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Peel test of spinnable carbon nanotube webs

Noman Khandoker [a,b,*], Stephen C. Hawkins [c,d], Raafat Ibrahim [a], Chi P. Huynh [b]

[a] Department of Mechanical and Aerospace Engineering, Monash University, Clayton, Victoria, Australia
[b] CSIRO Material Science and Engineering (CMSE), Bayview Avenue, Clayton, Victoria, Australia
[c] School of Mechanical and Aerospace Engineering, Queen's University Belfast, United Kingdom
[d] Department of Materials Engineering, Monash University, Clayton, Victoria, Australia

HIGHLIGHTS

- Peel tests of carbon nanotube webs have been conducted.
- Van der Waals energy between carbon nanotubes has been measured.
- The effect of nanotube orientation on Van der Waals energy has been determined.
- Experimental result validity is established by analytical calculations from literature.
- It is shown that energy for crossed nanotubes is lower than that for parallel nanotubes.

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ABSTRACT

This paper presents results of peel tests with spinnable carbon nanotube webs. Peel tests were performed to study the effect of orientation angles on interface energies between nanotubes. In absence of any binding agent the interface energy represents the Van Der Waals energies between the interacting nanotubes. Therefore, the effect of the orientations on Van Der Waals energies between carbon nanotubes is obtained through the peel test. It is shown that the energy for crossed nanotubes at 90° angle is lower than the energy for parallel nanotubes at 0° angle. This experimental observation was validated by hypothetical theoretical calculations.

1. Introduction

Since their discovery carbon nanotubes (CNTs) represent a prominent example of carbon nanomaterial. They have stimulated intense study for potential applications ranging from single nanotube based electronics to elevator cables to orbit, from bio-sensors to composites for aerospace and automotive structure [1]. Individual carbon nanotubes (Single Wall Nanotubes (SWNT) and Multi-Wall Nanotubes (MWNT)) are hollow cylinders of seamlessly folded graphite sheets and a lot of them are required to be assembled to make a macroscopic useful item. Carbon nanotube yarn is such a macroscopic item containing trillions of them. But such applications which aimed at exploiting individual nanotube strength succeeded neither as pure bulk material nor as composites [2,3]. CNT yarn is produced by spinning nanotubes from highly oriented drawable CNT forests [4]. In these drawable forests CNTs tend to interconnect together resulting into nanotube bundles as a consequence of the Van der Waals interaction forces. These inherently weak inter-tube interactions obstruct the utilisation of the individual strength of CNTs at macroscopic scales by allowing slippage of nanotubes within a bundle before large macroscopic stresses are reached. Hence, it points out the need of better understanding the characteristics of inter-tube interactions in order to harness and manipulate the individual nanotube strength in macroscale structures.

The experimental work on quantitatively evaluating nanotube interactions is very limited, which is quite obvious due to technical challenges associated with the nanoscale mechanical manipulation of nanostructures. The innovations and progresses in Atomic Force Microscope (AFM) techniques, in-situ Scanning Electron Microscope (SEM) and Transmission Electron Microscope (TEM) experimental techniques enabled such nanoscale studies to be conducted. However, theoretical studies on carbon nanotube interactions are quite large. Table 1 summarises the results from recent studies on interaction behaviours of carbon nanotubes. However, in these experimental studies the types of specimen materials and the

* Corresponding author at: Monash University, Department of Mechanical and Aerospace Engineering, Clayton Campus, Clayton, Victoria 3800, Australia.
Tel.: +61 3 990 510 88.
E-mail address: Khandoker.Noman@monash.edu (N. Khandoker).

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experimental conditions are wide-ranging. Hence, the obtained results are quite scattered. Moreover, no experimental studies on interactions of nanotubes at different orientation angles have been reported in the current literature.

The macroscale interaction experiments presented in this paper are conducted with carbon nanotube webs. Carbon nanotube web is a network of spinnable CNTs that can be densified into strong sheets which are as thin as 50 nm [5]. In such solid state processing of CNT web and yarn, peeling mechanism is the premier functional detachment technique of CNT bundles from the nanotube forests. Structural models of the zipping unzipping processes of CNTs from the spinnable forests (through shredded cheese like interconnections in a tree model [6] or super aligned CNT pulling model [7]) rely on the peeling mechanism. Hence, the macroscale CNT network interaction behaviour is studied with the well established peel test technique [8,9].

The objective of this paper is to experimentally investigate the interaction behaviour of spinnable CNTs at different orientation angles in macroscale without any physical or chemical bonding between them. The effect of the orientation angle on the interaction behaviour such as interface fracture energy of the spinnable CNT network is studied.

2. Experimental setup

2.1. Sample preparation

The carbon nanotube webs used in this study are prepared from spinnable MWNTs grown as a forest on silicon wafer by the chemical vapour decomposition (CVD) process. This used semiconductor grade Si substrates, with a thermal oxide layer of thickness 50 nm and an iron catalyst coating of 2.5 nm deposited by e-beam evaporation. A 44 mm inner diameter quartz reactor was fed with an acetylene concentration of 2.4% in helium (25 sccm in 1000 sccm He) with a running time of 10 min and temperature of 680 °C. More details of this process are published elsewhere [26]. The vertically aligned MWNTs in the forest can be drawn into a web of CNTs which is the major assessment indicator for the spinnability property of carbon nanotubes [27]. The web was initiated with the sharp edge of a scalpel plunged down into the forest and then by pulling it away perpendicular to the nanotube growth direction. The CNTs from the forest string out behind the scalpel and hold together without any presence of binding agent.

The peel test samples were prepared as symmetrical test specimens by laying CNT webs on an alumina block. Layers of the webs are shown schematically in Fig. 1. Before laying up of webs the alumina block was cleaned and rinsed with hot water and oven dried at 350 °C for 30 min. After cooling down to room temperature the block was attached to the web winding apparatus by a double sided Kapton tape. The web winding apparatus consists of a DC motor and a spindle with winding mechanism specially designed and set up in house for winding spinnable CNT webs. Ten layers of webs were put on the block for the bottom part without any pressure applied on them. The bottom part web is then densified with drops of acetone. Later on a polymer sheet was laid on top of the bottom part at one side of the block which acted as a separator between the top part and the bottom part of the webs. The block was then detached and manually repositioned on the apparatus, suitable approximately for the preferred average orientation angle of the top part. Subsequently ten CNT web layers for the top part were laid without any pressure applied on them and densified with drops of acetone. Very low concentration of acetone was used for the purpose of densification to avoid contamination. Sample specimens were then rested apart for the evaporation of acetone. Spinnable nanotubes tend to stick to each other when they are in contact. Therefore, as the layers are laid, nanotube webs are adhered together. The volumetric density of as produced single layer of web is 0.0015 g/cm^3. The densification process increases this single layer density to 0.5 g/cm^3 [5]. The evaporation of acetone during densification process causes surface tension, affecting shrinkage on each of the web layer thickness to ∼50 nm. Thus density of web is increased and contact is established between the top and bottom adherents except where the polymer separator is placed. As the densified web is a porous material, the contact between these two surfaces is discrete point to point contact in nature. Afterwards the samples were inspected under a digital microscope camera and measurements were taken for the orientation angle, width and effective overlap length of the specimens. From 0° to 90° five sets of orientation angles were intended for experimentation. The orientation angle data for each set presented in the results are the average angles of three specimens.

2.2. Experimental procedure

The peeling experiments were conducted on an Instron tensile testing machine with a static load cell of 2.5 N capacity. These tests were quasistatic in nature. Therefore, 2 mm/min load rate was

Table 1
Interaction properties of carbon nanotubes.

Materials	Method	Interaction	References
MWNT on graphite	Sliding experiment with AFM	Shear stress of 2 MPa	[10]
MWNT shells	In situ TEM experiment with a nanomanipulator	Van der Waals force 9 nN	[11]
MWNT	In situ SEM experiment with a nanomanipulator	Average surface energy of 0.56 N/m	[12]
MWNT	In situ TEM cyclic experiment with a nanomanipulator	Surface energy 0.2 J/m^2	[13]
MWNT	Overlap sliding experiment in TEM	Friction force 0.43 nN	[14]
MWNT on graphite substrate	Experimental nanoscale peeling	Adhesive energy 78 keV	[15]
Cross junction of MWNT and SWNT	AFM tapping mode experiments	Coefficient of friction	[16]
MWNT and different surfaces	Peeling force spectroscopy	Interfacial energy/length: 0.6 pJ/m for polyimide, 1.1 pJ/m for graphite and 1.7 pJ/m for epoxy	[17,18]
SWNT bundle	In situ SEM experimental nanopeeling	Adhesion energy/length: 0.13– 0.16 nJ/m	[19]
DWNT bundle	In situ SEM experiments	CNT – CNT interaction 1.7 ± ± 1.0 nN	[20]
MWNT	In situ SEM shear experiments	Shear forces from 15 nN to 50 nN	[21]
Layers of MWNT	Theoretical study using total potential invariants	Strongest interaction between commensurate tubes with achiral walls	[22]
SWNT with alkanethiols	First principles calculations	Binding energy of −50.58 kcal/mol	[23]
SWNT and methanol	Density functional theory study	Binding energy increased with larger diameter and higher chiral angle nanotubes	[24]
SWNT	Smeared out approximation	Energy for parallel nanotubes is higher than energy for crossed nanotubes	[25]