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Small-scale batch fabrication of carbon nanofiber probes

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Abstract. The batch-growth of linear-shaped single CNFs onto commercially available Si cantilevers (3 – 9 chips / batch) using the ion-irradiation method was challenged, and the growth parameters were optimized in terms of the sample temperature (up to 70°C) and the ion-irradiation time. In every growth condition tested here, the batch-fabrication of CNF probes was achieved. The length and the growth direction of CNFs were controllable well with the ion-irradiation time and the ion-incidence angle, respectively. Thus, oriented and linear-shaped single CNFs, even longer than 1 μm in length, were attainable. Under the optimized growth condition, CNFs were batch-grown uniformly in length with the standard deviation of less than ~10 %. CNFs showed almost no temperature dependence in growth rate, and fine CNFs tended to grow for short irradiation duration. Since AFM images of Si grating were attained repeatedly with a good image resolution using CNF probes thus batch-fabricated, the ion-irradiation method was believed to be quite promising to prepare practical CNF probes.

1. Introduction

Due to their high aspect ratios, nanoscale tip radii of curvature, high chemical stability and high mechanical strength, carbon nanotubes (CNTs [1]) and carbon nanofibers (CNFs) are thought to be an ideal probe for scanning probe microscopes (SPMs). Thus, much effort has been devoted to fabricate CNT- or CNF-based SPM probes [2-11]. Typical approaches to achieve this are the manual attachment of a CNT to the tip region of an SPM probe [2-5] and the direct growth of a CNF on an SPM tip by the electron-beam deposition (EBD). In these methods, CNT- or CNF-based probes are prepared by post-processing of commercially available SPM chips. However, the probe fabrication by these methods is time-consuming, because it is done one-by-one. An alternative approach to achieve CNT or CNF probes is the direct growth of CNTs or CNFs on SPM tips by chemical vapor deposition (CVD) [6-11]. This CVD method may be advantageous for the batch fabrication of CNT or CNF probes. This method is, however, generally ineffective for the selective growth of single CNTs on SPM probe tips and has a difficulty in the control of the growth direction. In addition, in the CVD method, the catalyst for the CNT growth must be deposited only on the probe tips. Due to these difficulties, the direct CVD batch-growth of single CNTs on commercially available SPM chips has yet to be achieved. Thus, the batch fabrication of CNT- or CNF-tipped probes is still quite challenging.

In our previous papers, it was demonstrated that Ar^+ ion bombardment on bulk carbon and carbon-coated substrates induced the growth of conical protrusions and CNFs, 20 -50 nm in diameter, grew only on the tips even at room temperature [12, 13]. Moreover, this ion-irradiation method was in principle applicable also to the direct growth of single CNFs on carbon-precoated SPM tips (blade-type tetragonal Si tips; Olympus) [14]. Although the previous fabrication of CNF-tipped SPM probes was made one-by-one using a microbeam ion gun, 350 μm in ion-beam diameter, their batch fabrication should be achievable using a large-scale ion gun. In the present work, we tackled this interesting and practically important subject.

2. Experimental

Samples employed were arrays of commercially available SPM cantilevers, (Figs. 1 (a) and 1 (b); tetragonal-type Si tips (non-blade-type); Olympus). After coating thin carbon layer, they (3 – 9 chips/batch) were ion-irradiated by a Kaufman-type ion gun, 3 cm in ion-source diameter, at room temperature, at 50°C and at 70°C in a nano-fabrication system [15]. The growth mechanism of ion-induced CNFs is described in detail elsewhere [14]. In brief, the CNF growth is due to the redeposition of sputter-ejected carbon atoms onto the side-wall of conical protrusions and the excess surface diffusion of the carbon atoms to the tips during Ar^+ sputtering. The Ar^+-ion-beam energy employed for the ion-induced batch-growth was 600 eV, and the growth duration was 3 to 32 min. The basal and working pressures were 1.5×10^{-5} Pa and 2×10^{-2} Pa, respectively.

After the ion-induced CNF growth, the surface morphology of cantilever tips was carefully observed by a conventional (JEOL; JEM-5600) and a field emission [FE (Hitachi; S-4700)] scanning electron microscopes (SEMs).

3. Results and Discussion

Figures 1 (c) and 1 (d) show typical SEM images of a CNF grown onto an Si tip using the ion-irradiation method. The growth duration (ion-irradiation time) and the growth temperature were 9 min and 70°C, respectively. As disclosed in Figs. 1 (c) and 1 (d), a linear-shaped single CNF, about 800 nm in length, pointing in the ion-beam direction, grew only on the tip. The CNF was almost uniform in diameter, 30 nm, along the growth direction. Ion-induced CNFs grown at room temperature on bulk carbon, semiconductors, metals and plastics are generally characterized by the amorphous nature and possess no hollow structure [13, 15]. Since the growth temperature employed here was low enough, the CNF grown on the Si tip was thought to be featured by the similar crystallographic natures. In addition, as confirmed by the energy-dispersive x-ray spectroscopy installed in the FE-SEM, an ion-induced CNF grown on an Si SPM tip composed of exclusively carbon.

Figure 2 shows a dependence of the length and the radius of CNFs on the growth duration attained at the sample temperature of 50°C. The CNFs increased in length with growth time. After the 32 min irradiation, for example, CNFs reached longer than 1.7 μm in averaged length. Thus, the length of ion-induced CNFs was well-controllable by the growth time. Ion-induced CNFs were linear in shape and almost uniform in diameter along the growth direction. At the initial stage of the CNF growth (< 10 min in Fig. 2), the CNF radius was almost independent of the growth time, ~ 20 nm in average, while the thick CNFs tended to grow for further prolonged growth duration.

The similar growth-time dependence of the CNF length and radius was investigated also at room temperature and at 70°C. At every temperature, CNFs were linear-shaped pointing in the ion beam direction and increased in length with growth time. Both the growth rate and radius of CNFs were almost independent of the growth temperature.

The uniformities in length, diameter and growth direction of CNFs grown under several typical growth conditions were investigated for batch-fabricated chips. Figure 3 shows a typical result on the length and radius uniformities evaluated for 9 CNFs batch-grown at 70°C for 9 min. The growth of ion-induced CNFs was confirmed for all 9 tips batch-fabricated. As shown in Fig. 3 (a), CNFs were distributed from 687 to 931 nm in length, with the mean length and the standard deviation of 770 nm and 80 nm, respectively. The minimum, maximum, mean radii and the standard deviation were 13 nm, 33 nm, 22 nm and 7.1 nm, respectively [Fig. 3 (b)]. It was also found that shorter growth time tended to yield better uniformity in length. For example, CNFs batch-fabricated (8 chips) at 50°C for 32 min were 1.76 μm in the averaged length with the standard deviation of 340 nm.

One of the most important applications for CNT or CNF probes is the precise analysis of deep trenches. For this application, probes should approach the bottom surface of the trench as perpendicularly as possible, in order to avoid the contact to the side wall of the trench with the probe. Thus, the controllability in the growth direction of CNFs is quite important. Since the ion-induced CNFs grow toward the ion beam direction [12-15], the direction control of the CNF probes was achievable quite readily. 9 tips in Fig. 3, for instance, showed a very narrow distribution in the growth direction with the standard deviation of about 2.6 deg.

In order to check the performance of the CNF probes thus batch-fabricated, a standard Si grating (TGX01, NT-MDT) with deep trenches, deeper than 600 nm, was analyzed using tetragonal-type Si probes [see also Fig. 1 (d)] with and without a CNF on the tip (Fig. 4). Compared with the atomic force microscope (AFM) image attained by the Si probe without a CNF on top [Fig. 4 (b)], the superiority in image is manifest in the AFM image obtained by the CNF probe [Fig. 4 (a)]: The sharp edges of the respective pillars and the symmetric structure of deep trenches are clearly observed in Fig. 4 (a), while the observed trench structure is asymmetric in Fig. 4 (b) due to the asymmetric triangle shape of the tip. From a viewpoint of practical applications, SPM probes must possess the sufficient lifetime. The ion-induced CNFs fulfilled this requisite; even after repeated scans for 90 min, almost no degradation in image resolution was detected. Thus, it was believed that the ion-induced CNF was quite promising as a practical SPM tip. We are now trying a larger-scale batch fabrication of CNF probes using this ion-irradiation method, and the detailed results will be given in a forthcoming paper soon.

4. Conclusions

The small-scale batch-fabrication (3 – 9 chips / batch) of CNF probes using the ion-irradiation method was demonstrated and the growth parameters were optimized in terms of the growth duration and the sample temperature. The length and the growth direction of CNFs were well controlled with the ion-irradiation time and the ion-incidence angle, respectively. Under optimized growth conditions, oriented single CNFs were batch-grown onto the commercially available Si SPM tips with an excellent uniformity in length. Since high quality AFM images were attained repeatedly using CNF probes thus batch-fabricated for deep trench structures, it was believed that the ion-irradiation method was quite promising to prepare practical CNF probes.

5. Acknowledgments

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Figure Captions

Fig. 1 (a) Optical microscope image of an array of SPM chips used for the batch CNF growth. (b) SEM image of the cantilever of encircled area in Fig. 1 (a). (c) and (d) Front-view and enlarged side-view SEM images of a typical tip after CNF growth (70°C, 9 min), respectively.

Fig. 2 Dependence of the CNF length and radius on the growth time. Sample temperature: 50°C.

Fig. 3 Uniformities in length and radius. Growth condition: 70°C, 9 min.

Fig. 4 AFM images of a Si grating attained using Si tips with (a) and without (b) CNF.

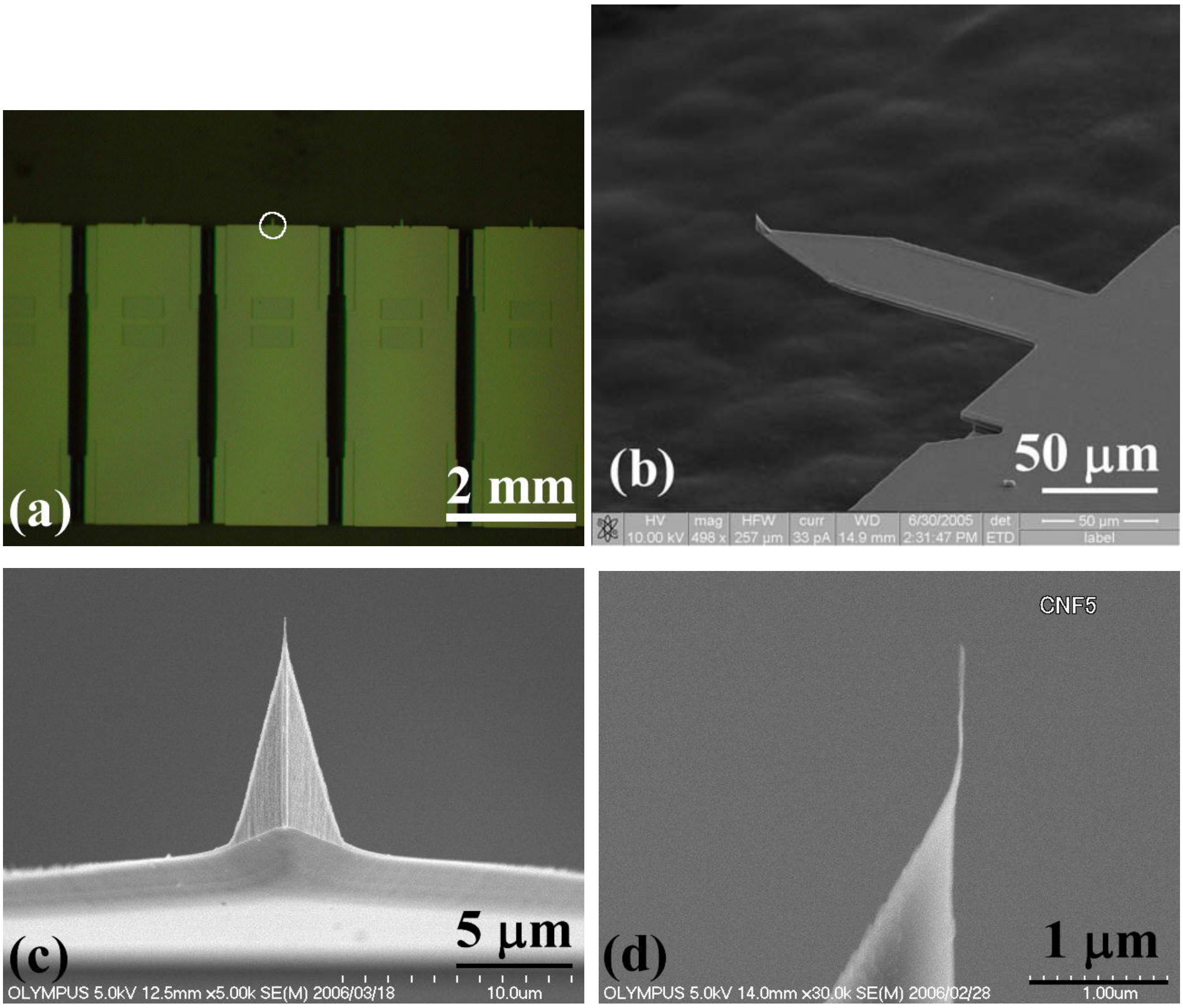

Fig. 1 (Tanemura, et al. CNF Probe)

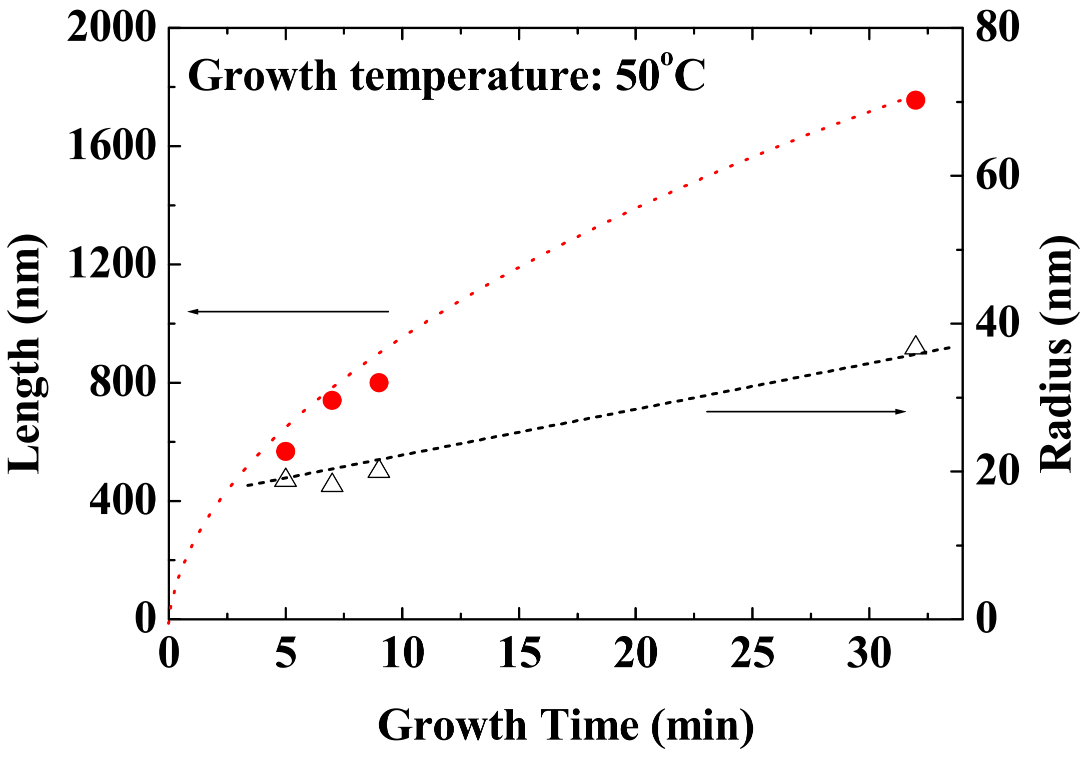

Fig. 2 (Tanemura, et al. CNF Probe)

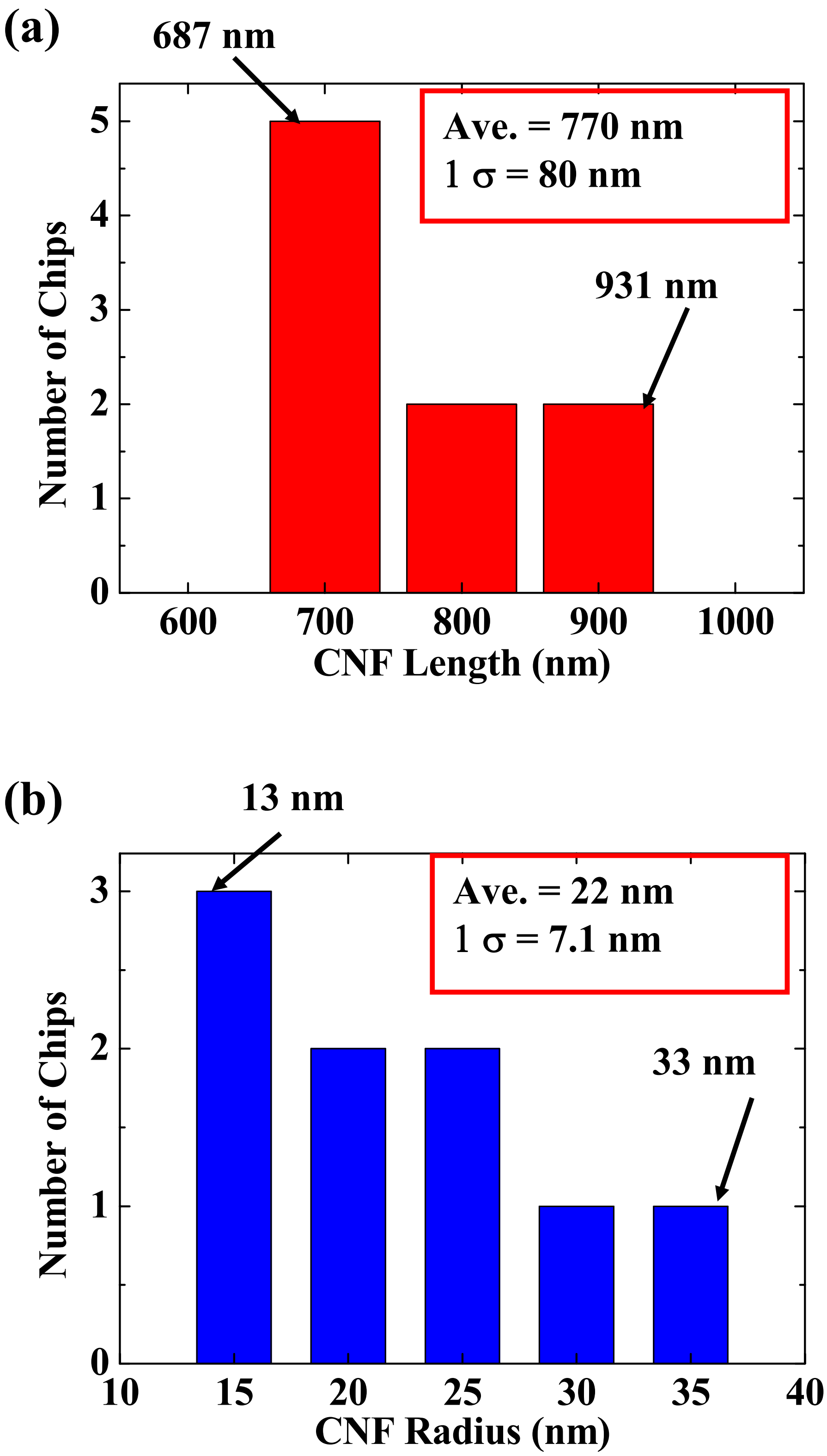

Fig. 3 (Tanemura, et al. CNF Probe)

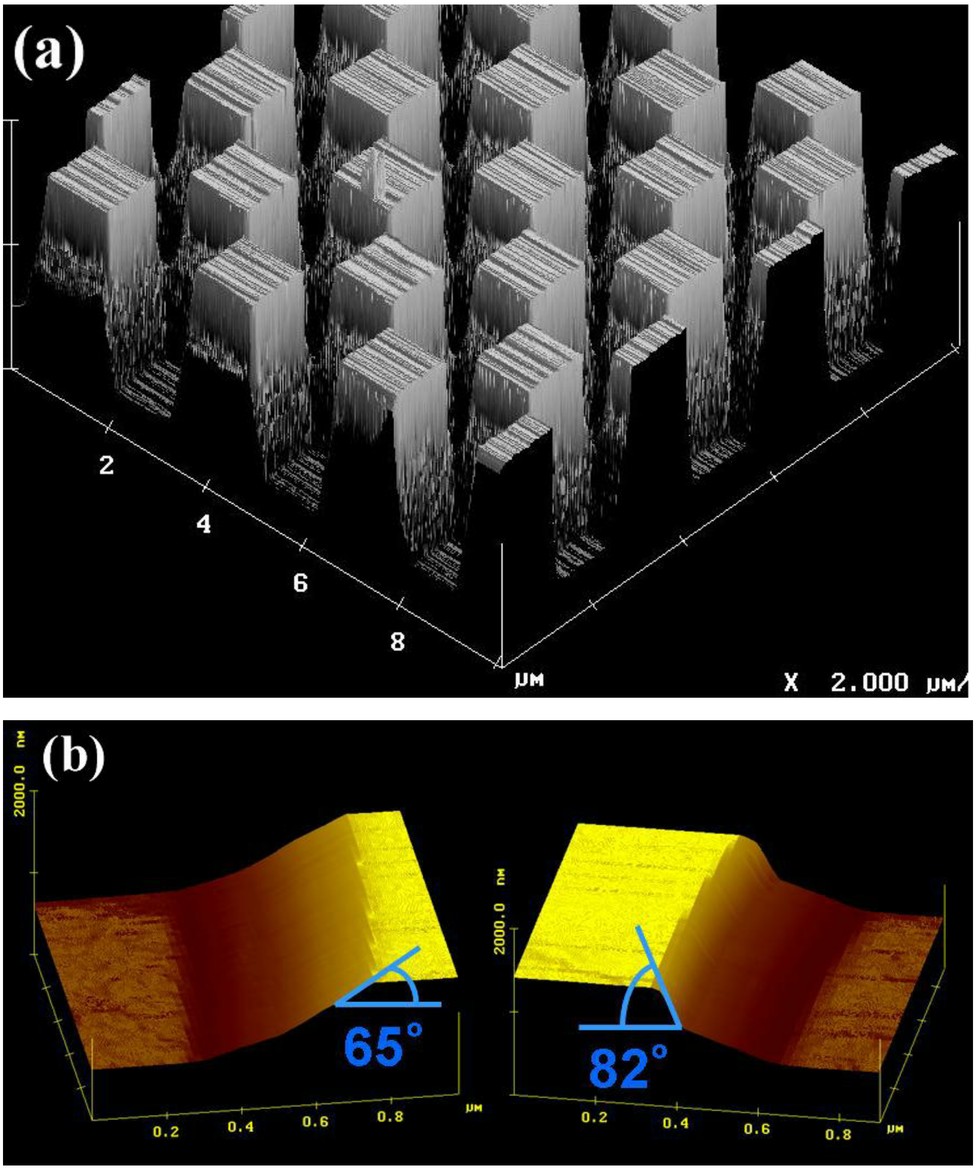

Fig. 4 (Tanemura, et al. CNF Probe)