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Mechanical Resonances of Helically Coiled Carbon Nanowires

SUBJECT AREAS:

MECHANICAL
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NANOWIRES

STRUCTURAL PROPERTIES

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Despite their wide spread applications, the mechanical behavior of helically coiled structures has evaded an accurate understanding at any length scale (nano to macro) mainly due to their geometrical complexity. The advent of helically coiled micro/nanoscale structures in nano-robotics, nano-inductors, and impact protection coatings has necessitated the development of new methodologies for determining their shear and tensile properties. Accordingly, we developed a synergistic protocol which (i) integrates *analytical*, *numerical* (i.e., finite element using COMSOL®) and *experimental* (harmonic detection of resonance; HDR) methods to obtain an empirically validated closed form expression for the *shear modulus* and *resonance frequency* of a singly clamped helically coiled carbon nanowire (HCNW), and (ii) circumvents the need for solving 12th order differential equations. From the experimental standpoint, a visual detection of resonances (using *in situ* scanning electron microscopy) combined with HDR revealed intriguing non-planar resonance modes at much lower driving forces relative to those needed for linear carbon nanotube cantilevers. Interestingly, despite the presence of mechanical and geometrical nonlinearities in the HCNW resonance behavior the ratio of the first two transverse modes f_2/f_1 was found to be similar to the ratio predicted by the Euler-Bernoulli theorem for linear cantilevers.

Nature has inspired scientists through its multitude of nonlinear complex structures that exhibit tailored properties and diverse functionalities on multiple length scales. For example, biomolecules including DNA, bacterial flagella and various proteins adapt nonlinear geometries, particularly spring-like or helically coiled morphologies (e.g., double-helix, α -coils or helices) which facilitate specific functionalities in living organisms. Indeed, this ubiquitous nanoscale helically coiled morphology has been proposed for enabling elastic memory devices¹, flexible electronics², impact protection³, nanoinductors, and efficient electromagnetic shielding⁴. Albeit their enormous potential, helically coiled topologies suffer from poor characterization, especially mechanical properties, and thus remain severely under-utilized in the realization of robust electronic, mechanical and optical devices. While a few studies have focused on investigating the elastic properties using axial elongation⁵/compression⁶ and atomic force microscopy (or force modulation microscopy)^{7–9}, an in-depth study of the dynamic mechanical response of helically coiled structures is incomplete due to the difficulties involved in exciting purely longitudinal/transverse resonances. This difficulty stems from the distinct helical geometry which exhibits convoluted shear and tensile motions when the singly-clamped coil is transversely driven. Theoretically, the resulting motion is a set of coupled 12th order differential equations^{10,11} that are nearly impossible to analytically solve for the Eigen modes and their associated frequencies. In view of this mathematical complexity, helical coils are often treated as straight pillars^{8,12} without completely accounting for the shear/tensile component, which prevents the acquisition of accurate Eigen modes and frequencies.

Here, we employed the Harmonic Detection of Resonance (HDR) method^{13,14} to experimentally resonate isolated helically coiled nanowires (HCNWs) and deduced their shear modulus by concurrently validating experiments with analytical and numerical models. In particular, we developed an analytical model that elucidates the contribution of shear stress when a helical coil is subjected to a transverse moment. By collating the individual components of shear and bending stresses, we derived a closed form expression for the fundamental transverse resonance of HCNWs as a function of both the Young's (E) and the shear modulus (G). The values of G obtained in our analytical model provided only a coarse estimate due to several limitations imposed by our assumptions. Accordingly, we employed the numerical finite element COMSOL technique to find accurate G values from the coarse analytical estimates (Fig. 1). Furthermore, we observed intriguing resonance modes (e.g., circularly polarized mode) in HCNWs with a characteristic signature in the HDR signal, thus facilitating the electrical detection of nonlinear resonance modes. In general, we demonstrated that the HDR technique in tandem with our

Figure 1 | The protocol followed during the study. Three methods viz. experimental (using Harmonic Detection of Resonance), analytical and computational (COMSOL) are used in conjunction to create a model that predicts the resonance frequency (f) of the helically coiled nanowire (HCNW) and calculates the shear modulus (G) of its material.

analytical model and COMSOL simulations can serve as a standard protocol for probing the shear properties of coiled geometries circumventing the need for solving 12th order differential equations.

Methods

A forest of randomly oriented HCNWs was synthesized using a previously reported two-stage, liquid precursor based thermal chemical vapor deposition (CVD) method¹⁵. The as-prepared HCNWs were characterized using scanning (SEM; Hitachi SU-6600) and transmission (TEM; Hitachi 9500) electron microscopy (Fig. 2). For further discussion, we introduced the following labels for the physical parameters of a HCNW (Fig. 3) r - radius of the wire; D - diameter of HCNW; p - pitch of HCNW; n - number of turns; l - length of HCNW (note that $l = n \cdot p$); M - Moment experienced by a turn of HCNW upon bending with a radius of curvature ζ ; and α - pitch angle of HCNW.

For HDR experiments, an isolated HCNW mounted on an electrochemically etched tungsten tip (W-tip) was used as the cantilever (see supplementary information Fig. S1). The W-tip was brought into contact with a double-sided conducting carbon adhesive tape (PELCO TabsTM; Ted Pella) and then used to isolate a single HCNW from a forest. The HDR setup consists of another bare W-tip which serves as the counter electrode (CE) and a three-directional nano-positioning stage (PI Block

Nano Positioning System) that facilitates a fine control of the relative orientation and gap distance between the cantilevered HCNW and the CE. The HDR setup was used with either an SEM chamber (Hitachi SU-6600 or S-3400) to provide a stable vacuum (<1 Pa) which mitigated damping losses, or a dark-field optical microscope (Nikon epiphot; 50 $\times$ and 100 $\times$ objective). Importantly, the mechanical resonances observed under the SEM were simultaneously detected “electrically” using the HDR method. The HCNW was actuated via an AC peak signal- V_{ac} (to induce periodic motion) with a DC offset V_{dc} (to overcome work potentials). To avoid a zero crossing in the net excitation voltage ($V_{net} = V_{ac} + V_{dc}$), V_{dc} was maintained at values higher than V_{ac} . For all experiments discussed below, V_{dc} was held constant at 9 V while V_{ac} was varied within ± 9 V. A detailed discussion of the HDR method can be found elsewhere^{16–18}. Briefly, in the HDR method, a cantilever is actuated by either static or dynamic charge induction depending on several factors such as the presence of static charges, functional groups, and intrinsic electrical conductivity of the cantilevered structure. In the absence of V_{dc} , the dynamic (static) charge induction mechanism often resulted in a vibration at twice (same) the driving frequency for well-grounded electrically conducting cantilevers¹⁹. Accordingly, we observed the motion of a cantilevered HCNW ~ 1 Hz under the dark-field optical microscope to confirm the presence of dynamic charge induction mechanism and ensure that HCNW was properly grounded. However, we observed that HCNWs were always positively charged when exposed to an electron beam, possibly due to the ejection of electrons. Nonetheless, the HCNWs were found to be neutral when the electron beam was turned off, as observed from our dark-field experiments.

In previous studies, we found a strong correlation between the presence of structural defects and the mechanical properties of nano-cantilevers²⁰. In view of this correlation and to eliminate the effects of sample heterogeneity in this study, we consistently selected all HCNWs from the same synthesis batch (all HCNWs are identical in terms of chemical composition and defect density albeit differences in structural parameters; HCNW1- HCNW4 shown in Table 1). It is worth noting that the harmonics and resonance modes overlap in the traditional doubly clamped geometry, e.g., strings in a guitar; therefore, we used the singly clamped geometry to distinguish the resonance modes of HCNWs from their harmonics.

Results and discussion

Modeling. The transverse resonance frequency (f) of a singly clamped cantilever can be calculated by the well-known relationship, $f = 1/2\pi (k/m_{eff})^{1/2}$ where k is the spring constant and m_{eff} is the effective mass of the cantilever²¹. In contrast to a straight cantilever, k for the transverse bending mode of a HCNW is a function of both E and G of the material, which makes it difficult to compute the resonance frequencies. This motivated us to adapt the following protocol (see Fig. 1) to obtain an experimentally validated closed form expression for resonance frequency (f_{ANA}). We employed the HDR method to obtain the frequencies of first two modes (f_1 and f_2) of HCNWs and deduced their shear modulus by simultaneously validating experiments with analytical and numerical models.

Initially, we consider one turn under a moment (M) in the plane of the HCNW axis. To simplify the model, we approximated one HCNW turn as a planar ring connected with a rigid rod as shown by

Figure 2 | (a) Scanning electron microscope image of as-prepared HCNWs. The coiled structures are solid wires as evident in the transmission electron microscope (TEM) image shown in (b). The dotted region of (b) shows layered stacking at the surface of the coil in the high-resolution TEM image shown in (c). The selected area diffraction pattern (d) reconfirms the polycrystalline nature of HCNWs.

Figure 3 | (a) Geometrical parameters of an HCNW (b) One turn of the coil modeled as a planar ring connected with a rigid rod, compensating for the pitch. (c) Left panel- one turn of the coil with no moment. Right Panel- Bending of the coil under Moment M . An imaginary cylinder (dashed lines) is a guide to understand the bending mechanism. (d) Vector diagram of the displacements upon bending as seen in (c).

Wahl²² (Fig. 3b). The bending of the HCNW due to the application of electrical driving force in our experiments naturally creates an internal shear stress. The resulting deflection angle φ (Fig. 3c) of a turn (under M) of the HCNW is a sum of bending (φ_b) and twisting (φ_t) angles:

$$\begin{aligned}\varphi &= \varphi_b + \varphi_t, \\ &= \frac{\pi MD}{2EI \cos^2 \alpha} + \frac{\pi MD \cos^2 \alpha}{2GJ},\end{aligned}\quad (1)$$

where $I = \pi r^4/4$ and $J = \pi r^4/2$ are the area and polar moments of inertia of the HCNW (See supporting information and Fig. S2). Note that an extra factor of $\cos^2 \alpha$ was included in equation 1 to compensate for larger pitch angles (see supporting information). For a complete turn of a HCNW, k is defined as F/z where z is the resulting displacement due to the force F acting on a single turn. As shown Fig. 3d, z may be expressed as a function of φ and p under the small angle approximation (see supporting information) as:

$$z = p \left(\frac{1 - \cos \varphi}{\varphi} \right) \approx p \left(\frac{\varphi}{2} \right)$$

Since $F = M/p = kz$ in a HCNW,

$$\frac{1}{k} = \frac{zp}{M} = \frac{\varphi}{M} \left(\frac{p^2}{2} \right). \quad (2)$$

Hence for one complete turn, equations (1) and (2) yield

$$\frac{1}{k} = \frac{p^2 D}{Er^4 \cos^2 \alpha} + \frac{p^2 D \cos^2 \alpha}{2Gr^4}. \quad (3)$$

Equation 3 is obtained by combining the components due to bending $\frac{1}{k_b} = \frac{p^2 D}{Er^4 \cos^2 \alpha}$ and twisting $\frac{1}{k_t} = \frac{p^2 D \cos^2 \alpha}{2Gr^4}$ of the HCNW.

For a HCNW of n turns, we can obtain the spring constant from equations (2) and (3) by incorporating an n^3 term, analogous to a straight cantilever (Fig. S3),

$$\frac{1}{k_n} = \frac{n^3 p^2 D}{Er^4 \cos^2 \alpha} + \frac{n^3 p^2 D \cos^2 \alpha}{2Gr^4}. \quad (4)$$

Hence, we can then obtain f_{ANA} as

$$f_{ANA} = \frac{1}{2\pi} \frac{r^2}{p} \left(n^3 D m_{eff} \left[\frac{\cos^2 \alpha}{2G} + \frac{1}{E \cos^2 \alpha} \right] \right)^{-1/2}, \quad (5)$$

which was validated by comparing to iteratively calculated resonance frequencies (cf. Fig. 1).

Next, using a finite element based COMSOL technique, we simulated the resonance frequency f_{COM} for various HCNWs with different geometric parameters (n , r , D , p). Based on our COMSOL results, f_{ANA} matched within 12% of f_{COM} , which was scaled by a dimensionless factor $s = 0.88$ to account for the simplification of a HCNW to a planar ring attached to a rigid rod

$$f_{ANA} = (0.88) \frac{1}{2\pi} \frac{r^2}{p} \left(n^3 D m_{eff} \left[\frac{\cos^2 \alpha}{2G} + \frac{1}{E \cos^2 \alpha} \right] \right)^{-1/2}. \quad (6)$$

The closed-form expression (equation 6) is a solution for HCNW resonance frequency, which includes the effects of both E and G . As described in Fig. 1, experimentally obtained resonance parameters of

Table 1 | Observed resonant frequency f_1 ; dimensions r , D , p , n , α ; and transverse shear modulus G_{COM} (and corresponding Poisson Ratios) calculated using $E = 200$ and $E = 80$ for the HCNWs

HCNW No.	f_1 (kHz)	r (nm)	D (nm)	P (nm)	n	α (degree)	G_{COM} (GPa)	
							$E = 200$ GPa (ν)	$E = 80$ GPa (ν)
1	30	63.5	290	876	48.4	43.9	8.6 (10.63)	10.0 (3)
2	82	112	750	1116	24	Variable	5.8 (16.24)	6.2 (5.45)
3	56	103	330	1014	40	44.4	7.9 (11.66)	8.3 (3.82)
4	70	73	256	1187	29.5	55.9	6.2 (15.13)	10.3 (2.88)
						Avg. =	7.1	8.7
						Std. Dev. =	1.3	1.9

HCNWs can be used in conjunction with equation 6 to obtain G . It is noteworthy that equation 6 is not limited to HCNWs and may be used to obtain the G (or f) of any helically coiled geometry whose E and f (or G) are known.

Measuring resonance parameters. The flexural spring constant of HCNWs is expected to be lower than its axial spring constant due to their moderately high aspect ratio (ratio of length to diameter of the HCNW $\sim 10^2$). Hence, the probability of actuating transverse resonance modes (perpendicular to HCNW axis) is much higher relative to axial modes (parallel to HCNW axis). Figs. 4a–4c and 4d–4e show the transverse modes for two different HCNWs (parameters listed in Table 1), HCNW1 (r- 63.5 nm; D-290 nm; p- 876 nm; l- 42.4 μ m) and HCNW2 (r- 112 nm; D-750 nm; p- 1116.3 nm; l- 26.8 μ m). A direct *visual* examination under the SEM revealed that the HCNW1 (HCNW2) resonated at frequency $f_1 \sim 30$ kHz (82.35 kHz) at its first mode (Fig. 4b) and at $f_2 \sim 190$ kHz (547 kHz) at its second mode (Fig. 4c). The corresponding HDR amplitude signal for the first mode of HCNW2 (excited with 5 V_{ac} at ~ 1 Pa; Fig. 4f) showed a single transverse resonance peak with a quality factor (defined as the resonance frequency/full width at half maximum of the power spectrum) of ~ 150 .

To examine the coupling between bending and shearing moduli of ‘geometrically’ nonlinear HCNW cantilevers, we actuated HCNWs at a higher driving force by either increasing the AC driving voltage ($\sim 8 V_{ac}$) or reducing the gap distance between HCNW and the CE. A higher driving force was observed to result in ‘mechanical’ nonlinearities such as the characteristic Duffing-like jumps in cantilever motion (see Fig. 5). Interestingly, as shown in Fig. 5, we found that: i) the HCNWs resonated in a non-planar circular mode when the

driving voltage was increased $>8 V_{ac}$ (or the gap distance is reduced $<5 \mu$ m), and ii) simultaneous with the observation of the circular mode, the HDR amplitude signal exhibited a bifurcation of an otherwise single transverse resonance peak (Fig. 5b). Through visual comparison, we confirmed that the lower (higher) frequency peak corresponded only to the planar transverse (non-planar circular) resonance. As we varied the driving frequencies (from 85 to 95 kHz), we observed the onset of the non-planar modes between 90–94 kHz, and a clear rotation of the plane of resonance (circular mode as seen in Fig. 5a). As shown in Fig. S4, the onset of non-planar motion was also evident for an independent HCNW3 (r- 103 nm; D- 330 nm; p- 1014 nm; l- 40.6 μ m, see Table 1). During the reverse frequency sweep, we observed a typical hysteresis (as indicated by the hatched area in Fig. 5b) associated with mechanical nonlinearities (i.e., non-planar circular mode). The hysteresis was more prominent for the higher frequency peak or circular mode (gray) relative to the lower frequency peak or transverse mode (green). It is worth noting that the bifurcation of the resonance signal disappeared upon restoring the initial actuation conditions (i.e., $V_{ac} \sim 5$ V) indicating that the difference between the driving forces necessary for actuating planar vs. non-planar mechanical modes is significantly low for HCNWs relative to straight cantilevers (e.g., multi-wall carbon nanotube or MWCNTs). Indeed, the high actuation threshold for non-planar modes in straight cantilevers has been a major bottleneck in understanding mechanical nonlinearities (e.g., Duffing-like circular modes). Although non-planar modes were reported previously in some straight nanocantilevers²³ (SiC nanowires, MWCNTs and Si nanocantilevers²⁴), the necessary driving force was sufficiently large to destroy the samples impeding detailed studies of their nonlinear mechanical behavior. The helically coiled geometry plays a pivotal

Figure 4 | SEM images of a singly clamped HCNWs. (a) HCNW1 (radius 63.5 nm; coil diameter 290 nm; pitch 876 nm) off resonance, (b) first and (c) second transverse modes of HCNW1 at driving frequency $f_1 \sim 30$ kHz and $f_2 \sim 190$ kHz. The dotted lines serve as a guide to the eye. (d) HCNW2 (radius 112 nm; coil diameter 750 nm; pitch 1116.3 nm) off resonance (e) first transverse mode of HCNW2 ($f_1 \sim 82.5$ kHz). (f) Harmonic Detection of Resonance (HDR) signal (amplitude in blue trace and phase in black trace) for transverse mode for HCNW2 with driving voltages $V_{ac} = 5$ V, $V_{dc} = 9$ V.

Figure 5 | (a) SEM image of HCNW2 resonating in circular mode at a driving signal ~ 93 kHz (b) HDR signal shows a bifurcation in the resonance signal. The peak at ~ 90 kHz corresponds to an in-plane transverse resonance that occurs before the onset of the circular mode (peak at 93.5 kHz).

role in enabling the actuation of otherwise inaccessible non-planar modes at significantly lower AC voltages (~ 8 V) due to two reasons: i) a low transverse k of HCNWs allows them to bend easily relative to their straight counterparts (e.g., MWCNT); and ii) the inherent asymmetry of HCNWs separates the otherwise degenerate x -, y -polarizations in mechanical resonance. As described below, our numerical analysis using COMSOL also confirmed that the helically coiled geometry leads to non-degenerate x -, y -planar modes that can be actuated simultaneously to produce the observed non-planar modes (see Fig. S5). Although the nature of the electrical force in HDR experiments is such that it favors the excitation of only one of the two (x or y) modes, the coupling between bending and shear moduli in a HCNW provides a channel for the exchange of energy between x - and y -modes resulting in non-planar resonance. Therefore, the resonance analysis of other materials (e.g., Si and ZnO) in their helically coiled geometry will allow for elucidating the nonlinear mechanical properties without breaking or destroying the cantilever. We also attempted to actuate a purely axial mode parallel to the axis of HCNW by placing a blunt W-tip in tip-to-tip geometry ($3 V_{ac}$, $9-10 V_{dc}$) with HCNW. However, the parametric nature of the driving force always resulted in an asymmetric combination mode (distinct from the circular mode) consisting of transverse resonance and axial stretching (Fig. 6).

Returning to Fig. 1, the analysis of transverse resonance modes in COMSOL revealed that the ratio of the second and first modes (f_2/f_1) is 6.27- a ratio predicted by Euler-Bernoulli (EB) theory exclusively for straight cantilevers. Our HDR experiments showed that $f_2/f_1 \sim 6.75 \pm 0.38$ for 3 different HCNWs, deviating only slightly from the straight cantilever case, in agreement with COMSOL. The experimental and COMSOL results prove that although EB theory is insufficient to predict the motion of HCNWs accurately, the ratio f_2/f_1 is a true transcendental constant independent of the geometry of the resonating cantilever. Such an observation allows us to predict the second mode of transverse resonance by the following relation

$$f_2 = \frac{6.27}{2\pi} \sqrt{\frac{k}{m_{eff}}} \quad (7)$$

The protocol described in Fig. 1 was applied to HCNWs to obtain their shear modulus (G). The HCNWs isolated for HDR studies were carefully characterized to accurately obtain r , R , p and n . We calculated the HCNW resonance frequency (f_{COM}) using Eigen-frequency study under the fixed-free boundary condition and a fine mesh construction for a numerically stable solution in COMSOL. We used equation 6, experimental HDR results, and an estimated $E = 120$ GPa (similar to MWCNTs) to obtain an initial estimate for

G_{ANA} for HCNWs. Subsequently, we implemented a series of COMSOL simulations on HCNWs 1–4 (see Table 1) by varying $E \sim 80-200$ GPa (in steps of ~ 20 GPa) and $G \sim 5-10$ GPa (in steps of ~ 1 GPa) to obtain a f_{COM} that matched f_1 . Since there could be more than one set of E & G values that could result in a $f_{com} \sim f_1$, we used the experimental and COMSOL data from HCNWs 1–4 to identify correct E and G values. As shown in Table 1, HCNW 1–4 resulted in a G_{COM} value ranging from 5.8 to 10.3 GPa with an average value of $\sim 8 \pm 2$ GPa.

The Poisson ratio ($\nu = E/2G - 1$) of HCNWs was found to be as high as 2.88–16.24 based on the values of G_{COM} shown in Table 1 suggesting that the HCNWs may be anisotropic *i.e.*, the elastic properties are different in the direction parallel and perpendicular to the axis, or that HCNWs are inhomogeneous.

Conclusion. In summary, we mechanically resonated HCNWs and studied their resonance modes using the HDR technique. Clearly the HDR electrical signals were found to inherently carry the signature of the excited resonance mode (circular vs planar) as seen under an SEM. We developed a model (based on experimental, analytical, and numerical methods) capable of predicting the first and the second transverse resonance modes applicable to any helically coiled

Figure 6 | SEM images depicting a mixed resonance mode in HCNW1 detected at ~ 29.5 kHz. This asymmetric mode results from the mixing of axial and transverse motions of the nanocoil. The inset depicts the same when the HCNW is off resonance. The dotted line is a guide to the eye.

cantilevers irrespective of their dimensions. Our experiments showed that HCNWs had a shear modulus $\sim 8 \pm 2$ GPa. The corresponding Poisson ratios suggested a possible anisotropy in the HCNWs.

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Author contributions

D.S., B.P., and D.D. have done the experiments. D.S., H.B. and M.J.S. developed the analytical model. D.S. validated experimental results using numerical methods. D.S., H.B., S.M.S., M.J.S., R.P. and A.M.R. analyzed the data. D.S., R.P. and A.M.R. drafted the results.

Additional information

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