HRTEM imaging of electron beam irradiation defect dynamics in SWCNTs at 80 kV

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The transmission electron microscopy technique, so far, remains the most efficient for imaging single-wall carbon nanotubes (SWCNTs). Recent advancements in the instrumentation and electron optics of transmission electron microscope (TEM) has resulted in the possibility of achieving high resolution imaging at low accelerating voltages of 80 kV. This is below the theoretically estimated knock-on damage voltage of carbon in SWCNT [1]. A knowledge of the inherent electron beam induced irradiation damage incurred while imaging is of fundamental significance for the precise discrimination of pristine SWCNT features from those due to beam damage. Owing to the novelty of high resolution imaging at 80 kV, relatively little work has been reported in the literature on electron beam-SWCNT interactions below knock-on damage voltages. In this study, time-sequence high resolution TEM imaging of SWCNTs under constant imaging parameters was performed to capture the evolution of electron beam irradiation induced defects using an objective lens Cs-corrector equipped FEI® 80-300 TITAN^{3} ™ TEM operating at 80 kV. The observations made in this study reveal that even below the knock-on damage threshold voltage (86 kV) the SWCNTs are susceptible to apparent irradiation induced structural changes during their imaging in TEM.

A comprehensive investigation of defect dynamics in SWCNTs was carried out using the following strategy. A sparse amount of as-synthesized SWCNTs were transferred to a lacey carbon coated copper TEM grids. A series of time-sequence images with constant time lapse of 5s between each frame were captured from each SWCNT of interest. The post image processing was done using fast fourier transformation (FFT) filtering of the region of interest (ROI) of each individual image in a time-sequence series set followed by inverse FFT to enhance the contrast of the ROI in each image. The band pass mask of Gatan® Digital Micrograph™ software was used for this process. The image processed individual frames were compiled into time-sequence movies for better visualization and analysis of defect dynamics evolution.

We observed the spontaneous self-healing and self-formation of defects in SWCNTs at 80 kV accelerating voltage. The defects observed, can be broadly classified into tube wall distortions, vacancy holes/tubes [2], and hillocks [3]. Under continuous electron beam irradiation during imaging , the thermodynamically unstable defect features are highly mobile due to the elastic and inelastic interactions between impinging electrons and carbon atoms. The combination of achievable high resolution and low accelerating voltage of 80 kV enables imaging of defect migrations assisted by either diffusion of adatoms, interstitials or stone-wales bond rotations [3] in SWCNTs. The defects formed during the synthesis were observed to govern the behavior of the defect dynamics during the TEM imaging. The dynamics of formation of hillocks and tube wall distortions can be observed in Fig. 1. On the contrary, the healing of tube wall hillocks through dissipation of carbon into vacuum could be successfully imaged as shown in Fig. 2. As theoretically predicted [4], the natural tendency of SWCNT lattice structure to spontaneously retain its structure through the of some carbon atoms was

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experimentally observed. The aberration correction assisted high resolution imaging at 80 kV provides more insight into the defect dynamics in SWCNTs, which are difficult to observe at higher accelerating voltages above the knock-on damage voltage. This work shows the possibility of imaging underlying atomic scale defect mechanisms as their understanding is necessary for successful nano-engineering.

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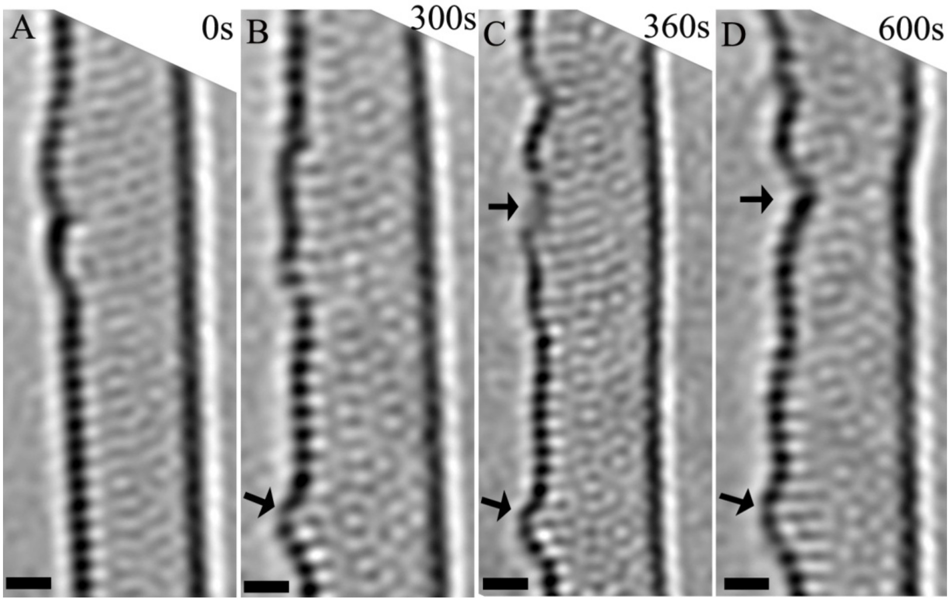

Figure 1. Time-sequence images showing the formation of hillocks (B) (arrow) and tube-vacancies (C) (arrow) and kinks (D) (arrow) due to electron beam irradiation during imaging at 80 kV. Scale bar is 1 nm.

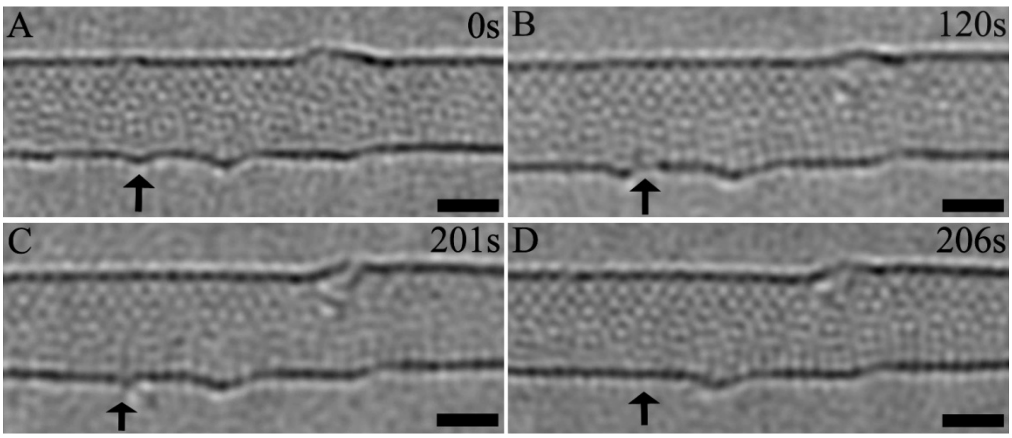

Figure 2. Time-sequence images showing the healing of the hillock indicated by the arrow(A), the dissipation of carbon for healing (B) , intermediate step of wall closing (C) and completely healed hillock defect site (D) due to electron beam irradiation during imaging at 80 kV. Scale bar is 1 nm.

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