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Short communication

# *In-situ* nanomechanical study on bending characteristics of individual multi-walled carbon nanotubes

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ABSTRACT

Bending characteristics of individual thin-walled carbon nanotubes (CNTs) are investigated through a novel *in-situ* nanoindentation in transmission electron microscopy. Unlike thick-walled CNTs, the graphitic layers of thin ones buckle into V-shaped kinks rather than Yoshimura ripples. These kinks are found to be entirely reversible without residual plastic deformation following unloading.

## 1. Introduction

Carbon nanotubes (CNTs) have a significant potential for the construction of nanoscale electronic, optical, mechanical and biomimetic devices of high functional density [1–4]. Soon it is found that the electrical performance and the mechanical strength of CNTs will be significantly changed and sometimes even degenerated due to the occurrence of buckle [5,6]. Consequently, avoiding buckling is a fundamental design problem. Iijima et al. [7] examined the formation of a single kink in single-walled CNTs (SWCNTs) with diameters of 0.8 nm and 1.2 nm, respectively. Poncharal et al. [8] and Bower et al. [9] observed the propagation of wavelike ripples in multi-walled CNTs (MWCNTs) during bending. In general, the results presented in [7–9] indicate that the buckling behavior of CNTs is a function of the nanotube diameter, length, and the wall thickness. Zhao et al. [10] reported that thick MWCNTs consisting of layer-stacked graphene experience irreversible buckling under bending due to unfavorable configurations and fractures on the CNT surface.

In fact, the bending induced shell buckling can generate multiple buckling ripples and various instable patterns, such as the Yoshimura formation [11]. Recently, Vaziri et al. demonstrated that the morphology of the deformed pattern is strongly influenced by the geometry of the tube [12]. By a comparison with buckling formations between thin-wall and thick-wall MWCNTs, it is found that more unfavorable and complicated buckling waves may occur at the circumferential direction of thick MWCNTs [13–15]. In contrast, for relatively thin MWCNTs (i.e. wall thickness $<4$ nm), the coaxial graphene shells tend to possess stable and homogenous solid tube and thus undergo great flexibility following buckling deformation [16–18]. As a result, thin-walled CNTs may be better suited for structural design applications; particularly those involving compressive stress and/or repeated bending moments. However, these previous literatures contain comparatively little reference to buckling morphologies and structural evolutions of thin-walled CNTs, particularly the graphene layer-by-layer transformations. Specifically, no studies have directly exhibited the particular case of thin-walled CNTs response beyond the elastic limit threshold or during buckling transition. Here the present study performs an *in-situ* investigation into the force–displacement (*F–D*) and buckling characteristics of an individual MWCNT during bending deformation, in which the greatest advantage of the *in-situ* transmission electron microscopy (*in-situ* TEM) technique is that the entire operation can be displayed live during the mechanical tests, such as examples like nanoparticles [19,20], nanopillar [21,22], nanotubes [17,23], nanowires [24,25] and thin films [26,27] have been investigated. Moreover, high-resolution transmission electron microscopy (HRTEM) observations provide useful insights into the bending-induced buckling instability of MWCNTs.

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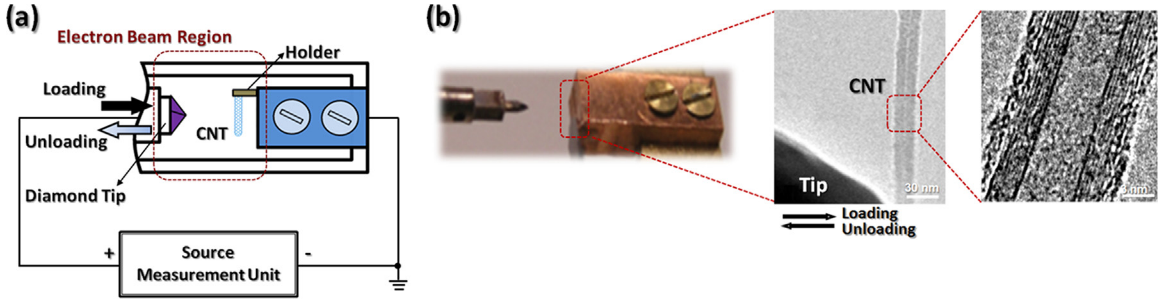

**Fig. 1.** (a) Schematic illustration showing experimental setup of *in-situ* nanoindentation; (b) TEM images showing initial position of the individual MWCNT against diamond tip.

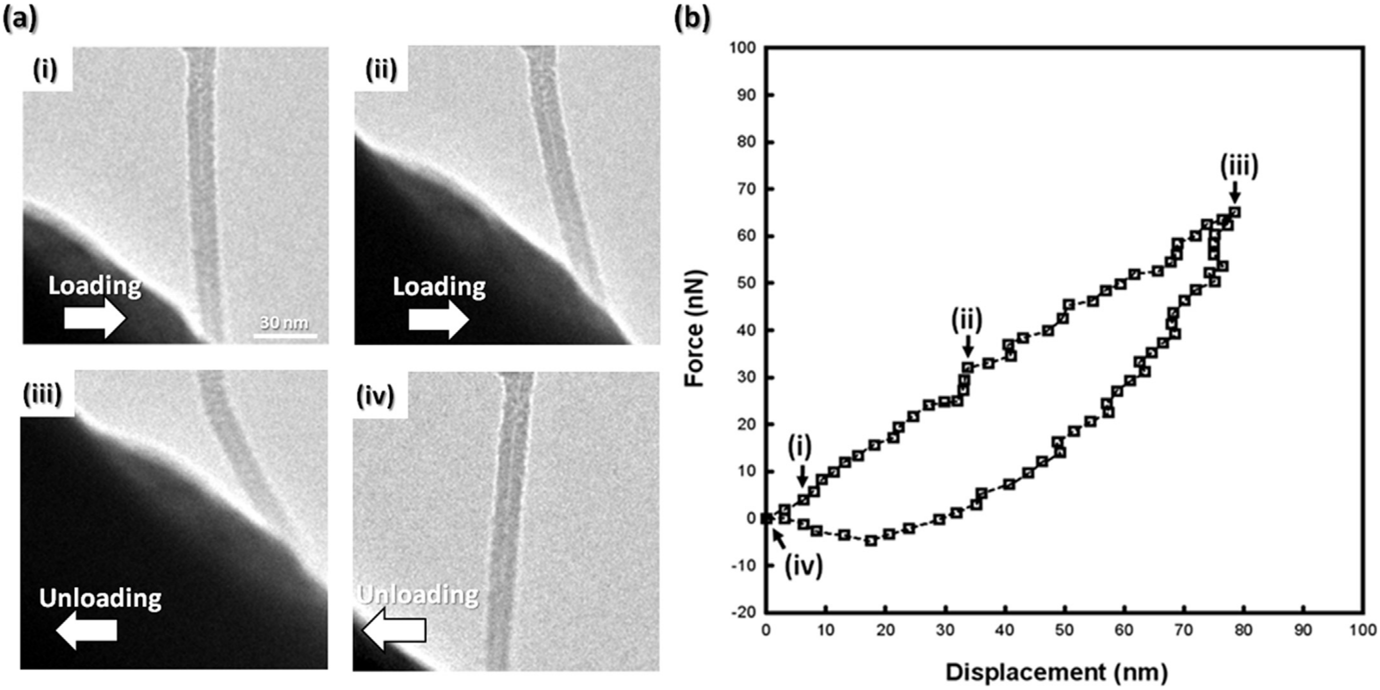

**Fig. 2.** (a) Real-time TEM images showing loading and unloading of individual MWCNT; (b) corresponding *F–D* curve [labels (i)–(iv) refer to TEM images shown in (a)].

## 2. Experimental

Well-structured MWCNTs were grown on top of a specially designed silicon wedge holder by microwave plasma chemical vapor deposition [28]. Fig. 1(a) shows that nanoscale compression tests were conducted inside a JEOL 2010 F transmission electron microscope (TEM) with a field-emission electron source, using an *in-situ* nanoindentation apparatus (PI-95, Hysitron), in which a designed miniature capacitive transducer use a piezoelectric actuator to drive an indenter into a sample with the ability to directly measure force–displacement (*F–D*) curves [29,30]. The compression processes were carried out at a constant displacement rate of 5 nm $s^{-1}$. A nominally flat (100)-oriented diamond tip was brought into contact with an individual MWCNT. Fig. 1(b) shows that the tube axis presents the vertical arrangement before contact. In addition, it also exhibits that the MWCNT had an outer diameter of approximately 15 nm and an inner diameter of around 9 nm. Thus, the wall thickness was equal to 3 nm; corresponding to approximately 10 walls, assuming an interwall spacing of 0.34 nm [31]. Note that normal imaging conditions (a beam current density of ~1–2 A/cm$^2$) were used at 200 kV during all structural observations, taking electron diffraction patterns and electron energy loss (EELS) into account to avoid electron beam damage [32]. All bending processes were videorecorded in real-time using a TV-rate charge-coupled device (CCD) camera with a 30 frames per speed.

## 3. Results and discussion

Fig. 2(a) presents a series of TEM images showing the deformation of the MWCNT during the loading and unloading process. In the initial stages of compression, the CNT undergoes uniform arc-like bending (labels (i) in Fig. 2(a)). However, as the MWCNT is pressed further into contact with the tip, a buckle appears (labels (ii)). This buckle transits to a strain-induced kink as the bending angle is further increased, which may reach about 26° or even more (labels (iii)). However, the nanotube fully recovers to a relaxed state after unloading (labels (iv)). At a certain retraction depth (i.e. 5–20 nm), the onset of adhesive pull-off is induced due to van der Waals forces and/or friction between the indenter and the MWCNT, and its interaction force becomes negative [33,34]. Fig. 2(b) shows the measured *F–D* curve over the loading-unloading process, in which the labels correspond to the TEM images shown in Fig. 2(a). Note that *F–D* curve possesses a hysteresis loop, which is an indicative of the intrinsic viscoelastic nature of the post-buckled configuration. During the loading path, the mechanical compression leads MWCNT to absorb strain energy. As