Selective Enhancement of Inner Tube Photoluminescence in Filled Double-Walled Carbon Nanotubes

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A highly selective enhancement of the optical response of the inner tubes of double-walled carbon nanotubes has been identified upon transformation of the residual C atoms inside the hollow core to linear carbon chains (LCC). By varying the growth conditions and using standardized suspensions, it has been observed that this optical response depends sensitively on the tube diameter and LCC growth yield. It is reported how the formation of LCC by postsynthesis annealing at 1400 °C leads to an increase of the photoluminescence (PL) signal of the inner tubes up to a factor of 6 for tubes with (8,3) chirality. This behavior can be attributed to a local charge transfer from the inner tubes to the carbon chains, counterbalancing quenching mechanisms induced by the outer tubes. These findings provide a viable pathway to enhance the low PL quantum yield of double-walled carbon nanotubes and proof the capability of inner tubes to exhibit photoluminescence.

1. Introduction

Double-walled carbon nanotubes (DWCNT) are a class of carbon nanostructures formed from two graphene sheets conceptually rolled into two concentric tubes. As such, they combine the outstanding properties of single-walled (SW) and multiwalled carbon nanotubes (MWCNT): The outer tube acts as a shield for the inner tube, granting a very high stability even when confronted with highly aggressive mechanical,[1] thermal,[2] and chemical treatments.[3] The latter is of special importance since the optical properties of SWCNT are easily altered or even suppressed due to environmental effects.[4] Iakoubovskii et al. could reveal in optical absorption and photoluminescence (PL) measurements that this shielding process in DWCNT is efficient even when ozone oxidation is applied,[5] and Hayashi et al. had similar observations of PL in fluorinated DWCNT.[6] Despite this fascinating effect, the major drawback for exploiting the optical properties of DWCNT is their seemingly low PL quantum yield (PL QY), which even raises the question if they are capable of exhibiting significant PL at all: Interactions between inner and outer tube are held responsible for an efficient quenching of inner tube photoluminescence.[7] Zólyomi et al. found that a charge transfer from outer to inner tube in the order of 0.001 e− per atom takes place, implying that the inner tube is always negatively charged.[8] The opinions of the research community still have not reached a consensus in this respect, whether any detected PL originates from the inner tubes of DWCNT or from SWCNT residing as impurities within a sample. A plethora of contradicting conclusions has been reported throughout the literature, ranging from observations of inner tube PL with highly diverging magnitudes[9–11] to the verification of its almost nonexistence.[12,13]

Sample preparation is a crucial factor, as we showed in our recent work where we could trace these differences to different sonication and purification techniques.[14] Still, the PL QY for the inner tubes of DWCNT was a factor of 50 lower than for the same type of tubes existent as SWCNT, similar to the value found by Green et al. for metalliclicity sorted DWCNT.[10]

To increase the photoluminescence of carbon nanotubes, several methods can be found in the literature: Oxygen doping has been shown to increase the PL QY compared to pristine SWCNT, which was assigned to the localization of the otherwise mobile excitons.[15,16] Piao et al. observed PL brightening by adding aryl diazonium salts which introduces sparsely distributed sp3 defects.[17] They attributed the amplification to the appearance of a new, low-energy peak that lies below the dark exciton state seen in the exciton fine structure thus increasing the population of possible PL emitters. However, due to the above mentioned shielding mechanisms in DWCNT, these forms of sidewall functionalization are not effective. Instead, filling the hollow core of the inner tube comes to mind: in an earlier report, we showed that SWCNT filled with metalorganic ferrocene molecules exhibit an increase in photoluminescence intensity due to a charge transfer between the nanotube and the ferrocene, cancelling out the doping effects originating from the opening process and the surfactant molecules.[18] As the ferrocene molecule is too big for encapsulation inside small diameter inner tubes, linear carbon chains (LCC) where used in this study. In two recent reports, we demonstrated not only the successful synthesis of these peculiar linear carbon allotropes,[19]...
but also that the LCC invoke a charge transfer from inner tube to the LCC which depends on their length, and that the length distribution can be tailored by using appropriate growth conditions (especially the synthesis temperature and inner tube diameter). This charge transfer from inner tube to LCC in return has the same order of magnitude as the one reported by Zólyomi et al. for the charge transfer from outer tube to inner tube, therefore potentially neutralizing the outer tube's influence in order to restore the inner tube's intrinsic optical properties. At last, an essential aspect for the discussion of the earlier mentioned capability of inner tubes to exhibit PL is that the LCC could only be grown inside the inner tubes of DWCNT and not within SWCNT of the same diameter. With this contribution we show for the first time that functionalization with linear carbon chains represents an ideal pathway to selectively enhance the optical response of inner tubes from DWCNT. By using standardized suspensions of DWCNT either pristine or filled with LCC grown at different temperatures, we demonstrate that the optical absorption and PL of the inner tubes does not only depend on their chirality but also on the chain length distribution within the sample. PL line scans clearly show a diameter dependent optimum for this enhancement and three different regions regarding the inner tube diameter. Concomitant to previous results, these regions can be assigned to nanotube species being ideal for the growth of the longest linear carbon chains as well as those tubes with an optimum balance between diameter and chain length for PL enhancement or tubes being too large.

2. Results and Discussion

2.1. Characterization of LCCs@DWCNTs Grown at Different Temperatures by Raman Spectroscopy

The DWCNT samples used in this study were synthesized with a procedure recently reported elsewhere. The same also accounts for the growth of linear carbon chains inside the inner tubes of the DWCNT. Both procedures and the details of their growth inside DWCNT with thin inner tubes are summarized in the Experimental Section provided after the main text. We have optimized the diameter distribution of the DWCNT for inner tube diameters between 0.6 and 1.1 nm suitable for near-infrared PL by a multifrequency Raman analysis of the radial breathing modes. Regarding the LCC filling, our previous study revealed that only DWCNT with inner tubes between about 0.6 and 0.9 nm are suitable for a high yield growth of LCC and that more than 90% of those tubes are filled by LCC. Details about the determination of this bulk filling with LCC are given by Shi et al. and in the Experimental section. Before deepening into the effect of LCC encapsulation inside DWCNT on the optical properties of their inner tubes, we have to revisit the Raman response of DWCNT samples filled with LCC grown at different temperatures. In bulk measurements, LCC can be detected by Raman spectroscopy, as they exhibit a Raman active mode in the range between 1790 and 2200 cm⁻¹. The frequency of the observed Raman shift of the mode assigned to the carbon chains (LCC-Band) is strongly correlated to the chain length, with longer chains giving smaller shifts. Consequently, when investigating on a bulk sample with different chain lengths, the transformation of the convoluted LCC signal line shape can be explained by different chain length distributions between the samples, meaning that a higher intensity on the low frequency side indicates a higher abundance of longer carbon chains and vice versa.

For our chains inside DWCNT a quasilinear relation between the inverse chain length and the red shift of the Raman frequency of the LCC-Band is observed. This is in strong contrast to chains in vacuum, where a clear saturation of the Raman frequency emerges when approaching the infinite chain limit. This shows that the environment of the DWCNT and especially the charge transfer between the inner tube and the linear carbon chains plays an important role. Recent ab initio calculations by Wanko et al. confirmed that, in agreement with the experimental observation, this charge transfer has linear relation on the red shift of the Raman frequency and becomes important for confined LCCs. It is consistent with the one between inner and outer tubes in a pristine DWCNT. This points out that the charge transfer to the inner tubes, which could be the main reason for the low PL response of DWCNT inner tubes, might be balanced by a charge transfer to the carbon chain in this LCC@DWCNT hybrid system.

In Figure 1a the Raman response of the G-Band and the LCC-Band after excitation at 2.18 eV (568.2 nm) is plotted with all signals being normalized to the intensity of the G-Band. Interestingly, the shape of the latter is only altered considerably when the annealing temperature of 1460 °C is reached, where two extra peaks appear as shoulders of the main peak that slightly diminish at even higher growth temperatures. The appearance of the G-Line resembles the one found by Andrade et al. for MWCNT with encapsulated carbon chains after a high pressure treatment. In general, the frequency of graphitelike modes of pristine (not filled) nanotubes is pressure dependent as the main peak can either shift up (compression) or down (strain). By performing temperature dependent Raman measurements, we found that the LCC and the inner tube are coupled by van der Waals interactions. In the case for the G-Band, the LCC may locally induce a strain in the inner tube, which then causes a compression in other parts of the same nanotube, enabling the observation of both effects. Also, the height of the two extra shoulders diminishes with increasing temperature, concomitant with the decomposition of the carbon chains, which we discuss later. For the LCC mode itself, several factors have to be taken into account: The utilized excitation energy of 2.18 eV (568.2 nm) allows to detect LCC of different lengths very efficiently as it is close to the resonance energy of long (>60 carbon atoms) and short (down to 10 carbon atoms) LCC. By assuming that the amount of material available for carbon chain synthesis compared to the nanotube abundance is similar for all samples, the intensity of the different chain length contributions to the overall Raman response solely depends on the number of LCC being most in resonance with the incident laser. The highest intensity was always found for a frequency of 1852 cm⁻¹, indicating that LCC containing ~40 carbon atoms are most in resonance with the laser.

The total LCC-Band is composed of signals from many different long chains, since the varying diameters and chiralities
of inner tubes provide different growth conditions for the LCC. Although it is not possible within the given spectrometer resolution to directly assign a certain chain length (i.e., Raman frequency) to only one specific (n,m) value, measurements on the radial breathing modes of the inner tubes revealed that only one specific (n,m) value, measurements on the radial breathing mode and the ideal nanotube diameter for LCC growth, this decomposition can be traced to inner tubes owing a diameter deviating too much from the ideal value. Therefore, they are not providing enough stability for the chains, in contrast to their thinner counterparts that provide more stability to the chains and are capable of containing and protecting the longest LCC. Eventually, by further raising the growth temperature, the aforementioned trend continues.

2.2. Optical Absorption of the Differently Treated DWCNT Samples

After the Raman measurements all samples were solubilized to elucidate on their optical properties. The parameters of the procedure on how homogeneous DWCNT suspensions are received are of utmost importance for the absorption and the photoluminescence of DWCNT. The applied technique follows a method which has proven that inner tube extraction can be avoided. The exact details can be found in the Experimental Section. **Figure 2a** shows the absorption spectra of a pristine, not-filled DWCNT sample in comparison with HiPco SWCNT before background subtraction as reported earlier, (n,m) assignment was performed based on the work of Weisman and Bachilo. Features of the excitonic transitions between corresponding van Hove singularities of DWCNT are strongly attenuated in comparison to SWCNT. On the one hand, a decrease in absorption features is often a result of carrier/hole doping to the carbon nanotube as observed for SWCNT. On the other hand, the very nature of absorption measurements has to be considered, as in ensemble measurements contributions from different inner and outer tubes can be observed. By using an ozone etching technique, Iakoubovskii et al. could deconvolute the signals and showed for their samples that the signals of E11 transitions of inner tubes and E22 transitions of outer tubes were overlapping, “blurring” the inner tube signal. A similar observation of overlapping inner and outer tube contributions was also reported by Miyata et al. who extracted the inner tubes. Since the diameter distribution for their samples was similar as it is in this work, such overlaps can also be expected.

**Figure 2b** shows the normalized (normalization procedure described in the Experimental Section) absorption spectra for E11 transitions of the pristine, not-filled DWCNT sample and two differently filled DWCNT samples: the DWCNT annealed at 1460 °C that inhibit the highest number of the longest chains and the DWCNT annealed at 1400 °C, as these showed the greatest change in absorption features compared to the pristine sample. For all filled DWCNT, the size and shape of the inner tube’s E11 peaks are altered compared to
the pristine sample. For example, the signal of the (7.5) species is much more prominent for all samples, also the (8.3) and (6.5). Another interesting feature is the peak around 1120 nm that represents a superposition of E_{11} peaks for the (8.4) (d_i = 0.84 nm), (7.6) (d_i = 0.89 nm), and (9.4) (d_i = 0.91 nm) species. The shape of this peak changes for the annealed samples compared to the pristine sample. This is a strong hint that the excitonic transitions for these tubes are affected differently by the encapsulated carbon chains which can be related to the growth capabilities for LCC given by the nanotube diameter.[19] Interestingly, as already mentioned, the strongest enhancement of absorption features is observed for the sample annealed at 1400 °C. On the one hand, we found the charge transfer from the inner tubes to the carbon chains to be strongest for the pristine sample. For example, the signal of the (7.5) inner tubes with d_t = 1.02 nm representing DWCNT filled with linear carbon chains several different species for each sample were measured with an excitation wavelength corresponding to their E_{22} resonance. Figure 3 shows three examples of the numerous PL spectra taken for pristine DWCNT and again the sample annealed at 1400 °C with the biggest change in absorption features and 1460 °C representing DWCNT filled with the longest LCC. The first line scans in Figure 3a show the change in the PL signal of (6.5) tubes with a diameter d_i = 0.76 nm. The intensity of this chirality is amplified greatly for the chain filled samples compared to pristine inner tubes, although, in contrast to the absorption spectra, the chains grown at 1460 °C even give a slightly higher intensity. From the intensity of the side peaks, i.e., tubes that are not in resonance with the incident laser wavelength, this seems to also hold true for the even smaller (6.4) tubes (d_i = 0.69 nm) but not for the bigger (7.5) or (8.4). This deviating behavior becomes apparent when other species are measured at their E_{22} resonances. For example, for the (8.3) inner tubes with d_i = 0.78 nm in Figure 3b the sample annealed at 1400 °C containing more relatively short chains gives the highest intensity enhancement, outperforming their long chain counterpart greatly. Although not in resonance, the same statement can already be made here for the (7.5) and (7.6) tubes.

2.3. PL of Inner Tubes Filled with LCC

When investigating on the PL of inner tubes filled with LCC, several factors have to be considered. Summarizing from our recent reports,[19,20] the interaction of a LCC with the inner tube depends on the following parameters:

- **Inner tube diameter**: For LCCs with similar length, the interaction with the nanotube depends on the distance between chain and nanotube wall, i.e., the nanotube’s diameter.
- **The length of the carbon chain**: The longer the LCC, the stronger the charge transfer from inner tube to LCC.
- **Growth conditions**: The eventual length that the LCC can reach is given by the inner tube diameter (with d_i = 0.71 nm being ideal for the longest chains) and the growth temperature. 1460 °C gives the longest chains, surpassing this temperature destabilizes the chains, as shown earlier.
(diameters $d_t = 0.83$ nm and $d_t = 0.89$ nm, respectively). Furthermore, as depicted in Figure 3c, the $(10,2)$, $d_t = 0.88$ nm, behave similarly. In order to quantitatively analyze the PL response, a line shape analysis for all spectra was performed to evaluate the correct PL signal of the species being in resonance. By doing so, we are able to receive the right correlation between PL enhancement, diameter dependence, and annealing temperature, i.e., growth yield of the LCC.

Measuring in total nine different species for all samples at their respective $E_{22}$ resonance wavelength and comparing their intensity to the pristine sample gives two types of correlations: First, the PL amplification as a function of the inner tube diameter is plotted in Figure 4 for three representative samples. The amplification reaches a maximum for the $(8,3)$ species with a diameter of $0.78$ nm for all samples annealed at temperatures $\leq 1480$ °C (here only $1400$ °C, red curve, and $1460$ °C, green curve, are shown for clarity), for the $1500$ °C (blue) and $1530$ °C sample (not shown) the $(6,5)$ species exhibits a maximum in the amplification. The appearance of a diameter-dependent optimum in the PL amplification is again similar to the PL enhancement of SWCNT filled with ferrocene as observed by Liu et al. (black curve),[18] although, since the ferrocene molecule demands larger tubes to be encapsulated in, this trend can only be observed for tubes with diameters $\geq 0.9$ nm. In the case of ferrocene, the size of the molecule is fixed and the charge transfer to the nanotube depends solely on the tube's diameter. Considering the aforementioned parameters determining the inner tube—LCC interaction[19,20] and the DWCNT’s intrinsic charge transfer from outer tube to inner tube,[7,8] an optimum between chain length, inner tube diameter and inner/outer tube combination must exist so that the influence of the outer tube to the inner tube is ideally compensated. For our DWCNT distribution the optimum is apparently reached for LCC grown at $1400$ °C. The overall decrease in PL amplification when increasing the growth temperature from $1400$ to $1460$ °C (i.e., receiving longer chains) is then caused by an overcompensation of the n-type doping of the inner tube by the outer tube. Consequently, the PL of the inner tubes becomes stronger

[Figure 3. Photoluminescence line scans of different inner tube species. Spectra were normalized according to their optical density and to the selected species being in resonance for pristine DWCNT so that $I_{Pristine} \equiv 1$, $(n,m)$ assignment based on the work of Weisman and Bachilo.[32] PL line scan at a) $569$ nm, the $E_{22}$ resonance of $(6,5)$ tubes, b) $670$ nm, the $E_{22}$ resonance of $(8,3)$ tubes and, c) $742$ nm, the $E_{22}$ resonance of $(10,2)$ tubes.

Figure 4. Amplification of DWCNT filled with carbon chains as function of the inner tube diameter. Growth temperatures: Red $= 1400$ °C, green $= 1460$ °C, and purple $= 1500$ °C. The appearance of a diameter-dependent maximum is similar to the observation of PL amplification found for SWCNT filled with ferrocene.[18] black curve.]
amplication is very low. Therefore, the amplification is hindered due to the lower stability given by the nanotube and sized tubes mostly contain shorter chains. Inside large tubes, LCC formations observed are found in this range, that the charge transfer established by the presence of the LCC reaches an optimum level for chains containing between 20 and 40 carbon atoms. As the chains grow longer when the growth temperature is increased from 1400 up to 1460 °C, the resulting charge transfer exceeds the optimum value and the PL intensity decreases. By surpassing 1460 °C, the chains become unstable and start breaking up, i.e., the charge transfer goes back in the direction of the optimum value. At growth temperatures higher than 1480 °C, this point has already been passed for this type of tubes and the PL intensity decreases monotonically. Furthermore, we can also see an overall decrease in the PL intensity when the diameter is increased from 0.78 to 0.89 nm which can be related to the decreasing interaction energy between chains and inner tubes. 

For inner tubes with a large diameter (d_i > 0.91 nm), the amplification is already very weak. The first maximum is only reached at 1430 °C which is related to the LCC formation process: As the diffusion of the carbon atoms is not restricted to a 1D path any more but small radial movements are also possible, the diffusion length along the nanotubes axis is reduced. Therefore, higher thermal energy is needed. After trespassing 1430 °C the progression follows the PL response of the medium sized tubes.

3. Conclusions

In summary, we present an efficient and simple method to increase the low PL quantum yield of inner tubes from DWCNT. Double-walled carbon nanotubes were filled with differently long linear carbon chains and were subsequently investigated by resonant Raman, optical absorption, and photoluminescence spectroscopy. The optical properties of the inner tubes are altered greatly by the presence of the LCC. Depending on the length of the chain and the inner tube’s diameter, the PL signal is increased up to a factor of 6 for tubes with a diameter close to ≈0.8 nm, while hosting carbon chains containing between 20 and 40 carbon atoms. Concomitant to previous studies, this enhancement can be assigned to a local charge transfer from the inner tube to the carbon chain, balancing out the DWCNTs intrinsic charge transfer from outer tube to inner tube. The method presented here demonstrates a pathway to untether
Characterization by Optical Absorption and Photoluminescence Spectroscopy: Optical absorption spectroscopy was performed using a Bruker VERTEX 80v Fourier transform spectrometer with the resolution set to 2 cm\(^{-1}\). For the PL measurements, different external lasers were coupled into a NanoLog spectrometer (Horiba Jobin Yvon): A tunable dye laser equipped either with Rhodamine 6G (565 nm < \(\lambda\) < 605 nm) or DCM (640 nm < \(\lambda\) < 680 nm), or a tunable titanium sapphire laser (\(\lambda\) > 695 nm). All measurements were done at room temperature. Normalization of the different suspensions with respect to their optical density was achieved by taking the minimum between the first and second optical transition of the inner tubes.[14,18]

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