substrate as the Figure 1(b) and (c) shown, in this way the strength of single nanowire could be measured and studied. This experiment can avoid the sample nanowire manipulation and it is a swift technology to determining the fracture strain of single nanowire. On the other hand, by using the stress-induced growth method [11], nanowires array could be generated at the sidewalls of sensor proof mass and Si substrate as the Figure 1(d) and (e) shown, the normal strength and shear strength of nanowires cluster could be measured and studied.

To measure the mechanical characterization of nanowires, the MEMS device can be designed and fabricated to test the displacement based on the different sensing methods, for example, magnetic, piezoelectric, optical and electrostatic. The methods are under a similar prin-

ciple: the sensor transduce force into displacement via a spring element, following on the equation of force = spring constant $\times$ displacement. Thus, the force resolution in the devices is primarily decided by the spring constant of the testing structure. To enhance the force and displacement resolution, for normal devices, the spring constant of structural units should be decreased with a smaller cantilever or beam. However, this prerequisite will result a trade off in the overall ruggedness, strength and robustness of devices.

In our proposed planar vibration sensor design, we implement the relative large proof mass and relative thin piezoresistor flexure to address the above issue. In this way, detection unit (piezoresistor flexure) can keep the constrain dimension scale with the measure sample nanowires, for maintaining the precision of testing results. Meanwhile, the motivation unit (proof mass of sensor) is much larger than nanowire, which can ensure the ruggedness, strength and robustness of device, and avoid the phenomenon of motivation unit deformation cause by interaction between the sensor and measured sample.

The dimension design of planar vibration is considered fulfilling the testing needsof nanowires. The single nanowire fracture strength and cluster nanowires bonding strength should be control in the sensor testing range. That means the torsional moment of sensor moti-

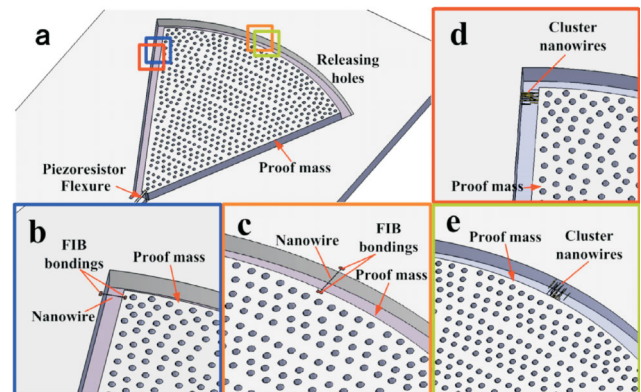


Figure 1. (a) Schematic view of our proposed planar vibration sensor. (b) and (c) Zooming in boundary between the proof mass and Si substrate, the strength of single nanowire could be studied. (d) and (e) Using the stress-induced growth method, nanowires array could be generated at the sidewalls of sensor proof mass and Si substrate, normal and shear bonding strength of nanowires cluster were measured.

vation unit should larger than above mentioned values. For single nanowire, it is obviously and definitely that if the proof mass is with a reasonable dimension, the testing requirement can be fulfilled. On the other hand, for cluster nanowires, because the normal bonding strength and shear bonding strength are up to the range of 5–9.6 Ncm⁻² and the moment value [12] of proof mass is

$$M = \frac{2at\rho r^3 \sin\left(\frac{\theta}{2}\right)}{3}$$

where the a is applied acceleration (1 g), ρ is the density of Si, r is the radius of proof mass, θ is the include angle of the proof mass and t is the thickness of device (with 50 μm). The proof mass generated moment should larger than the effective moment value of cluster nanowires bonding. By solving the equation, under the situation that, the include angle of the proof mass was designed with 60 degree, if the radius of proof mass is larger than 72.16 μm , the planar vibration can be used to measure the mechanical properties of cluster nanowires properly. In order to pursuit the high sensitivity and take the fabricating conditions into account, the width of sensor flexure should be designed and made with 6–8 μm for keeping the proportion with the nanowires dimension.

3. Fabrication of Planar Vibration Sensor and Nanowires

3.1 Planar Vibration Sensor Fabrication Procedure

The fabrication procedure is shown in Figure 2. The device of planar vibration sensor fabrication was done by front-side only process by using 5 photo-masks. The process started from SOI wafer with 0.01–0.02 $\Omega\cdot\text{cm}$ n-type active Si (100) layer and 2 μm -thick SiO_2 box layer. First, the oxide deposition layer approximately 200 nm is deposited by thermal oxide, and the alignment marks and diffusion patterns of piezoresistors are etched by deep reactive ion etching (DRIE), the piezoresistors with resistance of 100–300 $\Omega/\square$, were defined by boron diffusion (see Figure 2(a)). Then, the oxide deposition layer and boron diffusion residue are removed and the implants annealed while a 200 nm thickness passivation oxide is grown in a 950 $^\circ\text{C}$ wet ambient for 60 min.

After the contact holes and patterns were formed on the passivation oxide layer by etching method, the electrode was fabricated by sputtered AlSiCu metal layer, followed by annealing under 450 $^\circ\text{C}$ for 30 min for ohmic contact with piezoresistors (see Figure 2(b)). To avoid device releasing from backside of the wafer by expensive and time-consuming DRIE technique. Front-side releasing was used to suspend the planar vibration sensor. Thus, a amorphous fluoropolymer pattern (CYTOPTM, Asahi Glass Co., Ltd.) was successfully introduced in this work to protect surface components during following the structures release by using DRIE and vapor HF dry process (see Figure 2(d), (e)). CYTOPTM was proved very effective and useful approach for protecting the passivation oxide layer and metallic electrode, dur-

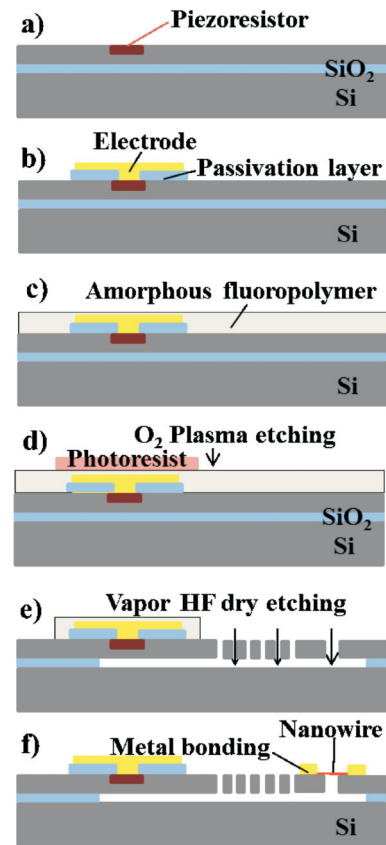


Figure 2. The fabrication procedure of vibration sensor device and measurement unit for nanowire(s). (a) Piezoresistor diffusing on the top surface of n-type SOI wafer. (b) Metallic electrode sputtering and pattern ion milling. (c) and (d) CYTOP-protect film coating and patterning. (e) Device structure and releasing holes etching and vapor HF releasing. (f) Nanowires locating and bonding.

ing the device structure releasing process. Finally, the O_2 plasma was used to remove amorphous fluoropolymer and other residuals. Figure 2(f) shows the measured nanowire bonded above the boundary slit between the proof mass and Si substrate.

Figure 3 shows the typical SEM image of planar vibration sensor structure unit. The planar vibration sensor was fabricated with the front-side release process, the proof mass was supported by one beam flexure. The silicon dioxide sacrifice layer was etched by vapor HF technique, and the proof mass of sensor was keeping the hanging gap with the Si substrate (see Figure 3(c)). The structure was released, and passivation oxide (silicon dioxide) layer and metallic electrode were protected well by amorphous fluoropolymer layer (see Figure 3(b)).

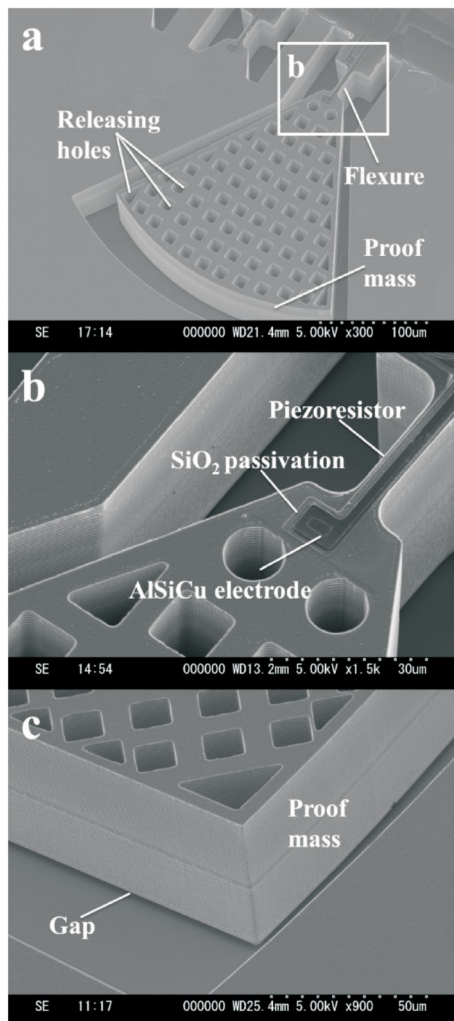


Figure 3. SEM image of planar vibration sensor structure. Fabricated by the front-side release process.

Precision microstructure was successfully obtained by front-side release process, which enables enough high sensitivity for nanowires characterization.

Figure 4 shows the tested I - V curve of the typical fabricated piezoresistor. It is observed that, the piezoresistor survives from vapor HF etching after using amorphous fluoropolymer for surface protection, the I - V character of piezoresistor can keep superior linear in the working scale from -1.5 to 1.5 V for fulfilling the measurement needs.

Owing to the planar vibration sensor consist of a relative large proof mass and a narrow flexure, the flexure attached to the proof mass, and a strain-isolation pedestal attached to the flexure, as illustrated in Figure 1. For getting the resonance frequency of the sensor structure, the vibration sensor is a rigid structure, could be set with solid element in FEA (software ANSYS), and calculated by Block lanczos method. The top end of pedestal is made all constraint, and proof mass and beam flexure keep the non-constraint. Figure 5 shows the calculated frequencies of the designed vibration sensor under the first five modals. The dynamic properties of vibration sensor with different width designed from 4 μ m to 12 μ m were studied with FEA method. The first resonance frequency of the sensor structure, which essentially sets the operating frequency range of the vibration sensor, is around 100 Hz (with different flexure width design) as determined by ANSYS (see Figure 5). It also can be seen that its frequency under the first resonance frequency is much far away that under the second resonance fre-

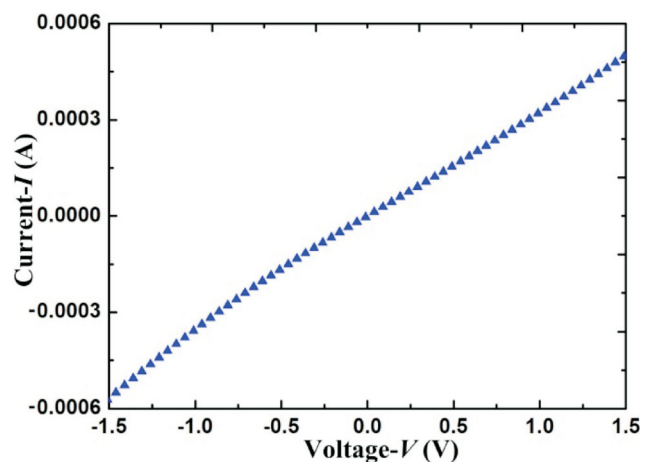


Figure 4. I - V relationship plot of the typical fabricated piezoresistor.

quency, which indicates that the sensor would effectively work in the expected vibration state and had low off-axis sensitivity.

3.2 Fabrication and Preparation of Nanowires

For the single nanowire, commercial alumina membranes (Whatman, UK) with the diameter of 200 nm were used as templates to fabricate Cu nanowires [10]. Before the electro-deposition, backside of the membrane should be coated with a 100 nm thickness metallic layer, which served as the electrode. The copper material was deposited into the pores by electroplating in a 0.4 M CuSO_4 bath, and the current was 5 mA. The Cu nanowires were then separated from the membrane by dissolving the alumina membrane in 2 M NaOH solution and sonication for several minutes. The nanowires were further washed with deionized water and centrifuging at 5000 rpm for 10 min. Using focus ion beam (FIB) the separated single nanowire could be bonded above the boundary slit between the proof mass and Si substrate.

The samples of cluster nanowires from which the copper oxide nanowires grew were fabricated as follows [11]. To improve the adhesion of the Si substrate and the subsequently deposited Cu film and prevent the diffusion of Cu atoms into the substrate. A 60 nm thickness Ta layer was deposited on sidewalls' target areas of sensor proof mass and Si substrate by electrical beam (EB) evaporation technique. The Cu film was then deposited on the Ta layer by EB to form the Cu/Ta/Si multilayer

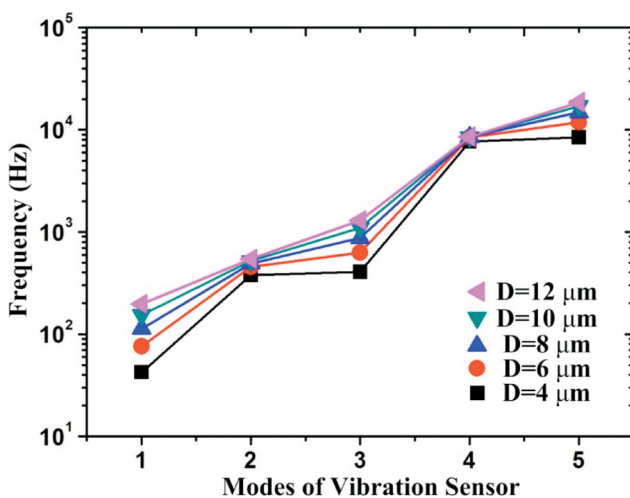


Figure 5. Modals versing frequencies of vibration sensor with different flexure width values, calculated by Block lanczos method.

sample. The typical thickness of the Cu film is 400 nm. The sample was heated on a ceramic heater in air, for produce thermal stress in the Cu film which is needed for stress-induced growth of nanowires. The room temperature was kept at 25 °C. The heating temperature can be adjusted to the required value, by controlling the voltage of the power supply. The heating rate of 66 °C/min was adopted in this experiment, by which the constant temperature of 330 °C was achieved 5 min after turning on the power of the heater. After being heated for 5 hours at target temperature, the samples were cooled in air gradually.

4. Measurement System

Figure 6 shows the schematic of the vibration sensor testing system set up. Using the transduction of dynamical information into the electrical information, the mechanical properties of nanowire could be evaluated out. The computer operate a swept sine control process consists of generating a sine wave output to controller, and excite the device under test. With the control signal input amplifier, by comparing the detected amplitude with the calibration amplitude, the measurement results could be evaluated appropriately.

5. Discussion and Summary

We presented a planar vibration sensor device which can be used to measure the mechanical properties of nanowires with the features of high force and displacement resolution. Also, the measured samples of single nanowire and cluster nanowires could be prepared well by the electro deposition and stress induced growth methods, respectively.

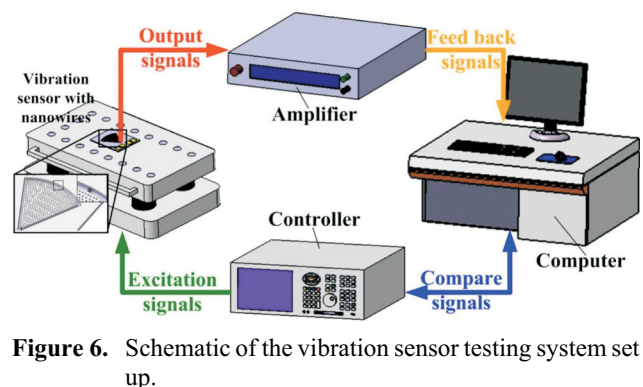


Figure 6. Schematic of the vibration sensor testing system set up.

Based on the I - V curve checking result, the sensor piezoresistor can keep superior linear in the required working scale and fulfill the measurement needs. Our free-manipulation and rapid-testing approach, for measuring the fracture strength of single nanowire and bonding strength of clusters nanowires, is expected to have applications in a wide range of electromechanical measurements.

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