TOPICAL REVIEW

Nanoelectromechanical device fabrications by 3-D nanotechnology using focused-ion beams

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Abstract
Nanoelectromechanical devices, which can be used as nanotools in nanofactories, were fabricated by focused ion beam chemical vapor deposition (FIB-CVD). The devices are made of diamond-like carbon (DLC), deposited on a Si substrate using gasified phenanthrene (C_{14}H_{10}) as a carbon source. The Young modulus and density of the deposited DLC were measured as 190 GPa and 3.8 g cm^{-3}, respectively. The work function was smaller for DLC (2.9 eV) than for W (4.7 eV) and Fe (5.2 eV) deposited by FIB-CVD. A nanomanipulator was manufactured by FIB-CVD and used for actual manipulations. A glass capillary based local field emitter was developed and produced as a tool for spot deposition, and its electron field emission was confirmed. FIB-CVD is proven as an efficient fabrication technology of novel nanoelectromechanical devices.

Keywords: nanoelectromechanical device, nanotool, focused ion beam (FIB), diamond-like carbon (DLC), manipulator, work function, emitter, Rutherford backscattering spectroscopy-elastic recoil detection analysis (RBS/ERDA), electron energy loss spectroscopy (EELS), Young’s modulus, density

(Some figures in this article are in colour only in the electronic version)

1. Introduction

Many nanomaterials, such as carbon nanotubes [1–6], graphene [7–9] and ZnO nanowires [10–12], have fascinating properties and can be used in nanoscaled devices. However, handling nanomaterials and measuring their properties are difficult so that the handling techniques have not been established yet. Therefore, there is an increasing demand for a ‘nanofactory’, in which various nanoparts are assembled into high-performance multifunctional nanodevices.

To create a ‘nanofactory’ for nanomanufacturing and nanosensing, its individual components (nanotools) have to be developed. The nanotools should be made of various materials; they should have desired characteristics, structure and shape. Two-dimensional (2-D) fabrication techniques, such as electron beam lithography and dry and wet etching can easily produce 2-D components with thicknesses of a few tens of nanometers, but not arbitrary 3-D nanostructures. The latter can be manufactured with focused ion beam chemical vapor deposition (FIB-CVD) [13].

Beam-induced deposition technologies such as FIB-CVD and electron beam (EB)-CVD can create nanostructures at any desired position. They can deposit various materials [14–16] including diamond-like carbon (DLC) [17, 18], tungsten [19, 20] and SiO_{x} [21, 22]. For example, a W/SiO_{x}/DLC heterostructure can be manufactured by altering the gas
1.0 µm
5.0 µm
3.0 µm
DLC
1 min 2 min 3 min 4 min 5 min
(d)
200 nm
Figure 1. SEM images of 3-D nanostructures fabricated by FIB-CVD. (a) W/SiO$_2$/DLC heterostructure, (b) ‘Leaning Tower of Pisa’, (c) ‘bacteriophage’ and (d) in-situ observation of the growth of a 3-D nano-doll by FIB-CVD.

source in FIB-CVD, as shown in figure 1(a). The capability to produce arbitrary 3-D structures is important for nanotool fabrications. Comparing the FIB-CVD and EB-CVD techniques, EB-CVD deposits cleaner materials (no Ga contamination) and has higher planar spatial resolution. However, vertical resolution is superior in FIB-CVD [23] because of the smaller penetration depth of Ga$^+$ ions than electrons at equal accelerating voltage. Furthermore, the deposition rate is higher in FIB-CVD than in EB-CVD [23]. As a result, FIB-CVD can produce such complex 3-D structures as wine glasses, bellows and coils [13]. In this article, we report the fabrication of 3-D nanostructures by FIB-CVD and their applications.

2. Fabrication of 3-D nanostructures by FIB-CVD

2.1. Growth

The deposition was carried out at room temperature, using two FIB-CVD systems SM9200 and SM2050MS2 (SII NanoTechnology Inc.) having the respective minimum beam diameters of ~7 and 5 nm. During FIB-CVD, the chamber pressure was $1 \times 10^{-4}$ Pa. To increase the gas pressure and to equalize the gas density, the systems were equipped with two opposite gas sources (see figure 2). The top parts of the gas nozzles are 300 µm apart and ~150 µm above the Si substrate, and the inner diameter of each nozzle is 0.3 mm. Phenanthrene source gas was produced by heating phenanthrene powder to 90 °C; it was supplied through the nozzles and was continuously covering the Si substrate. The phenanthrene molecules adsorbed on the Si substrate were irradiated by the 30-keV Ga$^+$ ion beam. The Ga$^+$ irradiation produced recoiled atoms and secondary electrons, which had energies of the order of few tens of electron volts. These atoms and electrons dissociated the adsorbed phenanthrene molecules resulting in carbon deposition. By continuously moving the Ga$^+$ beam, a free-standing nanowire can be grown as shown in figure 2. Here, precise control of the beam is essential for producing predefined nanoelectromechanical devices. It was achieved by controlling the beam position and
irradiation time with a 3-D computer-aided manufacturing system [24]. As a result, arbitrary 3-D nanostructures were successfully grown, as shown in figures 1(b)–(d) [25].

2.2. Characterization of deposited DLC

Structure of the carbon deposited on Si from phenanthrene by FIB-CVD was evaluated [26]. The atomic composition of a $5000 \times 5000 \times 0.4 \, \mu \text{m}^3$ carbon film was deduced as $C: Ga : H = 82:4:14$ by Rutherford backscattering spectroscopy combined with elastic recoil detection analysis (RBS/ERDA) [27, 28]. Gallium contamination originates from the Ga$^+$ beam used for the fabrication. The measured hydrogen content (14%) is small compared to typical values of 25–48% for other CVD methods [29–31]. The low H/C ratio of phenanthrene ($C_{14}H_{10}$) is a reason for the low hydrogen content in the deposit. Volatility of some products of phenanthrene dissociation, e.g., CH$_4$, might also reduce the hydrogen content.

A carbon pillar fabricated by FIB-CVD at a beam current of 0.4 pA was studied with scanning transmission electron

![Figure 3. STEM images (120 keV) of carbon pillar fabricated by FIB-CVD: (a) carbon pillar and its electron diffraction pattern, (b) STEM-EDX elemental analysis and (c) STEM-EELS C K-edge spectrum acquired from the center of the pillar.](image)

![Figure 4. Secondary electron images of nanomanipulator fabricated by FIB-CVD on a tungsten probe, without (a) and with (b) applying voltage of 700 V. Panel (c) present a relation between the applied voltage and the opening gap.](image)
Figure 5. Nanomanipulation system integrated into a focused-ion-beam system.

Figure 6. Manipulation of a glass ring: (i) approaching the ring while applying 50 V to the nanomanipulator. The ring is grabbed by switching of the voltage (ii), moved to a Si surface (iii, iv) and released (v) by applying voltage to the manipulator.

microscopy (STEM). Figure 3(a) shows an STEM image of a pillar with a diameter of 120 nm, and the inset presents an electron diffraction pattern. The diffuse character of the diffraction reveals that the carbon deposited by FIB-CVD is amorphous. STEM combined with energy-dispersive x-ray spectroscopy (STEM-EDX) reveals that the pillar has a Ga-rich core (dark part in figure 3(a)), as shown in figure 3(b). The formation of the Ga-rich core can be explained by the growth mechanism of the pillar. The scattering length of Ga\(^+\) ions in carbon is \(~20–30\) nm, and the dispersion length of secondary electrons produced by the scattering of Ga\(^+\) ions is \(~20\) nm. Therefore, Ga-rich core formed by the implantations of Ga\(^+\) ions is covered with carbon shell (bright part in figure 3(a)). The fine structure of carbon pillar was further analyzed by STEM combined with electron energy loss spectroscopy (STEM-EELS). The EELS spectrum of figure 3(c) was recorded by focusing the electron beam at the center of the pillar. This spectrum shows a sharp peak
at 285 eV and a broad feature at 290 eV. They are attributed to the C 1s → π* and C 1s → σ* transitions, respectively, and confirm the DLC structure of the pillar. An sp² ratio of 78% was deduced by a standard analysis of the EELS spectrum [32], which indicates that the deposited DLC has relatively high graphite content.

3. Fabrication of nanomanipulator on W probe

In this study, we have fabricated a 3-D nanomanipulator [33–36], whose Ga⁺ FIB-induced secondary electron image is shown in figure 4(a). The deposition time was 18 min at a beam current of 7 pA. The nanomanipulator has 3-D fingers enabling to firmly grip a target. It is important that the nanomanipulator can be controlled just by a single electrode. The driving force is the electrostatic repulsive force induced by accumulation of electrical charge. Application of voltage to the manipulator opens its fingers, enabling to grab an object (figure 4(b)). The voltage does not need to be applied during the grabbing. Therefore, even a conducting target can be manipulated by the finger without making short circuits. The opening gap between the fingers is shown in figure 4(c) as a function of the applied voltage. The response time of the opening movement was too short to be observed in the secondary electron images. These results demonstrate that the produced 3-D nanomanipulator is a capable manipulation tool.

We have evaluated the properties of the DLC material which constitutes the fingers of the nanomanipulator, such as the Young’s modulus and density [37]. The Young’s modulus was measured by the cantilever contact method [38] using the Hooke’s law. DLC cantilevers were fabricated on the edge of a Si substrate by FIB-CVD, and the Young’s modulus was deduced as 187 GPa. The DLC density was evaluated by the vibration frequency method [39, 40] using an electron beam. This method is a very useful for examining mechanical characteristics of nanomaterials, and it yielded density of 3.8 kg m⁻³ for our DLC. This density is considerably higher compared to other carbon materials: for example, density of diamond is 3.5 kg m⁻³. This high DLC density can be attributed to Ga impurity originating from the Ga⁺ beam.

After the fabrication, the nanomanipulation system was set up in the vacuum chamber of the FIB system. The tungsten probe of the nanomanipulator was connected to a commercial manipulation system (MM3A, Kleindiek Nanotechnik), as shown in figure 5, which was in turn moved by a piezo-device. As a result, the nanomanipulator could freely move in any direction. A test manipulation of a glass ring was performed in the vacuum chamber of the FIB system, as shown in figure 6. The ring was formed by FIB etching of the tip of a glass capillary. The width and outer diameter of the ring were 600 nm and 6 μm, respectively. Using the nanomanipulator, we put the glass ring onto the Si surface as demonstrated by the scanning electron microscopy (SEM) of figures 6(i)–(v): first, we brought the nanomanipulator near the glass ring (figure 6(i)) and applied a voltage of 50 V to the nanomanipulator to open its fingers. Then, we gripped the glass ring by turning off the voltage (figure 6(ii)).

Next, we cut off the support of the glass ring using the FIB beam. Then, we carried the glass ring to the Si surface by moving the commercial manipulator and the stage of the FIB system (figure 6(iii)), and placed the glass ring on the Si surface (figure 6(iv)). Finally, we released the glass ring by applying voltage to the nanomanipulator and withdrew the nanomanipulator (figure 6(v)). This demonstration shows that the performance of the produced 3-D nanomanipulator is adequate for its use in a nanofactory.

4. Glass capillary based local field emitter

We fabricated a field electron emitter on a glass capillary aiming at a nanotool for spot deposition [41, 42]. Such small emitter has a high 3-D degree of freedom, and its electron beam can irradiate in various directions. Work function is the most important parameter of a field emitter. It was measured using photoelectron spectroscopy for DLC, tungsten and iron thin films deposited by FIB-CVD. Gas sources for W and Fe were tungsten hexacarbonyl (W(CO)₆) and ferrocene (Fe(C₅H₇)₂), respectively. Table 1 shows the measured values. There is no significant difference in the work functions of standard W and Fe [44, 45] and of W and Fe produced by FIB-CVD. The work function is smaller for FIB-DLC (2.9 eV) than for FIB-W (4.7 eV) and FIB-Fe (5.2 eV). The work function of FIB-DLC is comparable to those of ZrO/W and LaB₆ field emitters, and it is smaller than that of carbon nanotubes—a potential field emitter. These results indicate that FIB-DLC is a promising material for the fabrication of field electron emitters.

We operated the DLC field emitter in the chamber of a FIB-CVD system, at room temperature, in a vacuum 5.0 × 10⁻³ Pa. A positive voltage was applied to the DLC anode,

| Material | Work function (eV) | Atomic ratio (%) |
|----------|--------------------|-----------------|
| FIB-DLC  | 2.9                | C : Ga : H = 82 : 4 : 14* |
| FIB-W    | 4.7                | W : C : O : Ga = 31 : 54 : 8 : 7** |
| FIB-Fe   | 5.2                | Fe : C : Ga = 26 : 51 : 23** |
| Ga       | 4.3 [43]           |                 |
| W        | 4.58–5.26 [44, 45] |                 |
| Fe       | 4.73–5.05 [45]     |                 |
| ZrO/W    | 2.4–2.9 [46]       |                 |
| LaB₆     | 2.7 [47]           |                 |
| CNT      | 4.5–5.4 [48, 49]   |                 |

Table 1. Work functions of DLC, W and Fe deposited by FIB-CVD and measured by photoelectron spectroscopy. The atomic compositions were measured by RBS/ERDA (*) and SEM-EDX (**). Work functions of several conventional materials are provided for reference.
and a negative voltage was applied to the DLC tip. Figure 8 shows the characteristics of the field emission from the DLC tip. The threshold voltage of the field emission from the DLC tip was 180 V. The linear Fowler–Nordheim (F–N) plot (inset of figure 8) verified that the measured current was the field emission current. These results confirmed the operation of the FIB-CVD field emitter and indicated its validity for nanomanufacturing.

5. Conclusions

In this study, nanoelectromechanical devices were fabricated by FIB-CVD as potential components of a nanofactory. The constituent material, DLC, was deposited on a Si substrate from phenanthrene gas source. The structural characteristics of a DLC pillar were evaluated. The Ga and H contents were 4 and 14%, respectively. The pillar had a core-shell structure attributed to the deposition mechanism in FIB-CVD. The Young’s modulus and density of the deposited DLC were measured as 190 GPa and 3.8 g cm⁻³, respectively.

A nanomanipulator was produced by FIB-CVD and used for manipulation of a glass ring. Also, a glass-capillary-based local field emitter was fabricated as a tool for spot deposition. The work function (2.9 eV) of DLC deposited by FIB-CVD was smaller than of Fe and W metals deposited by FIB-CVD. Electron field emission from the local field emitter was confirmed. The results of this study reveal that FIB-CVD is a very useful technique for fabricating novel nanoelectromechanical devices.

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