

## CHAPTER 11

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### ELASTIC PROPERTIES OF FULLERENES

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#### ABSTRACT

The recently synthesized fullerenes and their close relatives, nanotubes, constitute unusual materials with interesting, if not spectacular, mechanical properties. Because the field of mechanical properties of fullerenes and nanotubes is young and rapidly evolving, many of the current “values” of the mechanical constants (elastic moduli, tensile strength, etc.) are preliminary and subject to refinement with improved theoretical and experimental methods. Large uncertainties or inconsistencies between results of different research groups are readily apparent in many parameter values quoted below.

Both theoretical and experimental studies have been performed relevant to mechanical fullerene and nanotube response. This brief review tabulates the most important results to date. Although theoretical findings are quoted, the emphasis in the text is on experimental methods and experimental results. The reference list is extensive and is intended as a primary resource for obtaining theoretical and experimental details.

#### 11.1. INTRODUCTION TO FULLERENES AND NANOTUBES

Fullerenes and nanotubes have their roots in carbon chemistry. The bonding structure of the fundamental “molecular” units of both fullerenes and nanotubes is planar  $sp^2$ . The basic units (either small spherical molecules [1] or elongated molecular tubes [2]) may organize into higher-order structures including crystalline solids, where the primary

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attraction energy between the covalently bonded subunits is most often relatively weak van der Waals. Hence, there are strong analogies between these structures and rare-gas solids and layered graphitic-like materials.

Fullerenes are closed-cage pure-carbon shell structures, where the "fabric" of the shell is in part graphene-like. Graphene is a single atomic sheet of hexagonally bonded graphite. To achieve closure, however, the fabric cannot contain only hexagons of carbon; there must be exactly 12 carbon pentagons per closed fullerene molecule. The first, most famous, and most extensively studied fullerene is of course  $C_{60}$ , Buckminsterfullerene [1].  $C_{60}$  is a highly spherical molecule, with the location of the carbon atoms perfectly matching the seam vertices of a soccer ball. This molecule was first discovered in 1985 in the gas phase and was mass produced in bulk quantities several years later [3]. It is a convenient molecular source of pure carbon. Stable fullerenes with carbon number both smaller [4] and larger [5] than 60 have been produced.  $C_{60}$  molecules are easily produced in isolated form and they are relatively unreactive. Large collections of  $C_{60}$  molecules can arrange into close-packed forms, and single crystal specimens with dimensions several millimeters on a side have been grown. Below we consider the mechanical properties of the isolated molecules and of the "bulk" molecular crystals. Sizable single crystals of non- $C_{60}$  fullerenes are rare and their mechanical properties are virtually unexplored.

The empty spaces between  $C_{60}$  molecules in solid crystalline  $C_{60}$  may be intercalated with various atomic and molecular species. The electronic properties of the resulting compounds are interesting and include relatively high temperature superconductivity [6]. The stoichiometry of the superconducting materials is usually  $A_3C_{60}$ , with  $A$  an alkali metal. The elastic properties of  $A_3C_{60}$  superconductors have only been superficially studied. Under appropriate synthesis conditions, the intercalant species (again often alkali metals) can induce polymerization [7] of the  $C_{60}$  subunits, with a common stoichiometry of the stable polymerized material  $AC_{60}$ . We also consider below the elastic properties of such  $AC_{60}$  polymerized fullerenes.

Carbon nanotubes are pure carbon tubules that can have dramatic geometrical aspect ratios. Nanotubes with diameters of order 1 nm and length several hundred microns are not uncommon. A carbon nanotube can be thought of as a highly elongated fullerene, where the pentagons needed for closure are concentrated on the "caps" at the ends of the nanotube. The wall fabric of the nanotube, in the ideal case, then consists strictly of carbon hexagons. A related visualization of nanotubes is to consider them as pure graphene sheets rolled up into a single seamless cylinder. This forms the so-called single walled carbon nanotube (SWNT). The electronic properties of carbon nanotubes can be highly sensitive to the diameter and chirality of the resulting tube [8, 9], and some studies have indicated that the mechanical properties of carbon nanotubes depend on tube diameter as well. If SWNTs are synthesized within a narrow diameter distribution, the tubes that emerge from the synthesis chamber may be in a "crystalline" form, where many tubes are assembled in a close-packed array with parallel longitudinal axes. Such collections of SWNTs are termed nanotube ropes or bundles [10].

Interestingly, it is possible to synthesize nanotubes in a "multiwall" (MWNT) form, where from two to many (over 100) tubes nest almost perfectly one inside the other (Russian doll-like) [2]. The interaction between adjacent coaxial tubes is assumed to be of the van der Waals type, while on a given tube the carbon-carbon bonds are strong covalent  $sp^2$ -like. Crystalline ropes or bundles of carbon MWNTs have not been observed.

Nanotubes are not limited to pure carbon. Indeed, high-quality nanotubes have been experimentally produced from many different "layered material" hosts such as transition metal dichalcogenides [11], boron-nitride [12], and other boron and/or nitrogen

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containing  $B_xC_yN_z$  compounds [13, 14]. The mechanical properties of noncarbon nanotubes have been considered theoretically or experimentally only for selected  $B_xC_yN_z$  species.

A good general introduction to fullerenes and nanotubes can be found in the text by Dresselhaus, Dresselhaus, and Ecklund [15].

### 11.2. MECHANICAL PROPERTIES OF FULLERENES

The strength of a chemical bond can often be inferred from its bond length. In  $sp^3$ -bonded diamond, the C–C bond length is 1.54 Å, while in  $sp^2$ -bonded graphite the in-plane C–C bond length is 1.42 Å. Indeed, graphite has an in-plane stiffness (Young's modulus  $\sim 1$  TPa) exceeding the corresponding elastic modulus of diamond (bulk modulus 441 GPa). In a  $C_{60}$  fullerene molecule, there are two bond lengths (associated with the bond separating two hexagons and the bond separating a pentagon and a hexagon); these are 1.46 Å and 1.40 Å. This suggests that an isolated  $C_{60}$  molecule might be quite "stiff." Theoretical calculations [16] suggest that a single  $C_{60}$  molecule has an effective bulk modulus of 903 GPa. A crystalline solid of  $C_{60}$  molecules is predicted to have an effective bulk modulus of 668 GPa at a pressure that makes the "hard spheres" of  $C_{60}$  "just touch." These are impressive numbers but they have not been confirmed experimentally.

An early room temperature compressibility study [17] of solid  $C_{60}$  powders performed using x-ray diffraction found a linear  $a$ -axis compressibility  $d(\ln a)/dP = 2.3 \times 10^{-12}$  cm<sup>2</sup>/dyne, within experimental error the same as the interlayer ( $c$ -axis) compressibility of hexagonal graphite. The same study found for solid  $C_{60}$  a room temperature volume compressibility  $-d(\ln V)/dP = 6.9 \pm 1 \times 10^{-12}$  cm<sup>2</sup>/dyne, 3 and 40 times the values for graphite and diamond, respectively. These results confirm the weak intermolecular bonding of  $C_{60}$  solid. Subsequent studies [18–24] have found a range of compressibilities. Other studies have also examined the velocity of sound and Debye temperature [25–27].

Solid  $C_{60}$  undergoes a molecular rotational orientation ordering transition at  $T = 260$  K. Above this temperature, the crystal is fcc; below it is sc. This first-order structural transition affects the mechanical properties. In the high-temperature fcc phase the bulk modulus is significantly higher than in the sc phase (8.8 GPa vs. 6.8 GPa) [18]. Details of the transition are spectacularly revealed in single-crystal vibrating reed studies, where anomalies in the Young's modulus of order several percent are found at the 260 K fcc-sc transition [20, 28]. The vibrating reed studies also indicate a strongly temperature-dependent Young's modulus (43% decrease in the modulus from 6 K to 300 K), suggestive of large intermolecular anharmonicity. This anharmonicity has been accounted for within the framework of van der Waals bonding between molecules [28].

Table 11.1. Mechanical properties of fullerenes.

	$C_{60}$	$C_{70}$	$A_3C_{60}$	$A_1C_{60}$
Compressibility	$6.9 \times 10^{-12}$ cm <sup>2</sup> /dyn <sup>17</sup>		$-d \ln a/dP$ : $Rb_3C_{60}$ : $1.52 \times 10^{-2}$ GPa <sup>-1.32</sup> $K_3C_{60}$ : $1.20 \times 10^{-2}$ GPa <sup>-1.32</sup>	
Bulk modulus	8.8, 6.8 GPa <sup>18,19</sup> 13.4 GPa <sup>24</sup>	11 GPa <sup>29</sup>	$Rb_3C_{60}$ : 22, 20.5 GPa <sup>30,31</sup> $K_3C_{60}$ : 28 GPa <sup>30</sup>	$RbC_{60}$ : 28.7 GPa <sup>33,34</sup> $KC_{60}$ : 27.4 GPa <sup>33,34</sup>
Young's modulus	15.9 GPa <sup>20</sup>			
Velocity of sound $v_l$	$2.1 \times 10^5$ cm/s <sup>20</sup>			
Velocity of sound $v_t$	$3.6\text{--}4.3 \times 10^5$ cm/s <sup>19,27</sup>			
Debye temperature	$\sim 100$ K <sup>28</sup> 185 K <sup>25,26</sup>			

The elastic properties of solid  $C_{70}$  have also been examined. The  $C_{70}$  molecule is closely related to  $C_{60}$  but it is not spherical; rather it is elongated like a rugby ball. The bulk modulus of rhombohedral  $C_{70}$  crystal is 11 GPa [29].

The “doped” fullerenes are of special interest because of their dramatic electronic properties, including superconductivity. Extensive experimental and theoretical mechanical studies have not been performed. Experimental elasticity studies find for the bulk moduli of  $K_3C_{60}$  and  $Rb_3C_{60}$  28 GPa and 22 GPa, respectively [30]. A separate study found the bulk modulus of  $Rb_3C_{60}$  to be 20.5 GPa [31]. The pressure dependences of the lattice constant for  $K_3C_{60}$  and  $Rb_3C_{60}$  are, respectively,  $-d \ln a/dP = 1.20 \times 10^{-2}$  (GPa $^{-1}$ ) and  $1.52 \times 10^{-2}$  (GPa $^{-1}$ ) [32].

Polymerized  $AC_{60}$  crystals containing  $K$  and  $Rb$  have been investigated structurally using diamond anvil cell techniques and synchrotron scattering. These materials are mechanically highly anisotropic; the  $a$ -axis is the polymerized direction. As anticipated, the pressure dependence of  $a$  is much less than that of  $b$  or  $c$ . The room temperature zero-pressure bulk modulus is 28.7 GPa for  $RbC_{60}$  and 27.4 GPa for  $KC_{60}$  [33, 34]. These values significantly exceed the bulk modulus of pure (unpolymerized)  $C_{60}$ .

### 11.3. MECHANICAL PROPERTIES OF NANOTUBES

Carbon nanotubes predate the synthesis of other noncarbon nanotubes and they have been the the most extensively studied, both theoretically and experimentally. The virtually defect-free graphitic honeycomb network of carbon atoms of both single-walled carbon nanotubes and multiwalled carbon nanotubes suggests that various elastic moduli, such as the axial Young’s modulus, might experimentally approach “ideal” graphite values. Indeed this is the case. The weak van der Waals interaction between concentric nanotubes has also been exploited to fabricate low-friction bearings [35].

Many theoretical studies look at the elastic properties [36–49] and failure modes [50–53] of nanotubes. An early but comprehensive overview of the theory is presented by Ruoff and Lorents [37]. Here we will focus our discussion on experimental studies. A helpful review of recent experimental work is presented by Slavetac, Forro, *et al.* [54].

The primary experimental hurdle to overcome in accurately determining the mechanical properties of nanotubes is their small dimensions. Some measurements have been performed on “bulk” samples of nanotubes (collections of many nanotubes, usually randomly oriented and with ill-defined local density and intertube bonding) [55, 56], but in general such experiments are difficult to interpret in terms of intrinsic mechanical properties of individual nanotubes. Early studies of the mechanical properties of individual nanotubes were based on static transmission electron microscope (TEM) imaging of nanotubes in as-prepared samples [57–59]. Occasionally, external forces due to the interaction of a nanotube with its neighbors were found to cause a tube to buckle or collapse. From careful examination the failure modes, conclusions could be drawn about nanotube flexibility. In some instances, the nanotubes were observed to completely collapse into a ribbon-like structure, where the inner walls are separated by only the 3.4-Angstrom van der Waals distance [58]. The van der Waals attractive force makes this structure a metastable state, and the competition between the curvature energy to keep the tube open, and the van der Waals energy to keep the tube collapsed, yielded a direct measurement of the van der Waals force between graphene sheets [59]. Other examinations of van der Waals forces of nanotubes with other surfaces have been accomplished by atomic force microscopy [60, 61].

The first measurements of the Young’s modulus of nanotubes came from direct observation of thermally driven vibrations of a cantilevered nanotube [62]. The amplitudes of the oscillations could easily be imaged inside a TEM, and the amplitude as a function of temperature was mapped, giving an estimate of the Young’s modulus

Table 11.2. Mechanical properties of nanotubes.

	Material	SWNT	SWNT rope	MWNT Arc-grown	MWNT Catalytically grown	$B_xC_yN_z$ Nanotubes
Young's modulus	Theoretical	~500 GPa <sup>37</sup> ~1 TPa <sup>39,41,42,48</sup> ~350 GPa <sup>40</sup> ~1.2 TPa <sup>44,47</sup>	0.4–0.8 TPa <sup>41,46</sup>	~1 TPa <sup>39,41,46</sup>		BN: ~0.9 TPa <sup>44,47</sup> BC <sub>3</sub> : ~0.9 TPa <sup>44,47</sup> BC <sub>2</sub> N: ~1.0 TPa <sup>44,47</sup> C <sub>3</sub> N <sub>4</sub> : ~0.6 TPa <sup>47</sup> BN: 1.22 TPa <sup>76</sup>
	Experimental	1.25 TPa <sup>65</sup> ~1 TPa <sup>69</sup> 2.8–3.6 TPa <sup>73</sup>		1.8 TPa <sup>62</sup> 1.2 TPa <sup>67</sup> 0.8 TPa <sup>68</sup> 0.1–1.2 TPa <sup>70</sup> 0.27–0.95 TPa <sup>72</sup> 1.7–2.4 TPa <sup>73</sup> ~0.45 TPa <sup>39,41</sup>	30 GPa <sup>68</sup> 450 GPa <sup>55</sup>	
Shear modulus	Theoretical	~0.45 TPa <sup>39,41,48</sup>	~1 GPa <sup>69</sup>			
Bending strength	Experimental					
	Theoretical	0.155 nm/d <sup>2</sup> Crit. curvature <sup>38</sup>				
Tensile strength	Experimental			14 GPa <sup>67</sup>		
	Theoretical	5–10% strain <sup>50, 53</sup>				
Experimental		>45 GPa <sup>71</sup>	>45 GPa <sup>71</sup>	11–63 GPa <sup>72</sup>	1.72 GPa <sup>55</sup>	

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only the outer layers of the multiwall nanotube were truly under tension. This yielded a MWNT Young's modulus of 270 to 950 GPa, and a tensile strength of 11 to 63 GPa.

Also of note is a small collection of Raman spectroscopy studies that investigated specifically the elastic moduli of carbon nanotubes [73–75]. The phonons at  $k \rightarrow 0$  correspond to uniform compression or bending of the nanotubes, and thus give an ensemble measurement of the elastic properties of many nanotubes, with good statistics. For carbon SWNT, a Young's modulus of 2.8 to 3.6 TPa has been inferred, while for MWNT the Young's Modulus is 1.7 to 2.4 TPa.

There are several theoretical studies of the mechanical properties of noncarbon nanotubes. However, due to a dearth of suitable samples, there has only been one experimental investigation. The thermal vibrations of a boron nitride MWNT have been analyzed, and yield a Young's modulus of  $1.22 \pm 0.24$  TPa [76].

The extremely high moduli and strengths for nanotubes suggest that they might make good mechanical elements on the nanoscale. Macroscopic-sized components, however, would require the nanotubes to be embedded in some sort of composite. Some early studies have been done on nanotube composites [77–80], but results are still preliminary.

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