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Effects of irradiation-induced structure evolution on the adhesion force and instantaneous modulus of multi-walled carbon nanotube arrays

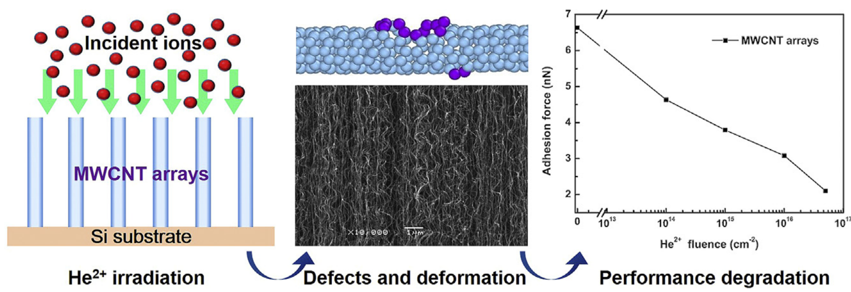
Jian Liu [a], Xiaobin Tang [a, b, *], Yang Li [c, d], Zhendong Dai [c], Feida Chen [a], Hai Huang [a], Huan Li [a], Huiping Liu [e], Da Chen [a, b]

[a] Department of Nuclear Science & Engineering, Nanjing University of Aeronautics and Astronautics, Nanjing 210016, China
[b] Jiangsu Key Laboratory of Nuclear Energy Equipment Materials Engineering, Nanjing 210016, China
[c] Institute of Bio-inspired Structure and Surface Engineering, Nanjing University of Aeronautics and Astronautics, Nanjing 210016, China
[d] College of Material Science and Technology, Nanjing University of Aeronautics and Astronautics, Nanjing 210016, China
[e] Institute of Modern Physics, Chinese Academy of Sciences, Lanzhou 730000, China

HIGHLIGHTS

- MWCNT arrays were subjected to 540 keV He^{2+} irradiation under various fluences.
- Raman spectra, SEM and TEM images were used to study the morphology of MWCNTs.
- The adhesion force of MWCNTs linearly decreases as the irradiation fluence increases.
- The instantaneous modulus has a peak value with increasing irradiation fluence.

GRAPHICAL ABSTRACT



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ABSTRACT

In consideration of the ubiquity of swift particles in space environment, understanding the effects of ion irradiation on the adhesion force and mechanical properties of multi-walled carbon nanotube arrays is significant to apply the arrays as adhesive materials in space in the future. In this study, multi-walled carbon nanotube arrays prepared through chemical vapor deposition were irradiated by He^{2+} under different fluences and then characterized via multiple methods. The arrays were severely damaged when the fluence reached or exceeded 1×10^{16} cm^{-2}. As the fluence increased, more amorphous carbons were generated from their original position in the carbon nanotubes. The adhesion force quickly decreased when the irradiation fluence rapidly increased, which limited the service life of the arrays as adhesive materials. Finally, the instantaneous modulus of the multi-walled carbon nanotube arrays initially increased and then decreased when the fluence increased because of the different contact modes between the indenter and the samples.

1. Introduction

Multi-walled carbon nanotube (MWCNT) arrays are promising dry adhesive materials [1–3]. MWCNT arrays can adhere to almost

* Corresponding author. Department of Nuclear Science & Engineering, Nanjing University of Aeronautics and Astronautics, Nanjing 210016, China.
E-mail address: tangxiaobin@nuaa.edu.cn (X. Tang).

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any object because their adhesion force is generated by van der Waals force [4,5]. Given this advantage, MWCNT arrays have attracted increasing attention as future adhesive materials in space, where conventional adhesion methods do not work because of the microgravity environment. As adhesive materials, MWCNT arrays should also be of enough mechanical strength to bear an external force [6]. However, collision of the high energy rays and swift particles in space with the surface atoms of MWCNT arrays is expected to cause massive defects and structure deformation on the arrays [7,8].

Many scholars have investigated the adhesion force and mechanical properties of MWCNT arrays. Zhao et al. [9] and Qu et al. [10] investigated the adhesive strengths in the normal and shear directions. Hou et al. [11] and Lu et al. [12] studied the mechanical properties of carbon nanotubes via different methods and under different deformation rates, respectively. Buchoux et al. [13] and Li et al. [14] employed atomic force microscopy (AFM) to research both the mechanical behavior and adhesion energy of carbon nanotubes on various substrates. In recent years, some researchers have proved simulation a feasible way to study the adhesion force and mechanical strength of carbon nanotubes. Buldum [15] studied the adhesion behavior and characteristics of carbon nanotubes via molecular dynamics (MD) simulation. All these scholars have found that the MWCNT arrays are well performed in mechanical behaviors and their adhesion force is around 80 N/cm^2 on average. Meanwhile, some scientists have conducted experiments and simulations to study the damaging effect of irradiation on the structure of MWCNTs. Lehtinen et al. [16] researched the effects of different ion irradiation intensities on MWCNTs through transmission electron microscopy (TEM) and Raman spectroscopy. Rui et al. [17] and Yang et al. [18] adopted high-resolution scanning electron microscopy (SEM) and Raman spectroscopy to study the mechanism of structure change of MWCNT films after 170 keV and 3 MeV proton irradiation, respectively. Krasheninnikov et al. [19] conducted a MD simulation on Ar^+ irradiation of MWCNTs and investigated the resulting defects and structure transformation. Xu et al. [20] also utilized MD to study the damage production in carbon nanotubes irradiated by various ion species. All the above-mentioned studies have proved that ion irradiation will surely cause damages to MWCNTs and leave deformed MWCNTs with defects and amorphous carbon behind.

These investigations have provided information on the adhesion force of perfect MWCNT arrays and on the effects of ion irradiation on MWCNTs. However, the adhesion force and mechanical properties of MWCNT arrays with defects and deformed structures induced by ion irradiation, which is inevitable in space, remain to be characterized. Defects and abnormal structure may significantly influence the adhesion force and mechanical strength of MWCNT arrays. Thus, the effects of ion irradiation on the adhesion force and mechanical properties of MWCNT arrays must be explored. Accordingly, the present study investigated the effects of ion irradiation on the morphology, adhesion force, instantaneous modulus, and mechanical properties of MWCNT arrays.

2. Experimental

2.1. Materials

MWCNT arrays were prepared by chemical vapor deposition. In particular, electron beam deposition was utilized to coat a 10 nm-thick Al_2O_3 layer as the buffer layer on the Si wafer, after which a 1 nm-thick Fe catalyst layer was coated on the buffer layer. The coated substrate was placed in a tube furnace (OTF-1200, KJMTI, China). Under protective Ar atmosphere at 720 °C, the MWCNT arrays were grown by flowing C_2H_2 as the carbon source and H_2 as the reducing atmosphere. After the growth period, the C_2H_2 and H_2 flow were cut off and the whole system was cooled down in protective Ar atmosphere. The height of the MWCNT arrays was controlled by adjusting the reaction time, usually 10–30 min, in the tube furnace. Finally, MWCNT arrays were obtained with a height of approximately 200–500 μm, a diameter of approximately 8 nm, and approximately five to seven layers of walls.

2.2. He^{2+} irradiation

Room-temperature He^{2+} irradiation experiments were performed at the Institute of Modern Physics, Chinese Academy of Sciences. Helium ions were generated on a 320-kV ECR platform for multi-discipline research with highly charged ions. To protect the whole irradiation equipment and investigate helium ions with energies of over 500 keV, the combination of He^{2+} and 270 kV acceleration voltage was chosen, thereby producing a He^{2+} energy of 540 keV. After He^{2+} was converted to an ion beam and was stabilized, the beam intensity was set to be constant and the fluence rate was set to 5.6×10^{12} cm^{-2} s^{-1}. The irradiation fluences were set to 0, 1×10^{14}, 1×10^{15}, 1×10^{16}, and 5×10^{16} cm^{-2}. The direction of the ion beam was along the MWCNT axes from the top of the array to the substrate and all the MWCNTs in the array were exposed to the ion irradiation.

2.3. Characterization methods

For microscopically characterizing the integral structure of the MWCNT arrays before and after irradiation, the morphology of the arrays was acquired by a high-resolution SEM (JSM-6160-LV, JEOL, Japan) with an acceleration voltage of 5.0 kV. Under such low electron acceleration voltage, the electron irradiation damage to the arrays can be ignored.

The Raman spectra of the MWCNT arrays were obtained on a Confocal Laser Micro-Raman spectrometer (LabRAM HR, HORIBA, Japan) using an Ar^+ laser with a wavelength of 514 nm to determine the hybridization form of the orbit of the C–C bond. The Raman spectra statistically show the structure of the MWCNTs within the area detected by the laser beam.

The MWCNT arrays were further examined in TEM (Tecnai G2 F20 S-TWIN, FEI, USA) with an acceleration voltage of 200 kV. The electron irradiation dose was maintained as low as possible by controlling the sample exposure time within 5 min to minimize the effects of electron irradiation. As a complementary characterization method of Raman spectra, TEM can reveal the specific structure of several MWCNTs.

The microscopic adhesion force of the arrays was acquired through AFM (Multimode 8, Bruker, USA) with contact mode in air at room temperature. The standard V-shape cantilever with Si_3N_4 tip was used during the measurement. The nominal deflection spring constant of the cantilever was 0.5 N/m. During the test of microscopic adhesion force, the tip contacted the open end of each MWCNT in the array.

The instantaneous modulus measurements of the arrays were performed on a nanoindenter (Nanoindenter G200, Agilent, USA), whose resolutions for load and displacement were 50 nN and 0.01 nm, respectively, on the nanoindentation test platform at Suzhou Institute of Nano-Tech and Nano-Bionics (SINANO), Chinese Academy of Sciences. The proper choice of an indenter shape is critical and primarily depends on the materials to be tested. Previous studies proved that the load-depth response of MWCNT arrays is similar to that of open-cell, foam-like materials [21,22]. The best choice for this kind of material is a flat indenter. Thus, the same cylindrical, flat-ended diamond indenter with a diameter of 215 μm, which is much smaller than the MWCNT array samples,