BCN Nanotubes as Highly Sensitive Torsional Electromechanical Transducers

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ABSTRACT: Owing to their mechanically tunable electronic properties, carbon nanotubes (CNTs) have been widely studied as potential components for nanoelectromechanical systems (NEMS); however, the mechanical properties of multiwall CNTs are often limited by the weak shear interactions between the graphitic layers. Boron nitride nanotubes (BNNTs) exhibit a strong interlayer mechanical coupling, but their high electrical resistance limits their use as electromechanical transducers. Can the outstanding mechanical properties of BNNTs be combined with the electromechanical properties of CNTs in one hybrid structure? Here, we report the first experimental study of boron carbonitride nanotube (BCNNT) mechanics and electromechanics. We found that the hybrid BCNNTs are up to five times torsionally stiffer and stronger than CNTs, thereby retaining to a large extent the ultrahigh torsional stiffness of BNNTs. At the same time, we show that the electrical response of BCNNTs to torsion is 1 to 2 orders of magnitude higher than that of CNTs. These results demonstrate that BCNNTs could be especially attractive building blocks for NEMS.

KEYWORDS: Nanotube, boron nitride (BN), boron carbonitride (BCN), nanomechanics, torsion, nanoelectromechanical systems (NEMS)

Owing to their outstanding physical properties, carbon nanotubes (CNTs) and, to a lesser extent, their inorganic analogue boron nitride nanotubes (BNNTs) have been at the forefront of nanoscience and nanotechnology research for the last two decades. Despite many structural similarities, CNTs and BNNTs exhibit very different electronic properties. While CNTs are either metallic or semiconducting depending on diameter and helicity, BNNTs are insulators, with a bandgap of \( \sim 5.5 \text{ eV} \) largely independent of nanotube dimension and chirality. Both the high electrical resistivity of BNNTs and the lack of synthetic control over CNT parameters determining their bandgap are still significant shortcomings toward the large-scale use of both materials in nanoelectronics. Consequently, exploiting the nearly identical lattice parameters of graphene and hexagonal boron nitride (h-BN), much attention has been devoted lately to the synthesis of composite, ternary boron carbonitride (BCN) compounds, where boron, nitrogen, and carbon atoms are arranged into a honeycomb hexagonal lattice. Indeed, theoretical calculations suggested that BCN layered compounds would exhibit semiconducting properties intermediate between those of C and BN layered materials. Later, experimental work conducted on boron carbonitride nanotubes (BCNNTs)7,10,11 BCN nanoribbons,7 and hybridized BN/graphene6 domains confirmed these predictions. These experiments showed that the bandgap of BCN nanostructures can be continuously tuned by controlling the C/BN ratio, thereby allowing the exploration of a whole spectrum of electronic responses, ranging from metallic and semimetallic, to wide bandgap semiconductors. While the electronic properties of BCN layered structures have been thoroughly studied, their mechanical properties have been notably unexplored so far—quite surprisingly, given the exceptional stiffness and strength of both C15,16 and BN17,18 layered materials. Theoretical calculations predicted that the Young’s modulus of BCNNTs could be as high as that of CNTs3,19 Yet, to date, no experimental study of the mechanical properties of BCN compounds has been carried out. One issue of particular interest is the extent of the mechanical interlayer coupling in multilayered BCN nanostructures. Indeed, it has been shown recently by our group that,

Received: June 10, 2014
Revised: September 11, 2014
Published: September 30, 2014
electromechanical transducers and attractive building blocks CNTs, which would altogether make BCNNTs optimal shown in the past with multiwall CNTs, deformation modes, torsion was chosen because the mechanical mechanics and electromechanics of BCNNTs. Of all Supporting Information for experimental details). We present here the first experimental study of the torsional mechanics and electromechanics of BCNNTs. Of all deformation modes, torsion was chosen because the mechanical response of multiwall nanotubes to an external torque provides a direct measure of their interlayer coupling, as it has been shown in the past with multiwall CNTs,21,22,26,27 WS$_2$ nanotubes,28 and BNNTs.24 Additionally, BCNNT electrical response to torsion is interesting per se because many envisioned NEMS are based on twisting elements, such as nanogyroscopes, nanooaccelerometers, and nanobalances.29 While BNNTs electronic properties have been predicted to be rather insensitive to torsion,30 twisting CNTs resulted in linear or oscillatory conductance variations, respectively, for single-wall31 and multiwall CNTs.32 Here, investigation of the torsional stiffness and strength of BCNNTs revealed that they exhibit a significant interlayer mechanical coupling, which as a consequence makes them up to five times torsionally stiffer and stronger than CNTs. Moreover, about half of the BCNNTs investigated displayed a dramatic change of electrical resistance upon torsion, suggesting that they could be promising building blocks for torsion-based NEMS.

The BCNNT samples employed in this study were synthesized by post-synthesis substitutional C-doping of high quality CVD-grown BNNTs, using an approach previously developed by several coauthors of this paper (see the Supporting Information for experimental details).11 This method has proved to produce a hybrid core−shell BN/BCN structure, with a BN core and coaxial BCN outer shells.13 Such structures were previously shown to display p-type semiconducting behavior, the electrical current flowing mostly through their outer shells.13 Additionally, we hoped that these core−shell BCNNTs would retain the faceted structure of the starting BNNTs,32−34 responsible for their outstanding mechanical properties.24 After their synthesis, BCNNTs were suspended in ethanol, deposited on transmission electron microscopy (TEM) grids, and analyzed using TEM (Figure 1a) and electron-energy loss spectroscopy (EELS, Figure 1b−c). High-resolution TEM (HRTEM) examination of the heterogeneous BCN/BN nanotubes revealed that they had preserved the nonbuckled tube−wall structures (Figure 1a and Figure S1), similar to the starting BNNTs. The most salient feature observed in TEM is the presence of a series of darker regions along the BCNNT walls. Such high contrast areas have been previously observed in BNNTs and are attributed to faceting.25,32,34 Similarly to BNNTs, but unlike CNTs, most BCNNTs are thus expected to display a partially polygonal cross-section. On another level, EELS elemental mapping confirmed the previous findings that carbon atoms are mostly incorporated into the outer shells of BCNNTs (Figure 1b).11 Quantitative analysis of EELS spectra (Figure 1c) indicate an overall carbon content varying from 5 to 10 at. %.

The torsional mechanics and electromechanics of BCNNT were investigated using torsional devices similar to those that we previously used to study CNT,22,26 BNNT,24 and WS$_2$ nanotube28 torsion. Each device consists of a suspended BCNNT clamped between metallic electrodes, with a pedal located on top of its middle section (Figure 2a); they were fabricated using electron-beam lithography, followed by wet etching and critical point drying (see Methods in the Supporting Information for details). While mechanical torsion is achieved by pressing against the pedal with an atomic force microscope (AFM) tip, a bias can be applied across the BCNNT, allowing to monitor its electrical conductance during mechanical torsion.22 Before the torsion experiment, BCNNTs electrical properties were characterized by measuring their DC current−voltage characteristics (Figure 1d). As in line with previous studies,10,11 BCNNTs exhibited I/V curves indicative of a semiconducting behavior, with a differential resistivity in the 10−100 MΩ/μm range.

BCNNT torsion was evidenced by pressing at different points along the long axis of the pedal (Figure 2a).21,22,24 Plotting the linear stiffness as a function of the lever arm showed that the system obeys Archimedes' law of the lever
Figure 2. BCNNT torsional mechanics. (a) AFM tapping-mode image of a suspended BCNNT with a pedal. The red dots along its long axis represent points where the pedal is pressed upon, thus twisting the nanotube. Scale bar: 300 nm. (b) Linear stiffness plotted as a function of the position along the pedal (the first measurement point is set to zero by definition). The red curve represents the best fit of the experimental data, using equation S1 (see Methods in Supporting Information). (c) Effective shear moduli calculated according to the (red) hollow cylinder and (black) solid rod assumptions (see text), plotted as a function of BCNNT diameter. The two dashed blue lines represent reference values: shear modulus of CNTs (440 GPa)\textsuperscript{21,24,28} and BNNT (400 GPa).\textsuperscript{24}

(Figure 2b) and allowed us to determine BCNNT torsional spring constant $\kappa$ for all nanotubes investigated (see Table S1 and Methods in Supporting Information for details). The measurements of $\kappa$ were found to be reproducible within the margin of experimental error.

Then, the degree of torsional mechanical coupling in BCNNTs was evaluated by computing the effective shear modulus $G$ and comparing the results with reference values, as previously done with C, WS\textsubscript{2}, and BN nanotubes.\textsuperscript{21,24,28} Indeed, while the torsional spring constant depends on the geometry of the torsional device, the shear modulus is an intrinsic characteristic of the nanotube that provides a measure for its stiffness. Classical elasticity theory gives $G = 2\pi L / [(\pi r_{\text{out}}^4 - \pi r_{\text{in}}^4)]$ where $L$ is the suspended length of the BCNNT, and $r_{\text{out}}$ and $r_{\text{in}}$ are the nanotube outer and inner radii, respectively.\textsuperscript{21,24,28} We calculated two boundary values for the effective shear modulus, corresponding to two extreme assumptions on the torsional interlayer coupling in BCNNTs. The “solid rod” shear modulus $G_s$ was computed assuming infinite torsional coupling; i.e., all walls are mechanically locked and twist together upon application of a torque, yielding $G_s = 2\pi L / (\pi r_{\text{out}}^4)$. On the other hand, the “hollow cylinder” shear modulus $G_h$ was computed assuming negligible torsional coupling; i.e., the outer layer slips around the inner layers upon torsion. In this case, we have $r_{\text{out}} - r_{\text{in}} = \delta r = 3.4$ Å (the interlayer distance), and then, $G_h = 2\pi L / (4\pi r_{\text{out}}^3 \delta r)$. Comparing $G_s$ and $G_h$ with reference values from the literature enables us to determine which model describes BCNNT torsion best, i.e., to assess the effective number of walls contributing to the torsional stiffness of BCNNTs. Based on the results of Hernandez et al.,\textsuperscript{9} who calculated that the Young’s moduli of BCN\textsubscript{2}N nanotubes were very similar to those of CNTs and BNNTs, one may consider BNNT and CNT in-plane shear moduli to be good comparison elements for $G_s$ and $G_h$ (see Figure 2c).

Figure 2c and Figure S2 display the solid rod and hollow cylinder shear moduli $G_s$ and $G_h$ plotted as a function of the nanotube diameter $d$. It can be seen by comparing $G_s$ and $G_h$ to the reference values that the solid rod assumption underestimates the BCNNT shear modulus, while the hollow cylinder assumption overestimates it. This shows that BCNNTs exhibit a significant torsional interlayer coupling, where several layers, but not all of them, effectively contribute to the BCNNT torsional properties. This represents an intermediate case between BNNT torsion (best described by the solid rod model) and CNT torsion (best described by the hollow cylinder model). On average, we found that $G_h = 960 \pm 490$ GPa, meaning that BCNNTs are on average two to three times stiffer than their carbon counterparts.\textsuperscript{22} Additionally, it should be noted that the C-doping step of BNNTs could have introduced defects in the nanotube outer layers, thereby affecting BCNNT mechanical properties. In such case, the elastic moduli of our BCNNTs are expected to be smaller than those of CNTs or BNNTs, yet our results point out to an even higher interlayer mechanical coupling. Defects could also account for the rather large scatter observed in the data between different BCNNTs. Unlike previous observations on BNNTs,\textsuperscript{24} the effective shear modulus of BCNNTs does not show a clear correlation with their diameter (Figure S2).

Besides their torsional stiffness, we were also interested in probing the torsional strength of BCNNTs. BCNNTs were twisted to large torsion angles, in both directions, by pressing successively on both sides of the pedal. For some nanotubes, a softening was observed as the number of pressing increases, indicating an elastic-plastic transition, which allowed us to determine the BCNNT strength. Other nanotubes proved to be more resilient as their mechanical properties did not decay over time; for these nanotubes, an underestimate of the torsional strength can be determined (Table S1). The torsional strength $\tau_{\text{BCNNT}}$ (calculated for the whole tube) is given by the maximal shear load applied before failure divided by the cross-section area, yielding $\tau_{\text{BCNNT}} = T_{\text{max}} / (\pi r_{\text{out}}^3)$,\textsuperscript{24} where $T_{\text{max}}$ is the maximum torque exerted on the nanotube. On average, we obtained $\tau_{\text{BCNNT}} = 1.1 \pm 0.4$ GPa, which is about three times smaller than the measured torsional strength of BNNTs,\textsuperscript{24} but about three times larger than the CNTs torsional strength.\textsuperscript{22} As expected, a strong correlation was observed between the degree of mechanical interlayer coupling, and the strength of each BCNNT.

The next step of our study of BCNNT torsion was the investigation of their torsional electromechanics, namely, their electrical response to torsion. Samples were mounted into chip carriers and wire-bonded, which enabled us to apply a voltage bias and measure the electrical current while the nanotube was mechanically twisted, and thus to monitor the torsion angle, torque, and electrical resistance simultaneously. The pedal was pressed on about halfway between the BCNNT (torsional axis) and the edge of the pedal, corresponding to lever arms ranging...
from 200 to 400 nm, depending on the device. We can thus consider that torsional effects are dominant over bending effects (see Figure 2a), while limiting the risk of the AFM tip slipping out of the pedal. The input signal was the sum of a DC and AC voltage. The DC bias (typically 2 V) was applied in order to work in a region of higher differential conductance (see Figure 1d) and thus increase the signal-to-noise ratio, whereas an AC bias was delivered by a lock-in amplifier. The AC amplitude (100 mV) was chosen to be much lower than the DC bias and its frequency (1.2 kHz) to be significantly higher than the acquisition rate (loop rate: 0.2 Hz; 512 measurements per loop). The torsion angle is defined as the angle between the plane containing the end of the AFM tip and the nanotube, and the horizontal plane of the sample; the negative angles in the approach curve thus correspond to points before contact (the pedal may be twisted to negative angles in the retraction curve due to capillary forces). Variations of the differential conductance upon twisting are observed for the three BCNNTs.

**Figure 3.** Electromechanical response of BCNNT to torsion. (a−c) Tapping amplitude of the AFM cantilever $A_{meas}$, (d−f) torque $T$, and (g−i) differential conductance $G$ (DC bias: 2 V, AC bias: 0.1 V, $\nu$ = 1.2 kHz), for three representative nanotubes (see also Table S1). Black: trace, red: retrace. Vertical arrows correspond to the first contact between AFM tip and the pedal. The tapping amplitude is plotted as a control: as expected, $A_{meas}$ goes down to zero when the AFM tip and the pedal touch. The torque is calculated as $T = k_C \cdot z_C \cdot X$ and the torsion angle $\phi$ as $\phi = \text{Arctan}\left(\frac{z_P - z_C}{X}\right)$ where $k_C$ is the spring constant of the AFM cantilever, $z_C$ its deflection, $z_P$ the piezo displacement, and $X$ the torsional lever arm (see Methods in the Supporting Information for details). The torsion angle is defined as the angle between the plane containing the end of the AFM tip and the nanotube, and the horizontal plane of the sample; the negative angles in the approach curve thus correspond to points before contact (the pedal may be twisted to negative angles in the retraction curve due to capillary forces). Variations of the differential conductance upon twisting are observed for the three BCNNTs.

Both an increase and a decrease of conductivity upon torsion were observed.

Although electromechanical responses to torsion had been previously observed in CNTs, the electrical response of BCNNTs to torsion is remarkable by its magnitude. While the conductance of multiwall CNTs changed by at most a few percent upon twisting, several fold variations were observed for BCNNTs (Figure 3). The electromechanical response of BCNNTs is therefore up to 2 orders of magnitude larger than that of multiwall CNTs. Given the geometry of the torsional devices, one might argue that this phenomenon is due to a poor adhesion between the nanotube and the metallic contacts, leading to changes in the contact area and thus to the overall conductance as a force is applied on the pedal. However, no breakage of the adhesion between the pedal and the BCNNTs was observed, which should have happened in case of loose contacts, since the pedal and the contacts are made by the same lithography process. Moreover, bad contact/nanotube adhesion would have caused an apparent decrease of the BCNNT torsional stress. If this were the case for the observed
torsional piezoresistance, we should have seen a correlation between electromechanical response and torsional strength (i.e., the BCNNTs that react electromechanically should have been softer). The data from Table S1 show that this is not the case, thus dismissing the loose contact concern. It therefore seems that the observed conductance variations are caused by an intrinsic structural change occurring in the BCNNT upon torsion.

Previous work has shown that, owing to the high bandgap of h-BN, current transport in BCN composites is mostly achieved via percolation paths and tunneling through the carbon network.39 Since BCNNT are synthesized via post-synthesis chemical doping of BNNTs with small aromatic molecules, carbon atoms are not homogeneously distributed, but rather form patches of carbon-rich areas along the nanotube.40 It is likely that, by twisting the nanotube, one brings the C-rich areas closer or further away one from the other, leading to an increase or decrease of the electrical conductance. Moreover, TEM imaging has shown that most BCNNTs are faceted, just like BNNTs. Recent work published by our group suggested that BNNT with diameters larger than 25–30 nm undergo unfaceting when twisted at large torsion angles,41 i.e., revert to a circular cross-section, or at least undergo a reduction of the degree of cross-section polygonalization. Unfaceting should be more energetically favored for the hybrid BCNNTs, because there are less possibilities of B–N eclipsed AA stacking in BCNNTs compared to BNNTs.40 This could account for the reversible softening observed at large torsion angle (Figure 3d and f). We suggest that the toggling between faceted and circular BCNNT geometries occurring during torsion could also contribute to the torsional piezoresistance observed in BCNNTs. Indeed, conductance variations have been mostly observed at large torsion angles, in the range that we associated with unfaceting (see Figure 3). Furthermore, considering that facet edges are high-energy regions and thus more susceptible to be affected by C-doping, a change in the cross-section geometry might dramatically alter the percolation paths through the carbon network and thus the BCNNT electrical conductance. In the future, theoretical simulations, as well as in situ BCNNT torsion experiments inside a TEM,25,35 should be carried out in order to corroborate this hypothesis.

In summary, we have presented here the first experimental investigation of the mechanical and electromechanical properties of composite BCN nanotubes. BCNNTs exhibit a significant torsional coupling, which makes them on average two to three times and up to five times torsionally stiffer and stronger than CNTs. Moreover, BCNNTs were shown to exhibit dramatic conductance variations when twisted: the torsional piezoresistance of BCNNTs is up to 2 orders of magnitude larger than for multiwall CNTs or BNNTs and up to 1 order of magnitude higher than for single-wall CNTs. These properties make BCNNTs highly sensitive torsional electromechanical transducers and thus promising building blocks for NEMS, which should foster more theoretical and experimental research on their syntheses and mechanical and electromechanical properties.

**ASSOCIATED CONTENT**

5 Supporting Information
(1) Close-up TEM bright-field image of a BCN nanotube. (2) Effective shear modulus calculated according to the solid rod assumption. (3) BCNNT electromechanical response for several consecutive actuations, for two representative devices.

(4) Torsional spring constant, effective shear moduli, torsional strength, and torsional electromechanical response for all nanotubes investigated. (5) Experimental methods. This material is available free of charge via the Internet at http://pubs.acs.org.

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**Notes**
The authors declare no competing financial interest.

**ACKNOWLEDGMENTS**

This work was supported by the Israel Science Foundation, the Israeli Ministry of Defense, the Minerva Foundation, the Kimmel Center for Nanoscale Science and Moskowitz Center for Nano and Bio-Nano Imaging at the Weizmann Institute, and the Dianogly, Alhadeff, and Perlman foundations, as well as the Center for Nanoscience and Nanotechnology at Tel Aviv University, the Humboldt Foundation and the Lise Meitner-Minerva Center for Computational Quantum Chemistry. D. Golberg acknowledges NIMS Grant No. BE063. W. Wang acknowledges support from NSF of China (21322304). We thank O. Hod and R. Tenne for helpful discussion.

**ABBREVIATIONS**
BN, boron nitride; BCN, boron carbonitride; NT, nanotube; NEMS, nanoelectromechanical systems; HRTEM, high-resolution transmission electron microscopy; EELS, electron-energy loss spectroscopy; AFM, atomic force microscopy

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**ABBREVIATIONS**
BN, boron nitride; BCN, boron carbonitride; NT, nanotube; NEMS, nanoelectromechanical systems; HRTEM, high-resolution transmission electron microscopy; EELS, electron-energy loss spectroscopy; AFM, atomic force microscopy

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Supporting Information

BCN Nanotubes as Highly Sensitive Torsional Electromechanical Transducers

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Figure S1: Close-up TEM bright-field image of a BCN nanotube. The dark high-contrast areas observed in the BCNNT indicate the presence of facets. Scale bar: 10 nm.

Figure S2: Effective shear modulus calculated according to the solid rod assumption (see text), plotted as a function of BCNNT diameter (close-up of Figure 2c).
Figure S3: BCNNT electromechanical response for several consecutive actuations (following the rainbow order from red to blue), for two representative devices: (a) device F, and (b) device E. Both sets of data show the reproducibility of the BCNNT electrical response to torsion.
Table S1: BCNNT torsional mechanical and electromechanical characterization.

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Notes: $d$: BCNNT diameter; $L$: BCNNT suspended length (obtained both from AFM topography); $\kappa$: torsional spring constant; $G_h$ and $G_s$: effective shear moduli according to hollow cylinder and solid rod assumptions, respectively; $\tau_{BCNNT}$: torsional strength (calculated for the whole nanotube). EM: electromechanical. The experimental error (EE) for $d$ is the standard deviation of several measurements performed along the BCNNT length. The EE for $L$ is derived from the resolution of the AFM topography image. The EE for $\kappa$ is the damped least-squares fitting error obtained by fitting linear stiffness versus lever arm plots to equation (S1) (see Methods). The EE for $G_h$ is obtained by combining the EE for $d$, $L$ and $\kappa$. The EE for $\tau_{BCNNT}$ is obtained by combining the EE for the maximum torque and nanotube diameter. N. M.: not measured.
Experimental Methods

*Synthesis:* BNNTs were synthesized by chemical vapor deposition (CVD) as described in [1]. BCNNTs employed in this study were synthesized by post-synthesis substitutional C-doping of CVD-grown BNNTs, as described in [2].

*Materials and Nanofabrication:* The torsional BCNNT-based NEMS were produced by methods similar to those reported for previous torsional devices.\(^3\text{-}^6\) Alignment marks and large contact pads were created on thermally oxidized silicon wafers (Si \(<100\), oxide thickness: 1 \(\mu\)m) by photolithography, metal evaporation and lift-off. BCNNTs were dispersed in ethanol by brief sonication prior to deposition. The nanotubes were mapped and their diameter was measured by AFM imaging. Pedals and electrodes were laid down on the selected BCNNTs by electron beam lithography, electron beam evaporation of Cr (5nm) and Au (80 nm) and lift-off in acetone. The sample was then mounted into a chip-carrier and wire bonded. Finally, the SiO\(_2\) layer was etched in aqueous HF/NH\(_4\)F (1:6) for 7 minutes; then, without drying the sample, the etching solution was consecutively replaced by water, ethanol and pressurized CO\(_2\), from which they were critical-point dried.

*BCNNT torsional measurements:* AFM imaging, mechanical and electromechanical measurements were performed on a Veeco Multimode/Nanoscope V equipped with a closed-loop scanner, using Olympus silicon tips (nominal resonant frequency 70 kHz). All experiments were performed under dry nitrogen flow in order to reduce humidity and
thermal fluctuations. Samples were first imaged by tapping mode (TM) AFM. We then zoomed at the desired position and pressed on the pedal with the AFM tip, which twisted the nanotube. The torsional stiffness of BCNNTs was measured by pressing at a series of points along the long axis of the pedal, and recording for each point the linear stiffness $K = k_c z_c/(z_p - z_c)$, where $k_c$ is the spring constant of the AFM cantilever, $z_p$ is the z-piezo extension, and $z_c$ is the deflection of the cantilever. The linear fitness was then plotted against the distance along the pedal (Figure 2b) and fitted with equation (S1):

$$K = \left[ \frac{(x-a)^2}{2\kappa} + K_b^{-1} \right]^{-1} \quad \text{(S1)}$$

where $x$ is the distance measured along the pedal (see red line in Figure 2a), the torsional spring constant ($\kappa$), the bending spring constant ($K_b$), and the lever arm ($a$) being left as floating parameters. More experimental details regarding nanotube torsional mechanics can be found in refs [3, 6-7].

Electrical signals were delivered by a Keithley 6517A electrometer (DC bias) and a Perkin Elmer DSP7280 lock-in amplifier (AC bias) connected via a summing amplifier. The lock-in amplifier (LIA) was interfaced to the AFM and a computer via appropriate data acquisition cards.

*Microscopic and EELS characterization:* HRTEM and EELS were performed at 300 kV with a FEI Tecnai F30-UT field emission TEM. EELS spectra and energy-filtered TEM were acquired using a Gatan Imaging Filter. EELS quantitative analysis was performed
by integrating the intensity (after background fitting and subtraction) of the edges corresponding to each element, using the Digital Micrograph software.

**Supplementary references**


