Mechanical properties of carbon nanotubes: a fiber digest for beginners

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Abstract

A condensed review of mechanical properties of carbon nanotubes is given. Theory as well as experiments is examined with a view to extracting the fundamental elements that should allow the reader to build his own perspective of the subject. © 2002 Published by Elsevier Science Ltd.

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1. Introduction

Carbon nanotubes (CNT), like whiskers, are acicular single crystals of high aspect ratio which contain only a few defects. It is chiefly this low density of defects that confers excellent mechanical properties to CNT. In addition, the intrinsic strength of the carbon–carbon sp^2 bond is expected to give CNT not less than the highest strength and modulus among all existing whiskers. This has been confirmed recently by a series of acrobatic experiments that are going to be discussed. Despite their early entry in the Hall of Fame of material science, it remains that 10 years after their discovery nanotubes are still more in the laboratories than on the stall. Does it reflect the natural time lag between discoveries and their exploitation, or may this stem from more fundamental problems? This paper aims to describe recent developments around mechanical properties of CNTs, both from a fundamental point of view and in the direction of applications, specifically those interested in reinforcements for composite materials.

In the course of a discussion about mechanical prop-

erties and for the clarity of analysis, it appears worthwhile to separate elastic properties from breaking strength. Indeed, establishing the elastic parameters of a solid from its microscopic elements (atomic composition and bonds) is nowadays a far less difficult task than predicting the way it may break. The fracture of materials is a complex phenomenon that requires a multiscale description involving microscopic, mesoscopic and macroscopic modeling. Most studies are based on a macroscopic classical continuum picture that provides an appropriate modeling except at the region of failure where a complete atomistic description (involving bond breaking in real chemical species) is needed. Classical molecular dynamics simulations using empirical potentials can be used to bridge the mesoscopic and microscopic modeling which can help to elucidate several relevant processes at the atomic level. To bridge micro-, meso- and macroscales it is desirable to find a hierarchy of models, both for ease of computation and for conceptual understanding. When models bridging different scales are worked out we will be able to analyze and optimize materials properties at different levels of approximation, eventually leading to the theoretical synthesis of novel materials. Nanotubes offer the possibility of addressing the validity of different macroscopic and microscopic models of fracture and mechanical response. A complete description of the mechanical properties and applications

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of carbon and composite nanotubes can be found in Ref. [1].

2. Theoretical investigations

2.1. Some concepts in mechanical properties: stiffness, strength and fracture toughness

The mechanical properties of a solid must ultimately depend on the strength of its interatomic bonds. Let us imagine an experiment, where a perfect rod of a given material is stressed axially under the force F. The rod length, l_0 at rest, will vary by dl. The macroscopic stiffness, F/dl, is directly related to the stiffness of the atomic bonds. In a simple harmonic model, the Young modulus, Y, can be written k/r_0, where k is the spring constant of the bond and r_0 is the interatomic distance. This distance does not vary much for different bonds but does k (between 500 and 1000 N/m for carbon–carbon bond and between 15 and 100 N/m for metals and ionic solids) [2]. Obviously, a low mass density is also often desirable for applications. Most polymers are made of carbon and have low density, but often have weak bonds between their chains and therefore are not stiff when non-oriented. This can be improved by forming composites of polymers with other stiff materials (like nanotubes).

It is a good job to synthesize a stiff material. It is also worth controlling that the material does not break at too small strain as can happen with ceramics. The theoretical strength of a material is described by the famous Orowan–Polanyi equation [3]: $(Yg/x_0)^{1/2} \approx Y/10$, where g is the free surface energy and x_0 the equilibrium spacing between the planes to be separated. This value stems from an estimation of the energy needed to cleave a rod leaving two fresh surfaces. However, most single-phase solids have much lower strength values, around $Y/10^4$. This lowering stems from defects like dislocations, stacking-fault, grain boundaries, voids, cracks etc.

In fact, 'The worst sin in an engineering material is not lack of strength or lack of stiffness, desirable as these properties are, but lack of toughness, that is to say, lack of resistance to the propagation of cracks' (Gordon, 1976) [4]. Following Argon in chapter 1.24 of the Comprehensive Composite Materials publication, we recall that in all engineering structural solids, the tensile strength and fracture toughness are two complementary behavior forms of interest [5]. Tensile strength of materials with some inelastic behavior and fracture toughness are inversely related and increases in toughness are generally achievable at the expense of tensile strength. Roughly speaking crack propagation allows stress to relax in the material under strain; thus, blocking cracks favors an earlier catastrophic rupture.

2.2. Computational investigations

2.2.1. Young modulus

The strong similarity between chemistry of carbon nanotubes and graphite allows theoretical analysis to be done based on empirical methodologies imported from studies on graphite. They range from the direct zone-folding of the results for graphite to the quantum-mechanical studies based on tight binding Hamiltonians fitted to graphite properties. Zone folding and force-constants neglect curvature altogether. Model potentials can only account for the different distances among the atoms. However, to compute properties for 'large' systems (say more than 500 atoms) or 'long' time-scales one has to resort to this faster (but less accurate) scheme. At larger scales, quasi-continuum models with a combination of empirical potentials and elasticity theory may be used [6]. On the other hand, tight binding captures part of the chemical strain through the geometry-dependence of its electronic matrix elements, even though their absolute values depend on the electronic structure of graphite. It is then important to be able to compute the different properties for any tube radius using a tool that does not depend on a fit to graphitic properties so as to study the narrow-tube properties with the same degree of accuracy as the wide ones. First principles total energy calculations giving bonding, electronic structure and atomic arrangement are nowadays based on density-functional theory (DFT). This allows calculation of the ground-state and dynamical properties of a many electron system from a simple one-electron Schrödinger equation.

An extensive study of the elastic constants of carbon nanotubes and ropes of SWNT has been reported [7] using an empirical pair potential. (The force-constant model described by the sum of pairwise harmonic potentials between atoms up to fourth-neighbour fitted to reproduce the elastic constants and phonon frequencies of graphite.) The obtained elastic properties are essentially independent of helicity and tube radius, and comparable to those of graphene, with $Y = 0.97$ TPa for all tubes, smaller than TB and ab initio results. This model does not reflect the changing nature of the chemical bonding as the curvature is changed. In Ref. [8], by computing the stiffness of SWNTs for different chiralities, it has been demonstrated that the Young modulus (Y) shows a small dependence on the tube diameter and chirality for the experimental range of nanotube diameters (between 1.3 and 1.4 nm). It has been predicted that carbon nanotubes have the highest Young's modulus of all the different types of composite tubes considered (BN, BC_3, BC_2N, C_3N_4, CN, etc.).

The conventional definition of the Young modulus involves the second derivative of the energy with respect to the applied strain. This definition for an SWNT requires adopting a convention for the thickness of the carbon layer in order to define a volume for the object. Rather than

adopting an ad-hoc convention, the stiffness of an SWNT independent of any shell thickness can be defined as

$$Y_s = \frac{1}{S_0} \left(\frac{\partial^2 E}{\partial^2 \varepsilon} \right)_{\varepsilon = 0}$$

where S_0 is the surface area defined by the nanotube at zero strain, which is a well-defined quantity. Given that $V_0 = S_0 h$, h being the tube thickness 0.34 nm, one can recover the usual definition of the Young modulus. The computed value of 0.43 TPa nm, which corresponds to 1.26 TPa modulus taking $h = 0.34$ nm in relation to the interlayer distance in graphite, is in excellent agreement with one of the experimental valuea for SWNTs of 1.25 TPa [9]. It is also in rather good agreement with the value of 1.28 TPa reported for MWNTs in Ref. [10].

Ab initio calculations [11,12] have reported a slight dependence of Y on the tube diameter. As the diameter becomes larger, C approaches a plateau value that corresponds to the value calculated for the flat graphene-like sheet of each nanotube composition. Interestingly, the approach to the limit value is from below, as can be expected, since bending a flat graphene sheet weakens the bonds by reducing the degree of sp^2 hybridization in the bond. Given that it is the strength of the chemical bonds that determines the actual value of the Young's modulus, it is natural that small-diameter (high curvature) tubes have smaller Young's moduli, and in the limit of large diameters, the mechanical properties essentially correspond to those of the flat graphene sheet. In contrast, the results in Ref. [7] are largely insensitive to the tube diameter. Furthermore, the Young modulus approaches, from below, the graphitic limit for diameters of the order of 1.2 nm. Also, TB calculations of the stiffness of SWNTs show that the Young modulus shows a small dependence on the tube diameter and chirality for the experimental range of nanotube diameters (between 1.3 and 1.4 nm) [8], in agreement with the first principles calculations reported above. It is predicted that carbon nanotubes have the highest Young's modulus of all the different types of composite tubes considered (BN, BC_3, BC_2N, C_3N_4, CN) [13].

Generally, the computed ab initio Young modulus for both C and BN nanotubes agrees well with the values obtained by the TB calculations and with the trends given by the empirical Tersoff–Brenner potential. These results support the use of the simpler models to describe the mechanical properties of nanotubes for more complex situations.

2.2.2. Poisson ratio

The Poisson ratio ν is given by the variation of the radius of the SWNT resulting from longitudinal deformations along the tube axis. In all cases the computed Poisson ratio is positive: an elongation of the tube reduces its diameter. The ab initio values are $\nu = 0.14$ (from 0.12 to 0.16) for the armchair (n,n) tubes, and a little larger for other chiralities: 0.19 for (10,0) and 0.18 for (8,4). The uncertainty of the obtained values is of the order of 10%. These results reveal a slight decrease of the Poisson ratio with the tube radius, and a stronger dependence with chirality. This result is close to the value of $\nu = 0.19$ obtained by using Tersoff–Brenner potentials [14], but considerably smaller than the value $\nu = 0.28$ given by a force constant model [7] and $\nu = 0.26$ from a tight-binding calculation [8]. The corresponding magnitude along the basal plane in graphite is $\nu = 0.17$ ($= C_{12}/C_{11}$) [15]. In summary, the ab initio calculations indicate that the Poisson ratio retains graphitic value except for a possible slight reduction for small radii. It shows a chirality dependence: (n,n) tubes display smaller values than (10,0) and (8,4).

2.2.3. Deformation of CNTs under stress

Recent molecular dynamics calculations based on the Tersoff potential predict that a (10,10) nanotube can support an axial compression strain of 0.05 before buckling [16]. In a recent experiment large compressive strains were applied to carbon nanotubes dispersed in a polymeric film [17]. The obtained buckling of thick tubes is in agreement with the simulations of Ref. [16] whereas discrepancies are found for the plastic collapse or fracture of thin tubes. The compressive strain is estimated to be larger than 5% and critical stress for inward collapse or fracture is expected to be between 100 and 150 GPa for thin tubes.

In Ref. [18] a new mechanism for the collapse and plasticity of compressed thin nanotubes was reported. The relaxation of the strain energy in the collapsed section of the tube causes immediate graphitic to diamond-like bonding reconstruction at the location of the collapse. This mechanism, as well as the estimated critical stress of 153 GPa, is in qualitative agreement with the experimental results [17]. The main outcome of those studies is that both thin and thick-walled carbon nanotubes exhibit compressive strengths one order of magnitude higher than any other known fiber. Also, large scale molecular dynamics simulations within a TB-model for SWNTs under large applied strain (both elongation and compression) shows that the 0 K stress is very sensitive to helicity, the zig-zag and armchair tubes being the stiffest [19].

In all simulations, carbon nanotubes, when subjected to large deformations, switch into different shapes that come with an abrupt release of stress-energy [16]. The bending is fully reversible up to very large bending angles despite the occurrence of kinks and highly strained tubule regions. This flexibility property stems from the ability of the sp^2 network C–C bonds to reversibly change hybridization when deformed out of plane. The higher the curvature, the stronger the sp^3 character of C–C bonds in the deformed

region. The mechanism of strain release in carbon nanotubes under uniaxial tension has also been investigated in an effort to address the question of the ultimate tensile strength of these nanostructures [16–20]. Classical molecular dynamics simulations have been carried out for tubes of various geometries with diameters up to 13 nm [16–20]. Such simulations, although limited by the physical assumptions used in deriving the interatomic potential, are still invaluable tools in investigating very large systems for the time scales that are characteristic of fracture and plasticity phenomena. Beyond a critical value of the tension, an armchair nanotube under axial tension releases its excess strain via spontaneous formation of a Stone–Wales defect (5-7-7-5) through the rotation of a C–C bond. This produces two pentagons and two heptagons coupled in pairs (5-7-7-5). Overall, after nucleation of a first 5-7-7-5 defect in the hexagonal network, either brittle cleavage or plastic flow is possible depending on tube symmetry, applied tension and temperature. Under high strain and low temperature conditions, all tubes are brittle. If, on the contrary, external conditions favor plastic flow, such as low strain and high temperature, tubes of diameter less than ~1.1 nm show a completely ductile behavior, while larger tubes are moderately or completely brittle depending on their symmetry.

3. The answer of experiments

There is a growing body of experimental evidence indicating that carbon nanotubes (both MWNT and SWNT) have extraordinary mechanical properties, this in spite of the technical difficulties involved in the manipulation of these nano-scale structures. A number of experimental measurements of the Young's modulus of nanotubes were soon reported. The first such study was that of Treacy et al. [9], who correlated the amplitude of the thermal vibrations of the free ends of anchored nanotubes as a function of temperature with the Young's modulus. Regarding a MWNT as a hollow cylinder with a given wall thickness, one can obtain a relation between the amplitude of the tip oscillations (in the limit of small deflections) and the Young's modulus. The main drawbacks of this method are the poor precision and the impossibility to strain the nanotubes at will. The atomic force microscope has offered a new opportunity to manipulate nanotubes [10,21]. With feedback control interface, tying a knot in the nanotube has almost become affordable [21]. It rapidly appears, however, that a single tip is not the top. A direct tensile rather than sideways pull of a multiwall tube or a rope has clear advantage due to simpler load distribution and available force range. An important step in this direction has been recently reported. In this work tensile-load experiments are performed for MWNTs [22] and SWNT ropes [23] reporting tensile strengths in the range of 11 to 63 GPa with no apparent dependence on

the outer shell diameter. This is not far from the theoretical value of about 100 GPa.

It could seem now that experiments aimed at measuring the CNTs' Young modulus have been needless, since it is clear that this figure depends mainly on the intrawall C–C bond strength, which is known from graphite. In fact, the interwall cohesion or shear strength in an MWNT or in an SWNT bundle may have a strong influence on the actual stiffness. Indeed, imagine the extreme case of an absence of cohesion between adjacent tubes. This would imply no transfer load to the inner part of the structure. Each tube acts independently which results in a dramatic loss of stiffness. Therefore, it is important to perform measurements on various kinds of CNTs. The results of the various experiments have, in fact, been contrasted. When MWNTs are submitted to a steady stress of low intensity, the cohesion is probably high enough to ensure a good load transfer [24]. Increasing stress should result in a lowering of Y if decohesion occurs between inner and outer shells. This is observed only for a few samples in Yu's experiments. On the other side, as the stress increases the section of the outer shell decreases, which may improve the charge transfer to inner shells. Thus, one may discuss the validity of Yu's assumption that only outer shells or tubes participate in the loading. This is probably more complex, as suggested by the shape of the stress–strain curves [22,23]. When highly excited at their resonant frequency by a transducer as did Poncharal et al., decohesion of the walls may result in a lowering of Y [25]. In case of SWNT bundles, the low shear modulus is responsible for a decrease of the apparent bending modulus as the ratio L/R decreases, L and R being, respectively, the bundle's length and its diameter [26].

Radial compressibility of CNTs has also been investigated with an AFM [27]. Two regimes are encountered when increasing the applied normal force. At low pressure, the modulus is similar to that of graphite along the c-axis (about 10 GPa), while at higher pressure repulsion forces from the more deformed sides increase the elastic modulus.

Collapse of nanotubes into nanoribbons has been observed [28], that can be explained theoretically by the competition between the van der Waals attraction and elastic energy. The basic physics can be understood by noticing that the elastic energy per unit length of a tubule is proportional to $1/R$ (R, radius of the tubule). After collapsing of the tubule, the energy per unit length is composed of a higher curvature contribution from the edges, which is independent of the initial tubule radius, and a negative van der Waals contribution from the surface ($\sim R$). The second term wins above a critical radius R_c that depends on the number of shells of the nanotube [28].

Taking advantage of the high compression modulus, Chesnokov et al. demonstrated mechanical energy storage with 1.36 nm diameter SWNTs [29]. In this kind of experiment, uniaxial pressure cycles are applied to an

SWNTs mat, and the volume reduction and expansion are measured. The volume reduction was found reversible up to 29 kbar, with an associated stored energy of 0.18 eV/C atom. In that case, elastic energy dominated and no irreversible flattening was observed.

4. Composites reinforced with CNTs

As well as polymers, non-oriented mats of SWNTs suffer a lack of stiffness. There is indeed a large similarity between mechanical properties of a polymer film and an SWNT mat. Both exhibit viscoelasticity that can be evaluated using a nanoindentor [30]. Plastic imprint in a nanotube mat is easily done, despite its overall resistance to pressurization. It is therefore important to be able to align nanotubes in order to make stiff macroscopic ropes or continuous composites. We have learned from the previous sections that a continuous rope of infinitely long CNTs would exhibit unrivalled mechanical properties. The achievement of such ropes would launch CNTs into a juicy market [31,32].

Other important studies have focused on composites reinforced with nanotubes and it is worth insisting on the most important points. The first is to recognize that a lot has been done with micron-sized carbon fibers and whiskers. It would take a very long time to replace carbon fibers by CNTs, if ever possible. Better search for a market segment where CNTs offer a new opportunity. The great novelty with CNTs is to achieve high stiffness combined with high strength. One has to solve a number of non-trivial problems before benefitting from this double quality. Alignment-ability apart, maybe the most critical point concerns the interfacial load transfer between nanotubes and the matrix. The seamless structure of nanotubes associated to a low friction coefficient is a priori not an advantage for a good 'adhesion' with the matrix [33]. Some studies are in progress aimed at functionalizing CNTs with reactive molecular groups that should improve the interfacial shear strength.

Various studies have been dealing with CNT-based composites, with polymer [34], ceramic [35] and metallic matrices [36]. All of them show that CNT do act as reinforcing elements. However, without alignment, performances in terms of strength and stiffness are far away from what is currently reached with traditional carbon fibers.

The last point would like to warn the non-initiated reader against a wrong belief that strengthening is the only goal of a composite designer. Toughening is also a major concern in composite structural elements that involve a brittle matrix. Remember that to achieve high strength in a short fiber reinforced polymer, it is necessary to use fibers that are longer than the critical length and to create a strong interfacial bond between the fibers and the matrix. For high fracture toughness, on the other hand, a weaker interface promotes energy absorption through fiber/matrix debonding. Since with weaker interfacial bond strength the critical length is increased, there will be fewer fiber fractures and consequently more fiber pull-out. Both debonding and fiber pull-out increase the amount of energy absorption in the composite.

All this seems to promote CNTs usefulness as toughening elements for brittle composites, since a weak interface is not an inconvenience in that case.

5. Conclusion

It stems from all these considerations that CNTs have indeed a great potential as reinforcing elements for composites. The two serious steps to get through are in fact the achievement of (1) a large-scale low-cost production method of high purity material and (2) a good alignment procedure. Massive investment in large production units is starting in the US and Japan, patents have been registered, and 'long' ropes of good purity have already been produced [32]. Be patient and wait a few years. The future could be full of surprises . . . .

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