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Structural characterization of single wall carbon nanotubes via AI assisted transmission electron microscopy

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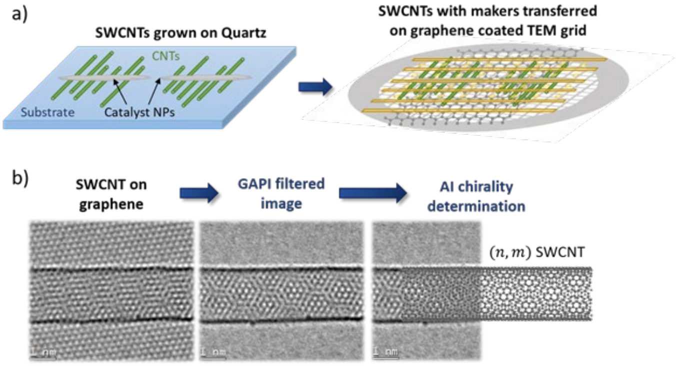
Correlating single wall carbon nanotubes (SWCNT) structural properties with growth conditions during catalytic chemical vapor deposition (C-CVD) is the key to understand the fundamental mechanism involved during this process. However, characterization of individual SWCNTs has always been a challenge, especially under real growth conditions. Recently, homodyne polarization microscopy was able to perform in-situ observations of SWCNT growth inside a CVD reactor [1]. While this technique provides high-throughput growth kinetic measurements, it is still unable to determine the atomic configuration, i.e. chirality, of each individual tube. On the other hand, transmission electron microscopy (TEM) is the technique of choice to directly image SWCNT at the atomic scale and therefore unambiguously determine their chirality [2]. Combining both characterization on the same tubes would reveal significant information about the growth mechanism of CNT. However, TEM sample preparation often relies on random CNT dispersion onto a TEM grid preventing any individual tube identification inside the microscope. In addition, manually determine the chirality for each tube from high-resolution (HR) TEM images is a long process limiting the analysis to a small number of tubes. Here, we aim to develop a new sample preparation protocol to transfer SWCNTs from a substrate to a TEM grid while preserving their location and allowing high quality atomic scale imaging. Furthermore, our goal is also to speed up chirality determination using artificial intelligence (AI) based image analysis.

First, SWCNTs were grown on a quartz substrate inside a custom CVD reactor allowing in-situ optical microscopy imaging. After growth, Au markers were deposited by optical lithography and scanning electron microscopy of the sample was performed to locate tube positions relative to the markers. Before transferring SWCNTs onto a TEM grid, the latter was covered with a graphene monolayer using the PMMA-mediated graphene-transfer method [3]. This layer acts as a mechanical support, preventing tube breaking above grid holes, and facilitating charge evacuation during TEM imaging. Then both SWCNTs and Au markers were transferred onto the graphene coated TEM grid using a similar wet transfer method. Atomic scale images of SWCNT on graphene were then obtained using an aberration corrected TEM equipped with a field emission gun and a monochromator. Finally, fast chirality determination was performed using an open source deep learning code based on convolutional neural networks [4]. This AI code is intended to analyze only SWCNT images; hence, the graphene layer signal must be removed. We developed a new filtering method called Geometric Amplitude and Phase Interpolation (GAPI) based on Fourier transform (FT) filtering that allows us to subtract the graphene signal even when it is superimposed over the SWCNT signal in both real and reciprocal space.

SWCNTs were successfully transferred with the Au markers on the custom TEM grid coated with graphene. Nanotubes, previously observed by in-situ techniques were imaged at high resolution inside the TEM. Both AI and manual methods were used to determine the chirality of the tubes of interest. The strong agreement between the chiral indices measured by both methods highlighted the efficiency of the AI algorithm. In comparison to other FT-based methods, the GAPI filtering method significantly improved AI chirality determination. Our protocol offers the possibility to track down defects and eventual chirality changes induced during growth. However, residual polymer contamination from the transfer processes prevented the analysis of all tubes across their entire length. Nevertheless, we were able to follow single chirality tubes across 10s of µm, providing a direct correlation between their structure and previously measured growth kinetic.

For the first time, the same individual SWCNTs were characterized by both in-situ optical and ex-situ HRTEM observations. The combination of a new image filtering method with automated atomic structure determination using AI, enabled a fast and unambiguous determination of their chirality providing a direct correlation between growth kinetics and tube structures. While the TEM characterization is still limited by polymer contamination, tubes chirality could still be measured in different areas, tens of µm apart.

Graphic:



Keywords:

Carbon nanotubes, HRTEM, artificial intelligence.

Reference:

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