The Study of Bundling Effects on Carbon Nanotubes
GM Pressure Coefficients via ODA Functionalized
Tubes

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Abstract

The study of carbon nanotubes(CNTs) G-Mode(GM) pressure coefficients, reflecting the stiff \textit{sp\textsuperscript{2}} bond pressure dependence, is essential to the understanding of their extraordinary mechanical properties as well as fundamental mechanics. However, it is hindered by the availability of CNTs samples only as bundles or isolated with surfactants. Octadecylamine(ODA) functionalized CNTs are mostly of a single diameter and can be stably dispersed in 1, 2-dichloroethane(DCE) and chloroform. Here we perform high pressure Raman spectroscopy on these tubes and obtain their experimental GM pressure coefficients, which are unaffected by surfactants, for individual CNTs and bundles. The \textbf{G}\textsuperscript{+} pressure coefficient for bundles is only about half of that for individual CNTs in DCE and is about two-thirds in chloroform. For bundles, the \textbf{G}\textsuperscript{−} pressure coefficient is about one-third of \textbf{G}\textsuperscript{+} in DCE and about the same in chloroform. These results are beyond the framework of the current understanding on the GM pressure dependence.

1. Introduction

Carbon nanotubes(CNTs) are known for their extraordinary mechanical properties. In particular, their exceptionally high Young’s moduli in the tera-
pascal range, resulting from the in-plane $sp^2$ bond between the carbon atoms, make CNTs the stiffest materials yet discovered [1]. To study and understand the huge resistance to compression, relating to the pressure dependence of the covalent $sp^2$ bond, Venkateswaran et al. performed the first Raman spectroscopy experiment on CNTs under high pressure and got the pressure coefficients of their in-plane vibrational modes G-Mode (GM) in 1999 [2]. If not specified, GM refers to the concentrated $G^+$ mode, vibrating along the tube axis, rather than the $G^-$ perpendicular to axis. The upshifts of CNTs GM frequencies with pressure are believed to be mostly induced by the $sp^2$ bond stiffening, as in the cases of graphite [3] and graphene [4]. Therefore, it is reasonable to consider that the GM pressure coefficients should be similar for graphite, graphene and CNTs. Not considering some extreme environmental effects, graphene does have similar GM shift rates to graphite [3, 4], while CNTs have a wide range of values, reported by different research groups over the past 15 years [2, 5, 6, 7, 8, 9, 10, 11]. Ghandour et al. attributed the disparity to the tube diameter and explained the diameter dependence of the GM pressure coefficients with a thick-wall tube model [12]. The researchers are now well-aware of such diameter dependence, although the model itself is not perfect, in a lower predicted graphene GM pressure coefficient than experimental value, and not taking recently reported chirality effects into account [13]. Instead of measuring tubes of mixed diameters, Raman measurements now typically involve tubes of a single diameter, which is mainly done in two ways: using resonance Raman spectroscopy (RRS), which in principle requires a tunable laser or using CNTs samples of a single diameter.

So far, lots of results on CNTs have been reported. Most of the works, as mentioned above, obtained different GM pressure coefficients from each other on mixed diameters samples at certain laser excitation wavelength in various pressure transmit media (PTM), as shown in Figure 1. These are examples of close-ended bundled tubes. The disparity comes from the mixture of diameter and environmental effects [12]—certain environment (PTM, bundling and surfactants) and excitation wavelength pick out tubes of a certain diameter.
Recently the results on CNTs in water assigned the GM pressure coefficients of 4.6 cm\(^{-1}\)GPa\(^{-1}\) to (9,1) tubes, 5.0 cm\(^{-1}\)GPa\(^{-1}\) to (11,0) and (10,2) mixture and 6.2 cm\(^{-1}\)GPa\(^{-1}\) to (12,1) and (11,3) mixture respectively [12].

Importantly, apart from intrinsic effects such as diameter and chirality, the pressure dependence of GM can be affected by exogenous effects, such as bundling, due to the van der Waals interaction between the tubes within a bundle. Moreover, bundling effects on the GM pressure coefficient of the tube picked out by RRS may vary with factors such as the diameters of the surrounding tubes, the bundling configuration (tangled, etc) and the degree of bundling (affected by the sample concentration but cannot be precisely controlled). The latter two are inaccessible for samples of a single diameter.
Researchers compared the GM shift rates of individual tubes to the ones of bundles. Due to van der Waals interaction, CNTs tend to form bundles. Surfactants stably disperse CNTs, which allows to exclude the bundling effects and their uncertainty while the surfactant effects (van der Waals interaction between typically large ambipolar surfactant molecules and CNTs) and their uncertainty (the surfactant distribution) are possibly introduced. It is worth noticing that though shifting the GM frequency, the contribution of the new ions in solution brought by surfactants does not affect considerably the CNTs pressure response (the GM pressure coefficients). Again, in literature most researches on individual tubes were done on CNTs samples of various diameters and reported various values of GM pressure coefficients of tubes surrounded by surfactants (see Figure 1).

Octadecylamine (ODA) functionalized CNTs provide a solution to study the GM pressure dependence, excluding the diameter effects and the strong van der Waals interaction between tubes or tube and surfactants. Venkateswaran et al. firstly applied the ODA functionalized tubes to the CNTs high pressure study in 2001. However, they used ODA tubes in solid (powder) form, which were still small bundles and no further research has been done on CNTs GM pressure coefficients by ODA tubes since then. In this work, we perform a more complete study, with a clear account for the advantages and disadvantages of using such tubes.

The typical bundle diameter of the solid form of ODA functionalized tubes is 2–8 nm while the length is 0.5–1 μm, which K.J. Donovan et al. considered as a very low degree of bundling. They can be stably dispersed in certain organic solvent, such as 1,2-dichloroethane (DCE) and chloroform, without the aid of surfactants. In fact, DCE disperses well even non-functionalized single-walled carbon nanotubes (SWCNTs) after probe-sonication to initially break the bundles, which can be imaged by STM techniques. The steric hindrance caused by the functional group coverage of SWCNTs between 1.8 and 3.2 ODA chains per nanometre stabilizes the suspension. It must be noted that as a result of acid treatment during the ODA functionalization, the caps at the
end of tubes are removed \[18\]. The band-gap transitions are largely disrupted \[19\] and therefore no resonance condition applies. It has the advantage that the contribution of CNTs in a given sample to the Raman spectrum is independent of the laser excitation wavelength.

2. Experimental

The ODA functionalized tubes were employed as purchased from Carbon Solutions. Fig 2 schematically shows an ODA functionalized tube \[15\]. They were synthesised by the arc discharge method, treated by nitric acid and functionalized with ODA \[19\]. The manufacturer specifies that the carbonaceous purity is over 90\%, in which SWCNTs loading is 65\%±15\%, determined by solution-phase near-IR spectroscopy \[20\]. When firstly synthesising ODA tubes, Chen \textit{et al.} reported a single radial breathing mode (RBM) Raman peak at 170 cm$^{-1}$ in CS$_2$, at 1064 nm excitation wavelength \[19\], indicating that they are of single diameters 1.41 nm, according to the commonly used relation \[21\].

$$d = \frac{215}{\omega_{RBM} - 18}$$  \hspace{1cm} (1)

We prepared 4 samples of ODA functionalized CNTs— bundled SWCNTs (b-SWCNTs) and individual SWCNTs (i-SWCNTs) in DCE, b-SWCNTs and i-SWCNTs in chloroform, following the recipe (initial probe-sonication time, power of the sonication, etc.), which Donovan \textit{et al.} used in their study \[15, 17\]. The dispersion was tested by samples dichroism \[15\] and viscosity \[17\], which were determined by samples polarizability, thus sensitive to bundled/individual status. The concentrations of the samples were $1\times10^{-4}$ wt\% for b-SWCNTs in DCE, $1\times10^{-6}$ wt\% for i-SWCNTs in DCE, $1.5\times10^{-4}$ wt\% for b-SWCNTs in chloroform and $1\times10^{-6}$ wt\% for i-SWCNTs in chloroform. The signal to noise ratio of their Raman spectra is low, due to the extremely low samples concentration. It also must be emphasized that the Raman experiments were performed under non-resonance conditions as the band-gap transitions of these
tubes are largely disrupted and thus the peak intensities were up to 6 orders of magnitude lower than those under resonance conditions.

Room temperature non-polarized Raman spectra of the samples were obtained in the backscattering geometry with a Horiba T64000 Raman system with a confocal microscope that had a resolution of 0.6 cm$^{-1}$, a single 1800 grooves/mm grating and a 100-µm slit, and was equipped with a liquid N$_2$-cooled CCD detector (Jobin-Yvon Symphony). Suitable edge filters for 488 nm, 514 nm and 647 nm lines of a Coherent Innova Spectrum 70C Ar$^+$-Kr$^+$ laser could be used with the system. We kept the laser power on the sample below 5 mW to avoid laser-heating effects on the probed material and the concomitant softening of the observed Raman peaks.

For the high pressure experiments, we used a membrane diamond anvil cell with two diamond anvils of 500 µm culet size and very low fluorescence (Type IIa). Ruby luminescence R1 line was used for pressure reference $^{22}$. The 4 samples were separately loaded into the cells. We used a 20× objective on the b-SWCNTs, i-SWCNTs in DCE and i-SWCNTs in chloroform in the pressure
cell and a 40× objective on b-SWCNTs in chloroform. We set the pinhole size in confocal configuration at 200 µm. We obtained RBM and GM spectra of all the samples at 488, 514 and 647 nm lines after loading them into cells, where pressure has already been applied to prevent solvent from evaporating, and took Raman at further pressure points at 488 nm for b-SWCNTs in DCE and b-SWCNTs in chloroform, and at 514 nm for i-SWCNTs in DCE and i-SWCNTs in chloroform. With the chosen objectives, pinhole size and excitation wavelength, the spectra qualities of the corresponding low-concentrated samples are slightly better than with other settings. The signal to noise ratio of the GM spectra decreases with pressure and therefore the study is at low pressure range, well below 10 GPa, which is the experimental collapsing point of CNTs of a diameter distribution [23].

The hydrostaticity in this high pressure experiments has been studied. DCE solidifies at 0.6 GPa [24] and chloroform between 0.60–0.79 GPa [25]. We obtain very similar ruby R1 wavelength from two different ruby chips in the cell: 694.66 and 694.67 nm at 1.20/1.24 GPa, 694.86 and 694.87 at 1.74/1.76 GPa for b-SWCNTs in chloroform, after observing the solvent solidification. The corresponding errors in pressure are 3.3% and 1.1%, showing an acceptable hydrostaticity in this high pressure study.

3. Results and Analysis

Figure 3 shows the raw RBM spectra of the dry sample on a glass slide and 4 prepared samples in cells. The spectra are vertically shifted for clarity to compare the spectra of i-SWCNTs to b-SWCNTs.

According to literature, we assign the peaks at 302 cm\(^{-1}\) to DCE [24] and 251 and 368 cm\(^{-1}\) to chloroform [25]. Fit CNTs RBM frequencies (Lorentzian fit) of the dry sample are at 164.9 and 179.5 cm\(^{-1}\), and correspond to tubes of diameters 1.46 and 1.33 nm, according to Eq. [1]. The ratio of the peaks’ integrated area is 9.85:1, former to latter. The small RBM peak cannot be detected for samples loaded into cells as the absorption by diamonds weakens the
signal. For i-SWCNTs which are at an order of magnitude lower concentration than b-SWCNTs, even the main peak is no longer detectable.

We need to consider the effect of concentration on CNTs GM pressure coefficients, as i-SWCNTs and b-SWCNTs, which we are going to compare, are of different concentration. Figure 4 presents the GM spectra obtained in high pressure measurements on b-SWCNTs in chloroform, from two different spots—one in a dark area, which is richer in bundles, making it observable under microscope, and the other in a transparent area, which is less concentrated, but still of bundles. We label the GM spectra as concentrated bundles and diluted bundles. The baselines are subtracted, and then the spectra are vertically shifted, proportional to pressure. Importantly, figure 4 shows that the GM frequencies are unaffected by the sample concentration at the first four pressure points and therefore it is reasonable to consider that the GM pressure coefficients are independent of the sample concentration in the low pressure range in this study.

Figure 5 exhibits the GM spectra of b-SWCNTs and i-SWCNTs at similar pressure points in both DCE and chloroform. For b-SWCNTs in chloroform, the spectra are of concentrated bundles in Figure 4. For i-SWCNTs in DCE and chloroform, the Raman intensities are multiplied by a factor 200, in order to get clear comparisons to the ones of b-SWCNTs. As a result, the noise of i-SWCNTs spectra is obviously increased. The baselines are subtracted, and then the spectra are shifted vertically, proportional to pressure. The narrow peak at 1554.4 cm\(^{-1}\) in the GM spectrum of i-SWCNTs in DCE is assigned to an oxygen vibrational Raman peak from the air between the microscope and the cell \[27\]. It is on top of a wide peak, which can be from carbonaceous impurities in the samples. The GM peak is right next to the wide peak and the signal to noise ratio is low.

We fit the GM spectra of i-SWCNTs in DCE and chloroform in Figure 5 with a single Lorentzian and the GM spectra of b-SWCNTs in DCE and chloroform in Figure 5 with two Lorentzians. In the latter case these correspond to the \(G^+\) and \(G^-\) peaks, which are initially well separated but cannot be told apart with pressure increased. We fix the integrated area ratio of \(G^+\) to \(G^-\) at the
Figure 3: The RBM spectra of ODA functionalized tubes are shown for (a) dry samples on a glass slide, (b) b-SWCNTs (black) and i-SWCNTs (red) in DCE and (c) b-SWCNTs (black) and i-SWCNTs (red) in chloroform. In (b) and (c) the spectra are vertically shifted for clarity. Laser excitation wavelengths are at 488 nm for b-SWCNTs in DCE and b-SWCNTs in chloroform, and at 514 nm for i-SWCNTs in DCE and i-SWCNTs in chloroform. Raman shifts do not vary with the excitation wavelength.
Figure 4: The GM spectra of b-SWCNTs in chloroform collected from (a) the dark area labelled black and (b) the transparent area labelled blue are presented. The spectra are vertically shifted, proportional to pressure. The pressure, under which the spectra are obtained, is labelled. Laser excitation wavelength is at 488 nm.
Figure 5: The GM spectra of i-SWCNTs (blue) and concentrated b-SWCNTs (black) are shown in (a) DCE and (b) chloroform. For i-SWCNTs the Raman intensities are multiplied by 200. The spectra are vertically shifted, proportional to pressure. The pressure, under which the spectra are obtained, is labelled in corresponding colours to b-SWCNTs or i-SWCNTs. The oxygen vibrational Raman peaks are labelled. Laser excitation wavelengths are at 488 nm for b-SWCNTs in DCE and b-SWCNTs in chloroform, and at 514 nm for i-SWCNTs in DCE and i-SWCNTs in chloroform. Raman shifts do not vary with the excitation wavelength.
Figure 6: The GM frequencies of b-SWCNTs in DCE are plotted against pressure. The frequencies are obtained by fixing the integrated area ratio of G⁺ to G⁻ at the free fitting value at 1.01 GPa (black) and releasing all the fitting parameters of the two Lorentzians (blue). The squares are for G⁺ and the circles are for G⁻. The error bars are shown, where they exceed the size of the points.

The value obtained by free fitting at the first pressure point during the whole fitting, to avoid unphysical fitting results as larger G⁻ than G⁺ peak, obtained by releasing all the fitting parameters of the two Lorentzians. Figure 6 shows how these two fitting procedures lead to different GM frequencies of b-SWCNTs in DCE. The difference is mainly at the uncertain frequencies of the weak G⁻ peak. We plot GM frequencies, obtained by fixing the integrated area ratio, of all the samples against pressure with error bars in Figure 7. Linear least square fits are shown, excluding the points of b-SWCNTs in DCE at 3.23 GPa, for the similar pressure range to that of i-SWCNTs in the same solvent and the point of i-SWCNTs in DCE at 0.63 GPa as an abnormal point, which is exactly at the DCE solidification point. The excluded data points are labelled green.
Figure 7: The GM frequencies of all the samples are plotted against pressure. The colour black is for $G^+$ frequencies of b-SWCNTs, red is for $G^-$ of b-SWCNTs and blue is for $G^+$ of i-SWCNTs. The squares are for samples in DCE and the circles are for chloroform. The error bars are shown, where they exceed the size of the points. The linear fit is presented in solid line for DCE and dash line for chloroform, excluding the points of b-SWCNTs in DCE at 3.23 GPa and i-SWCNTs in DCE at 0.63 GPa, which are labelled green.
Table 1: GM pressure coefficients for all measured samples

|                  | $G^+$ | $G^-$ |
|------------------|-------|-------|
| b-SWCNTs in DCE  | 6.6±0.7 | 2.0±0.1 |
| i-SWCNTs in DCE  | 13.8±0.6 |
| b-SWCNTs in chloroform | 7.1±0.3 | 6.7±0.7 |
| i-SWCNTs in chloroform | 10.2±1.3 |

We present the GM pressure coefficients in Table 1 from the linear fit in Figure 7. The errors are from the linear fit, the Lorentzian fit for the peak position and the system resolution.

Figure 7 and Table 1 present the key result that the $G^+$ pressure coefficient for bundles is only about half of that for individual CNTs in DCE and is about two-thirds in chloroform. For bundles, the $G^-$ pressure coefficient is about one-third of $G^+$ in DCE and about the same in chloroform.

4. Discussion

At the moment we do not fully understand these results, on the following points. 1) Unlike the pressure dependence of RBM, which is due to the decrease of the distance between tube shell and absorbed fluid layer and therefore sensitive to the environment [28], the pressure dependence of GM is commonly considered as determined by the shortening of $sp^2$ bond length, which has little to do with the environment, contradicting the apparent bundling and solvent effects reported here. 2) According to the thick-wall tube model [12], the $G^+$ and $G^-$ pressure coefficients of 1.46 nm tubes should be 6.0 and 8.0 cm$^{-1}$GPa$^{-1}$ respectively. The model is based on individual tubes but the $G^+$ values of i-SWCNTs are much higher than the predicted ones. The $G^+$ values of b-SWCNTs agree well, as the previous work on bundles [12]. 3) Tangential stress is always larger than axial stress for a tube under hydrostatic pressure. The pressure coefficient of the vibrational mode along tube circumference ($G^-$) should be always larger than the one along tube axis ($G^+$). This is again against our observations in
DCE. 4) Considering that the end of the tubes has been removed, the solvents may possibly enter inside the tube. If the internal pressure (pressure inside the tube) is at a value between 0 and the external pressure, the pressure coefficients for both $G^+$ and $G^-$ should lie between the graphene value and the thick-wall tube model predictions. Results of i-SWCNTs in Table 1 are out of such range. The normal way to judge whether tubes are solvent-filled by the shift of RBM frequency is not possible in this case [29], because no close-ended ODA functionalized CNT is available for comparison (caps removed during the ODA introducing).

ODA functionalized CNTs, as samples to study the CNTs GM pressure coefficients, have the following advantages. First, given that they are mostly of a single diameter and their band-gap transitions are largely disrupted, the contributions to the RBM and GM signals from tubes of different diameters may be taken proportional to their contents in samples, regardless of the laser excitation. In Figure 3, we obtained the RBM integrated area ratio of 1.46 nm to 1.33 nm tubes at 9.85 to 1. The $G^+$ signal contributes from 1.46 and 1.33 nm tubes, also with a ratio of 9.85 to 1. It is reasonable to attribute the GM pressure coefficients in Table 1 to 1.46 nm tubes only. Second, the ODA side chains offer the steric hindrance and therefore provide us with SWCNTs samples stably dispersed without the aid of surfactants. This is the main reason we use ODA CNTs in this study.

There are related disadvantages, namely the limited PTM we can use for dispersion and the potential side chain effect on GM pressure coefficients. DCE and chloroform are effective in dispersing CNTs samples but are not considered as good PTM for their low solidification pressures. While excluding the inter-tube or tube-surfactant van der Waals interaction, we introduce the side chains. The ODA chains coverage between 1.8 and 3.2 chains per 150 carbon atoms may be high from the Chemistry point of view, but too low to make a comparable effect on the pressure dependence of in-plane vibrational frequencies to the bundles or surfactants. The upshift of GM frequency with pressure is induced by the increasing overlap of electrons of carbon atoms. In the case of bundles
or surfactants, each carbon atom is under the influence and the behaviour of its electrons can be affected, while in the case of ODA tubes, electrons of most carbon atoms are very unlikely affected by the $sp^3$ defects (1.8–3.2 in 150).

5. Conclusion

The $G^+$ pressure coefficient for bundles is only about half of that for individual CNTs in DCE and is about two-thirds in chloroform. For bundles, the $G^-$ pressure coefficient is about one-third of $G^+$ in DCE and about the same in chloroform. Such comparison excludes the effect of surfactants, which can only be achieved by using ODA functionalized tubes at the moment. The origin of the bundling and solvent effects on GM pressure coefficients is unclear at the moment and the reported shift rates values in this work are beyond the framework of the current understanding, including a previously reported thick-wall tube model.

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