Nanofixation with Low Melting Metal Based on Nanorobotic Manipulation

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Abstract— **We propose a nanofixation method with melting/solidifications of a low melting metal based on nanomanipulation inside an electron microscope. Indium micro particles are experimentally used as the low melting metal. Formation changes of their melting/solidification are observed by scanning electron microscope (SEM) images. To control the position, temperature and pressure of samples, the nano-manipulation system is constructed inside an environmental-SEM (E-SEM). The nanomanipulators are newly constructed with 6 degrees of freedom (DOFs) and 3 units inside the E-SEM. Bio-cells are experimentally observed on water-containing conditions with non-drying treatments. The individual carbon nanotubes (CNTs) are experimentally fixed by melting/solidification of indium micro particles.**

Keywords- nanofixation; nanosoldering; nanorobotic manipulation; carbon nanotubes; low-melting metal

I. INTRODUCTION

Nanofixation, or the fixation of nanomaterials, is one of the most important technique for Nano-Electro-Mechanical Systems (NEMS). It is also a key technology on the nanomanipulation for nanofabrication and nanoassembly. The important factors of nanofixation are fixation rigidity, low contact resistance, fixation positioning accuracy, operability, fabrication time, and so on.

Carbon nanotubes (CNTs) have been widely investigated to develop NEMS being their extraordinary mechanical and electrical properties. Some of typical nanofixation methods for carbon nanotubes are listed as follows;

(1) Electron-beam-induced deposition (EBID) methods are readily used for nanofixation based on nanomanipulations [1-4]. This method has high operability by electron beams of electron microscope. By specific precursor of various metal-containing gases, micro to nano metal contained deposits can be made for nanosoldering. One of difficulties is high contained metal deposits [5].

(2) Adhesive glues, for example silver glues, are used for the fixations of the bulk nanosamples easily [6, 7]. However, it is difficult to control the high precise fixation sites. And drying time and some impurities might be adverse points for applications.

(3) The liquid metal, such as mercury, is used for the electrical fixation for the measurement of single conductance of multi-walled carbon nanotubes [7].

(4) When the contact area is wide, van der Waals forces have relatively high fixation rigidity for micro to nano scale samples. In previous works, the electrical characteristics of carbon nanotubes are investigated by fixing on Pt wire [8], Au wire [9], and so on. Reducing and stabilizing of contact resistances are important problems to be solved.

(5) Polymer or metallic films are deposited on the nanomaterial after positioning. In previous works, Au film [10], Nb film [11], PMMA film [12, 13] are deposited on carbon nanotubes for certain fixations. Increasing of the fabrication processes and adjusting of the deposition parameters are needed.

In this work, we propose the fixation method with melting/solidifications of a low melting metal based on nanomanipulation. The schematic diagrams of this methodology are shown in Fig.1. By heating the low melting metal, the nanomaterials are embedded with it. Then the metal solidifies by cooling with embedded nanomaterials. Using the low melting metal, the nanomaterials can not be destroyed. Low contact resistance, rigid fixation, and short fabrication time might be realized through this fixation method. By control of the melting/solidification state, pick and place manipulations might be possible. In this paper, the carbon nanotubes are investigated for the fixation experimentally inside an electron microscope.

II. NANOROTIC MANIPULATION SYSTEM INSIDE A ENVIROMENTAL SCANNING ELECTRON MICROSCOPE (E-SEM)

Nanomanipulation is an effective strategy for carrying out the basic property characterizations of individual nano-scale objects to construct nano-scale devices quickly and effectively. We have been constructed hybrid nanorobotic manipulation system integrated a transmission electron microscope (TEM) nanorobotic manipulator (TEM manipulator) and a scanning electron microscope (SEM) nanorobotic manipulator (SEM manipulator) [3, 14]. This system realizes an effective sample

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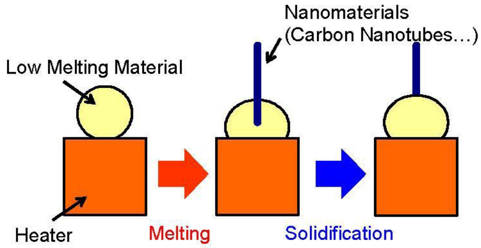

Figure 1. Schematic diagram of the nanofixation using low melting metal.

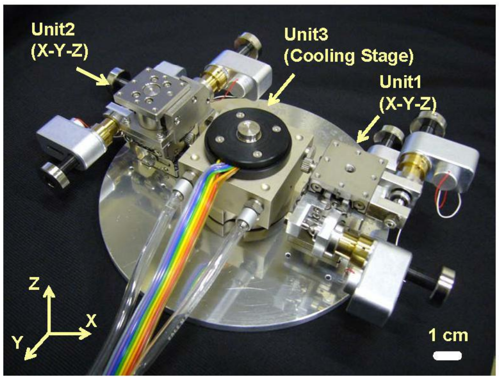

Figure 2. Overview of the nanorobotic manipulator of E-SEM.

preparation inside a SEM with wide working area and many degrees of freedom (DOFs) of manipulation. It also possesses a high resolution measurement and evaluation of samples inside a TEM. The sample chambers of these electron microscopes are set under the high vacuum (HVAC) to reduce the disturbance of electron beam for observation. To observe water-containing samples, for example bio-cells, drying treatment processes are additionally needed. Hence, direct observations of water-containing samples are normally quite difficult through these electron microscopes.

In this paper, we report the new type of nanorobotic manipulators inside an environmental-SEM (E-SEM). The E-SEM can be realized the direct observation of water-containing samples with nanometer high resolution by specially built secondly electron detector. The evaporation of water is controlled by the sample temperature (~0 – ~40 °C) and sample chamber pressure (10 – 2600 Pa). The overview of the constructed nanomanipulator is shown as Fig. 2. It has been constructed with 3 units and 6 degrees of freedom (DOFs) in total. The temperature of sample is controlled by the cooling stage unit, as Unit3. The detail specifications of the manipulator and the E-SEM are listed in Table I. In this paper, following experiments have been done through this system.

III. Observation of Bio-Materials by E-SEM

The unique characteristic of the E-SEM is the direct observation of the water-contained samples with non-drying treatment. Recently the evaluation of bio-samples has gotten much attentions for nanobio applications in nano-biotechnology. Water is an important component for them to keep the life with chemical reactions. Nanomanipulation inside the E-SEM is considered to be an effective tool for a water-containing sample with nanometer resolution.

TABLE I. Specifications of Nanorobotic Manipulation System inside an Environmental Scanning Electron Microscope (E-SEM)

Items	Specifications
DOFs	Unit1, Unit2: 3 DOFs (X-Y-Z) Total: 6 DOFs
Actuators	6 Picomotors™, (Unit1, Unit2)
Work. Space	~ 16 mm × ~ 16 mm × ~12 mm
Positioning Resolution	~ 30 nm (Unit 1, Unit2)
Cooling Stage	Unit3 (Normal Temp. ± 20 °C)
Environmental Scanning Electron Microscope (E-SEM, FEI, Quanta 600)	
Vacuum Mode	E-SEM Mode (10–2600 Pa) Low Vacuum Mode (10–130 Pa) High Vacuum Mode (~10^{-4} Pa)
Acc. Voltage	0.2 ~ 30 kV
Resolution	3.5 nm (E-SEM Mode) 15 nm (Low Vacuum Mode) 3.5 nm (High Vacuum Mode)
Obs. Space	150 mm × 150 mm × 65 mm
Max. Obs. Area	ϕ0.5 mm (E-SEM Mode) ϕ18 mm (Low and High Vacuum Mode)
Detectors	SED, RED

In this work, alga and Escherichia cells are observed with E-SEM. Their E-SEM images are shown in Fig. 3 (a) and (b). The samples were cooled as 0.8 °C by cooling stage Unit 3. The accreted voltage of E-SEM was set 7 kV. The pressure was ~600 Pa under these observation conditions. Decreasing the pressure from ~700 Pa, the water is gradually evaporated and the samples show up from evaporated water.

IV. Fixation of Carbon Nanotubes with Low-Melting Materials

For the fixation of nanomaterials with low melting metal, three basic components are needed for fixation probe; heater, heat-resistant material and low melting metal. The melting point of the low melting material is lower than the heat resistant point of nanomaterials to prevent from destructions. The heat resistance of carbon nanotubes is considered to be almost same with the synthesized temperature (~600 – ~1300 °C). The heat resistance is also deepened on the environmental pressure. High vacuum pressure is better to prevent from destructions.

Figure 4 shows the classified fixation methodologies with basic components of the fixation method. It is mainly divided in two types; integrated and separated types, whether these three components are separated or not. The integrated types (Fig.4 (a) – (c)) are relatively easy to downsize. By positioning the low melting material on the heater, the fixation sites are easily controlled. This method can be readily used as end-effector of nanomanipulator. On the contrary, the separated types (Fig.4 (d), (e)) are relatively easy for control of the ultra-small site fixation area. When laser or electron beam are used for the heating source, nano meter scale local fixations might be possible.

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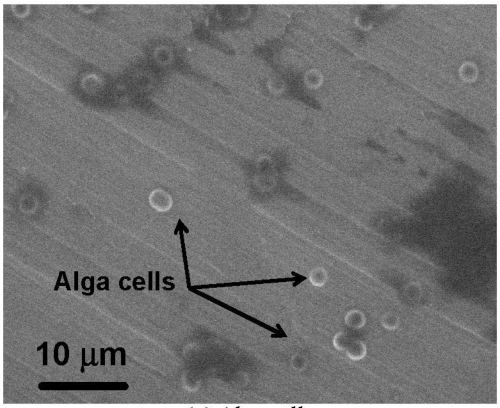
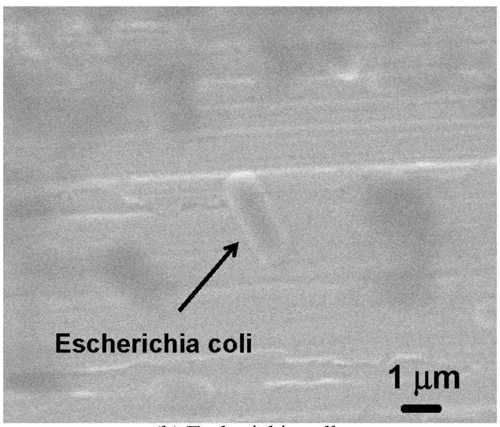


(a) Alga cells



(b) Escherichia cells

Figure 3. The observed bio cells with E-SEM.

This work is investigated on the integrated type as shown in Fig. 4 (c). Indium (The Nilaco Corp., 99.99 %) is used as the low melting materials. This material has a low melting point (~156 °C), low vacuum pressure (1.42×10^{-17} Pa at melting point), and low mechanical property (tensile strength ~3 MPa). Ceramic heater and titanium are used as heater and heat-resistant material.

A. Melting/Soldification of Indium Micro Particles

The formation changes of the indium micro particles are investigated on melting/solidification states from SEM images. The hemisphere shapes of indium micro-particle are self-assembled by heating with small amount indium metal on the ceramic heater. Fig. 5 (a) and (b) show SEM images of before and after heating of the indium micro particles. The pressure of E-SEM sample chamber is set under 10^{-4} Pa. Before heating, the facet shapes have been observed on the surfaces of the solidified indium particles (Fig. 5 (a)). By heating with only ~10 sec, the surfaces are changed to smooth shapes (Fig. 5 (b)). This formation change shows that the indium particles are changed melting state and solidification state. After stop of heating, the indium particles are suddenly returned the facet shapes after approximately 5 min.

B. Fixation of Carbon Nanotubes

The individual multi-walled carbon nanotubes (MWCNTs) are picked up by the indium micro particles. The low melting

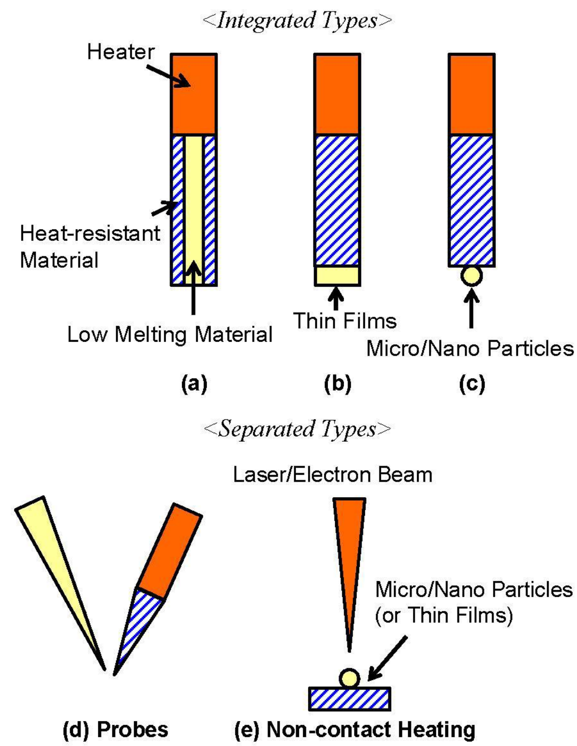


Figure 4. Clasification of the fixiation methods using low melting metal.

metals with heating probes can be used as an end-effector tool base on nanomanipulation. In this work, bulk samples of the MWCNTs synthesized by arc-charge method are used [15].

At first, the indium particles are melted by heating with ~10 sec. During the particles are cooling, the individual CNTs are kept contact with the surface of particles by the manipulator to compensate the thermal drifts. Fig. 6 (a) – (c) show the fabricated CNT probes on the solidified indium micro particle. The CNTs are protruded from the surface of the indium micro particles. At the base of the picked up CNTs, slightly exuded indium is covered. Fig. 6 (c) shows the telescoping carbon nanotube structure, which is partially peeled off its outer layers. This result shows that the fixation rigidity is higher than the tensile strength of carbon nanotubes.

For the future works, the basic characterizations of the picked up CNT probes on the low melting metal will be evaluated, such as contact resistance, fixation rigidity, and so on. The applications will be also investigated, such as the CNT probe emitters, CNT nano indenters for bio-samples.

V. Conclusion

The nanofixitaion method with melting/solidifications of a low melting metal has been proposed. Indium micro particles were used as low melting metal. Formation change of the melting/solidification of the indium particles were determined from SEM images. To control the position, temperature and pressure of samples, nanomanipulation system has been newly constructed inside an E-SEM. Bio-cells were experimentally observed on the water-containing conditions with non-drying treatments. The individual carbon nanotubes were

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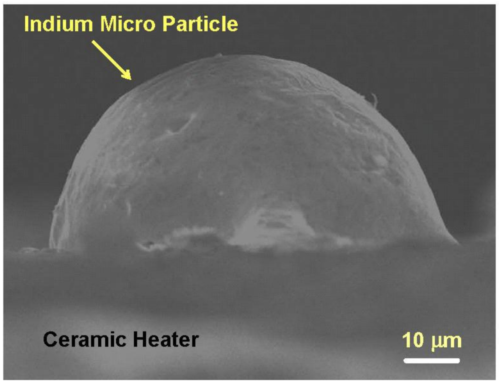
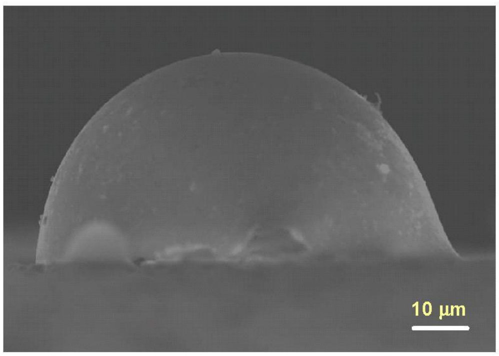


(a) Before heating

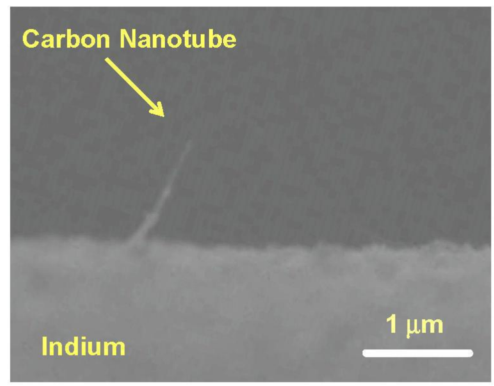


(b) After heating

Figure 5. Melting/Solidification of indium micro particles.

(a) CNT prove No. 1

(b) CNT probe No. 2



(c) CNT probe No.3

Figure 6. Fabricated carbon nanotubes probes on solidified indium micro particles.

experimentally fixed by melting/solidification of indium micro particles. As future works, the detailed evaluations of basic properties and the applications of fabricated nanoprobes will be presented.

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