論文の内容の要旨

Development and Application of 3-D Piezoresistive Helical Nanobelt Force Sensor
(3次元ピエゾ抵抗ヘリカルナノベルト力センサの開発と応用)

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In nanomanipulation for nanoelectromechanical systems, measuring the force in nanostructures allows to enable mechanical property characterization for their growth and assembly direction control. Conventional force sensors such as atomic force microscope for this purpose usually require the external optical read-out device which prevents the incorporation with nanomanipulators. Therefore, the force sensor with the features of nano-Newton range, μm displacement range, a few grams weight and arbitrary directional force sensing is required. The objective of this work is to propose 3D helical nanobelt(HNB) force sensors with piezoresistivity which fulfill these requirements. First, The three-dimensional HNBs with metal connectors are self-formed from 27nm-thick n-type InGaAs/GaAs bilayer using rolled-up techniques, and assembled onto electrodes on a micropipette using nanorobotic manipulation. The piezoresistivity of as-fabricated HNBs with metal connectors are characterized using home-made nanorobotic manipulation and measurement system. Coupled axial and bending piezoresistivity of HNB force sensor was emulated to measure force. Then, the nanorobotic assembly processes were investigated to assemble HNB force sensor. Electrostatic and electromagnetic force assisted alignment of HNBs were proposed and experimentally proved to reduce the assembly steps. For the interconnection soldering, gold nanoink deposition method was extended to in-situ scanning electron microscope compatible process. Further extension to chemical-free resistance-spot-welding of HNBs was demonstrated. Both soldering processes were considered as mutually complementary manner in different assembly configurations. As a result, piezoresistive HNB force sensor was assembled and characterized as having the features of nN range force sensing, μm displacement range, a few grams weight and arbitrary directional force sensing. The proposed HNB force sensor is expected to be the promising force sensor in nanomanipulation which meets the requirements described in the objective of this dissertation.

The outline of this dissertation is the following:

Chapter 1 Introduction

The goal to develop nano-Newton force sensors using HNBs for the nanorobotic manipulation with the required features of large displacement range, light weight, and arbitrary directional force sensing is described.

Chapter 2 Force Sensing for Nanomanipulation

The scope and approach of HNB force sensor in this dissertation is described. The state-of-the-arts nano-Newton force sensing technologies including AFM, optical tweezer, micro needle and etc. are summarized and problems to be applied to nanomanipulation are discussed. Piezoresistive force sensors are chosen as the mechanism to meet the requirements in nanomanipulations. HNBs are described to be promising building blocks for the piezoresistive force sensors with above discussed features.

Chapter 3 Self-scrolled Helical Nanobelts as Force Sensor Element

The principles of self-scrolled HNBs were explained then functionalized HNBs with metal connectors were fabricated for the stable piezoresistivity characterizations with better electric contacts and the electrostatic, magnetic field assist assembly in the Chapter 4 and 5.

Chapter 4 Material Property Characterizaton of Helical Nanobelts

Clear piezoresistivity with the gauge factors, piezoresistance coefficient of as-fabricated InGaAs/GaAs HNBs in the Chapter 3 was experimentally achieved to show its high potential as the nano building blocks to create electromechanical devices such as force sensor. To create the HNB force sensor, the material property characterization of HNBs was performed in Chapter 4. Clear piezoresistivity in axial, bending, rotating directions was characterized using nanorobotic manipulations of as-fabricated HNB elements with metal connectors in the Chapter 3. HNBs showed negative piezoresistivity in full elastic linear range and much higher piezoresistance coefficients (294-890 times higher than the one of conventional Bn doped Si piezoresistors) compared to the 1-D nanostructures such as CNTs, NWs and etc. Through multi axial piezoresistivity characterizations of the HNBs, the HNB force sensor was estimated to show the piezoresistivity to precede to assembly the HNB force sensor in the Chapter 5.

Chapter 5 Nanorobotic Assembly of Helical Nanobelts Force Sensor

A novel field-assisted assembly using electrostatic and electromagnetic field is experimentally investigated. Furthermore the proper soldering method such as gold nanoink deposition using fountain-pen method and its extension to the in-situ SEM soldering and finally to the chemical-free resistance spot welding was successfully demonstrated by assembling the HNB force sensor. As an alignment method of as-characterized HNBs (Chapter 4) with metal connectors (fabricated in the Chapter 3) onto the pipette electrode, external field assisted assembly using electrostatic and electromagnetic force was proposed and experimentally proved to be useful to HNB assembly. In-situ extension of gold nanoink deposition method and further process simplifications by the chemical-free resistance-spot-welding were proposed. We could tell that both alignment and soldering methods reduced the process steps and useful to make ohmic contacts to create HNB force sensors characterized in the Chapter 6.

Chapter 6 Characterization of Helical Nanobelt Force Sensor

HNB force sensor is force calibrated using as-calibrated AFM cantilever using in-situ SEM nanomanipulations. Prototype HNB force sensors using the proposed assembly processes in the chapter 5 and the piezoresistivity measured in the chapter 4 were characterized using as-calibrated AFM cantilevers and nanorobotic manipulations. It proved the proposed HNB force sensor configuration could measure the force based on its intrinsic piezoresistivity. We could confirm the proposed design is eligible to measure the piezoresistivity of the HNBs when the axial force was applied.

Chapter 7 Conclusions

This dissertation is concluded to propose the HNB force sensor based on piezoresistivity. From the characterized HNB`s piezoresistivity in multi axes and the proposed assembly method to create the prototype force sensor, we discuss about the potential of the HNB force sensors to the higher resolution and wide range force sensors.

First, the contributions of this dissertation are to present the 3D piezoresistive HNB force sensor which fulfills the aforementioned requirements for nanomanipulation described in the Chapter 1 and 2. Therefore, the goal was to develop HNB force sensor with the features such as nN force sensing, μm displacement range, light weight in a few g, arbitrary directional force sensing capability.

In this dissertation, HNBs with piezoresistivity sensing mechanism were proposed as the promising elements to fulfill these requirements. Since there were no piezoresistivity property known at the beginning of this dissertation work, material property characterization was inevitable.

Since HNBs have large elastic displacement range (nm ～ few μm), high resistance (MΩ range) and thin film bilayer (～ 27nm), nanorobotic manipulation system (highest positioning resolution 0.25nm) inside SEM with high resistance electro meter (minimum measurable current 1 fA) was constructed as the measurement setup.

For the characterization of the piezoresistivity in HNBs without damaging the thin bilayer, metal connectors were attached to both sides of HNBs. Electric contacts of both sides of HNBs and tungsten probes installed at the end of nanomanipulators were made using EBID. These axial piezoresistivity characterizations of HNBs confirmed that HNBs are promising nanostructures to create the force sensors with nN scale force sensing, a few μm displacement range. Achieved piezoresistance coefficients (π_l^{ρ}) of HNBs were ranged between -9.96 ～ -35.6 [10^{-8} Pa^{-1}]. The value can tell that HNBs are the highest responsive in the piezoresistivity amongst conventional piezoresistors including Si, Bn-Si, CNT and SiNW (the highest π_l^{ρ} was reported as 35 ～ 3550 [10^{-11} Pa^{-1}] in SiNWs for their size effect). From these results, nN range force sensing is within the achievable range in the scope of this dissertation.

By further characterizations in bending piezoresistivity, we could confirm that the proposed triangular design of HNB force sensor can measure the force without the cancelation effect by the coupled piezoresistivity in axial and bending directions. This property to measure piezoresistivity in both axes can also explain the possibility of arbitrary directional force sensing using HNBs.

As was introduced in the Chapter 1, HNB force sensor was developed based on the NEMS-on-a-tip concept to fulfill the light weight and easy integration. Nanorobotic assembly was the only way to create such a force sensor even though it is the time-consuming processes with many failures. Therefore, the electrostatic and electromagnetic field assisted alignments, in-situ gold nanoink soldering and RSW were proposed and experimentally has proven to be useful to reduce the assembly steps and increases success rate when to assemble the HNB force sensor.

Gold nanoink is non-contact method and is able to be used to any type of material layers by adding gold nanoink as the sacrificial layer. But it requires additional gold nanoink pre-deposition step. The RSW is basically the extended method from the in-situ gold nanoink method by eliminating the chemical pre-treatment step. RSW can reduce the nanoink pre-deposition step so it can be easily integrated to other in-situ assembly steps. But it needs mechanical probing by nanomanipulation which should be carefully operated. Force sensing possible mechanical probing can be useful direction in the future. Since it uses intrinsic high contact resistance of two contact materials, some calculations to see the welding possibility on different materials are required. The electromagnetic field benefits to this mechanical probing step by magnetizing the ferromagnetic metal connectors of HNB and the electrode. Both soldering methods were proved to be useful to assemble HNB force sensor. Therefore, it can be said that both methods can be utilized in a mutually complementary manner on different assembly configurations.

For the HNB force sensor assembly, as-fabricated pipette electrodes were mounted onto nanomanipulator. It is approached close enough (a few μm) to the suspended HNB on chip. Electrostatic force was used to align HNB onto the electrode then it is soldered together using the gold nanoink deposition method (in this case pre-deposition of gold nanoink is necessary) or RSW. HNB is released from the chip by breaking the connection using probe. Then, the same process was used to assemble another HNB on the other side of electrode. Once both suspended HNBs are assembled onto the electrodes, electrostatic force was used to close the HNBs then the EBID with WCO6 precursor was used to solder the contacts of HNBs.

The assembled HNB force sensor was mechanically characterized (calibrated as K=0.03125N/m) using as-calibrated AFM cantilever (K=0.132N/m). Linear piezoresistivity curve between the applied force [nN] and resistance change [%] was achieved from the calibration from 13.2 nN to 154 nN. The minimum measured steps was 13.2 nN from the resolution limit of SEM (100 nm) and AFM cantilever stiffness (K=0.132 N/m). Minimum detectable force resolution using individual HNB was estimated as 0.91 nN by considering the standard deviation of the measured noise (0.03nA) which is within the range of the high resistance electro meter (measurable up to 1fA). From these results, we can conclude that the HNB force sensors can measure the force in nN range, arbitrary directional force sensing (axial, bending), mountable weight (～ 5 g) fulfilling the goal set in the Chapter 1 and 2. This dissertation created HNB force sensor which have the potential to the mechanical property characterizations of the CNTs, NWs, and etc. for their device applications.