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## Structural properties of Double Wall Carbon Nanotubes as revealed by TEM

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Single wall carbon nanotubes (SWCNTs) and 2D materials are new classes of materials, which can be considered as isolated nano-objects or can be assembled together to get multifunctional heterostructures. A key question is how components of such hybrid structures interact together, whether their properties are coupled or not and the possible impact on their respective properties. In line with this, SWCNTs have exhibited, since their discovery [1], extraordinary properties but also an extreme sensitivity to any slight fluctuation in their environment. This difficulty could be solved by employing double wall nanotubes (DWCNT), where the outer layer is assigned to play a protecting role with respect to the inner layer. However, the interaction between the layers requires to be known in detail. To that respect, DWCNT represent the ideal and simplest case to investigate the nature of the interaction between two walls within a multiwall nanotube.

This is the purpose of the study we have undertaken. DWCNT are produced using a CVD technique described in [2] and serve as long standing reference samples in numerous studies. Structural identification of the nanotubes is presented through a robust procedure based on the use of a Cs-corrected JEOL ARM200 operating at 80kV, making possible the recording of HRTEM phase contrast images with an atomic resolution and a statistical analysis based on the inspection of numerous nano-objects.

Figure 1 shows a typical HRTEM image, which displays a complex moiré pattern that arises from the projected view of four rotated hexagonal networks. In order to achieve a fully atomic-resolved structure reconstruction from such an image, we defined a data processing sequence composed of different steps detailed in [3] and summarized in Figure 2. A complete structure identification is provided by the determination of respective diameters and helicity angles of both outer and inner tubes. Diameters are determined from the intensity profiles related to the set of dark/bright fringes lying on each side of the tube images. Helicities are extracted from the numerical fast Fourier transform (FFT) of the image, which is analysed as a diffraction pattern according to the procedure developed in [4]. Final step consists in defining the atomic structures of both inner and outer tubes fitting at the best diameters and helicities and leading to the best matching simulations of both HRTEM images and its FFT. This approach reveals that inner and outer tubes of DWNTs are not randomly oriented, suggesting the existence of a mechanical coupling between the two concentric walls (Figure 3). Furthermore, we frequently observe that the inner tube is locally defective whereas the outer remains perfect. With the support of atomic-scale modeling (DFT calculations), we attribute it to the presence of incommensurate domains which structures depend on the diameters and helicities of both tubes and where inner tubes try to achieve a local stacking orientation to reduce strain effects [3]. Furthermore Monte Carlo (MC) simulations have confirmed that experimentally forbidden DWCNT are unstable and that the instability results in structure modification of the inner layer as experimentally observed [3] (Figure 3).

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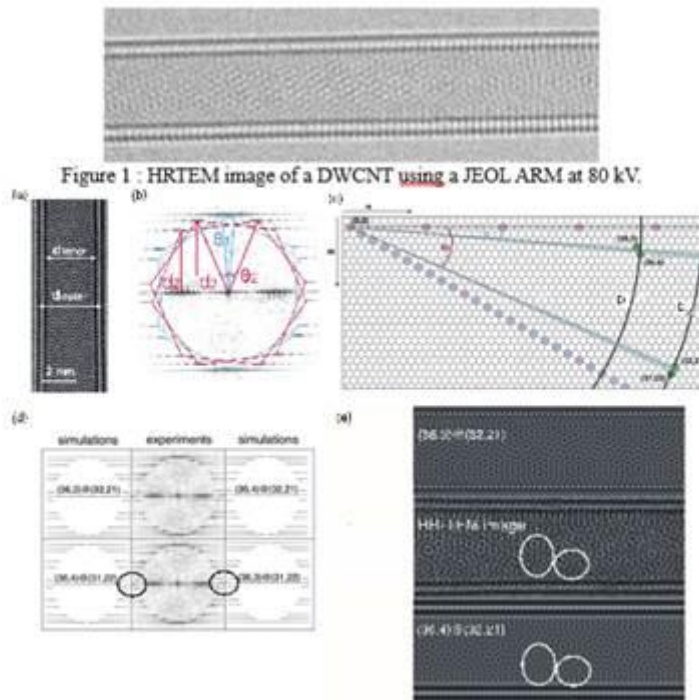


Figure 1 - HRTEM image of a DWCNT using a JEOL ARM at 80 kV.

Figure 2. (a) HRTEM images of a DWNT. (b) Its corresponding FFT. From the measurement of the layer line spacings  $d_2$  and  $d_3$ , chiral angles can be obtained with an error bar of  $\sim 0.5^\circ$ . (c) Distribution of possible chiral indices after the analysis of the layer lines. This leads to four configurations colored in green. (d) Comparison of the FFT from the HRTEM image and simulated results for previous solutions: two configurations can be ruled out because some differences (marked with circles) are noticed.

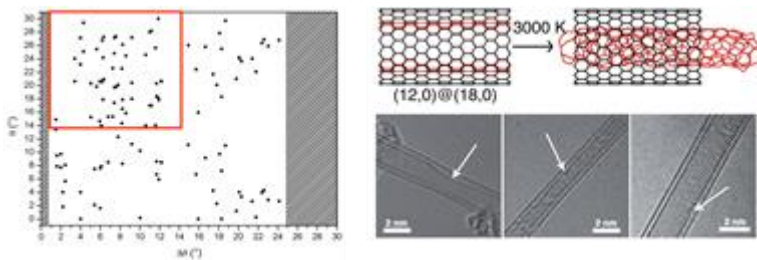


Figure 3 : Left: Statistical analysis plotting outer helicity versus helicity difference between outer and inner tubes: favored (red square) and non observed (dashed areas) configurations are marked . Right top : Relaxed configuration of forbidden DWNT after MC simulation. (c) Defected DWNTs observed experimentally where the inner tube diameters locally decrease (white arrows) as found in MC simulations.