D-9-3 Selective Processing of Individual Carbon-Nanotubes Using Atomic Force Microscopy Installed in Transmission Electron Microscope

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1. Introduction

After the discovery of carbon-nanotubes, various types of the application to structural elements or electronic devices have been proposed [1-5]. The development in the processing technology of individual nanotubes has been required to assemble them, while the synthesis and properties have been studied. The bending and fatigue of single- and multi-walled carbon nanotubes have been investigated [6-10]. As the scale of electronic devices decreases further, however, the new conceptual design using nanoscale materials and the nano-processing technology are essential techniques to assemble the nanotubes, fullerenes and other nanometersized elements into the electronic devices. It has been reported so far that individual single-walled nanotubes deposited on a substrate were manipulated by atomic force microscopy (AFM), and conductance of the junction was measured [11-13]. In this study, we manipulate individual multi-walled nanotubes using AFM cantilever inside a highresolution transmission electron microscope (HRTEM), and have performed the cutting and the bonding of their tips under controlled manner.

2. Experimental

Multi-walled carbon nanotubes synthesized by the conventional carbon dc arc-discharge method were bundled and aligned along one direction by drawing sheathed with C₆₀ crystals in a silver tube [14]. A drawn wire was desheathed by heat treatment at 1243K for 54ks, and a rod specimen was obtained. The C60 crystals were changed into amorphous carbon during the heat treatment. The nanotubes were sticking out of the fractured surface of the rod. In order to perform the in-situ observations at the applied electric field, we newly designed the specimen chamber of HRTEM. In this study, we used the HRTEM (JEM-2010, JEOL) equipped with the two specimen holders system shown in Fig.1 [15]. The rod specimen with aligned nanotubes was mounted on a specimen holder. In another specimen holder, silicon (Si) cantilevers for AFM coated with amorphous carbon or gold (Au) was fixed as an opposite electrode. Both specimen holders were inserted in the specimen chamber of the HRTEM with an accelerating voltage of 200 kV. The tips

of the cantilever approached to the nanotube at the distance from 5 mm to 5 nm, and then contacted. The applied voltage between both tips ranged from 0 to 10 V. The structural variation during the contact and retraction was in-situ observed at the lattice resolution of 0.1 nm using a TV rate system.



Fig 1. Schematic illustration of in-situ HRTEM observation system. The specimen chamber is equipped with two sample holders independently.

3. Results and discussion

Figure 2 shows a time-sequence series of the HRTEM images of the contact process between tips of the nanotube (A) and an amorphous carbon (B) at the applied voltage of 5 V. First, two tips (A, B) are separated. The cap of tip A is originally closed. Tip B approaches, and is contacted repeatedly with tip A (Fig.2 (a)-(d)). The cap of the tip A is burst and disappears at the contact. The current measured during the contact is 0.4 μ A. The closed cap of the nanotube A is opened after several contacts as shown in Fig.1 (d). During the contact process, each opened carbon layer of the nanotube A is terminated with the neighboring layer as shown by arrows in Fig.2 (b)-(d). The length of the nanotube is reduced about 1~10nm at one contact, showing that cutting of the nanotubes can be performed by the contact at applied voltage (CAV) method.



Fig 2. In-situ HRTEM images of the nanotube tip during a contact process at applied voltage of 5V. The closed cap of the nanotube A is burst and evaporated by contact, and then the tip A is opened as shown in (d). Each carbon layer of tip A is terminated with the neighboring layer. Arrows show the carbon layer terminated with the neighboring layer.

After several contacts of the Au tip and the nanotubes, the nanotube was bonded at the Au tip at the applied voltage of less than 2 V. The nanotube fixed on the opposite tip was pulled out of the rod specimen by piezo-driving. We could then contact another tip of the nanotube with the other nanotubes. Figure 3 shows a series of HRTEM images of the



Fig 3. In-situ HRTEM images of the bonding process between two nanotubes. The applied voltage is 2V. A larger arrow in (d) shows the tensile stress direction. The expansion of the layer spacing at the tip shown by small arrow demonstrates the bonding interaction between two nanotubes. bonding process. The nanotubes are tightly bonded at the contact when the applied voltage is 2V as shown in Fig.3 (b). The current through junction is 25 nA. The conductance is $\sim 7 \times 10^{-4}$ (2e²/h) (e; electron charge, h; Plank constant). Both tips are separated by the piezo-driving, and the junction is fractured. The spacing between the layers near the junction is spread before the fracture as shown in Fig.3 (d). The bonding-strength of the junction is estimated from the critical bending of the cantilever at the fracture and is ~0.6 MPa. This strength is similar to the exfoliation strength of the graphite layers, i.e. 0.4 MPa. The nanotubes was also bonded with the Si, Au and amorphous carbon at the same voltage. We also observed the melting of Si tip after the contact at 5 V. It shows that the temperature of the junction increased at least up to the melting temperature (~1687K) by Joule heating. It is deduced that the bonding of the nanotubes, i.e., the bonding of the carbon atoms of both tip surfaces, is caused by the heating. For the burst and evaporation of the tips, electromigration will contribute in addition to the evaporation due to the heating. The CAV processing described above can be selected by controlling the bias voltage.

4. Conclusion

We have performed the nano-processing of individual nanotubes by CAV method. By control of the applied voltage between nanotube and the opposite tip, we can cut or bond the nanotube tips. The threshold voltage of the processing is 2 V. Fabrication of nano-structured materials is the essential technology for miniaturization of electronic devices. By using CAV processing as one of the nano-processing, various assemblies of the nanotubes or nano-structured materials are expected.

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