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Single-walled Carbon Nanotube Growth Mechanisms Studied by In situ TEM and Ex-situ Raman Measurements

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Carbon nanotubes (CNTs) are considered as prototypical "new" materials in nanoscience and nanotechnology. Indeed, CNTs provide two particularly exciting prospects. Firstly, the versatility of their properties according to their structure and their dimensions makes them objects of fundamental interest. Secondly, their outstanding mechanical strength, electrical and thermal conductivity and opto-electronic properties offer many opportunities for industrial applications. The combination of these exceptional and versatile properties might lead to the development of electronic systems where both active devices and interconnects are based on the same material. However, in spite of longstanding efforts, no major electronics application involving CNTs is available in the market yet. The main obstacle in the development of a CNT-based technology is that many aspects of their growth mechanisms remain obscure. In particular, the relationship between the nucleating/growing nanotube and the catalyst nanoparticle is not well understood. Despite some remarkable improvements in the control of CNTs features at the synthesis stage during the last decade [1-4], the optimization of CNT growth conditions remain mostly empirical and nanotube samples are frequently a mixture of different structures (number of walls, length, diameter and chiral angle) and morphologies (straight, bundled or entangled). Here, we have employed an in situ approach combining high resolution imaging and Electron Energy Loss Spectroscopy using an environmental scanning transmission electron microscope (ESTEM) in order to probe the relevant instants of a nanotube life at the atomic scale. The composition and the crystal structure of the catalytic nanoparticles were investigated as a function of the growth condition (temperature, pressure) and the nanoparticle size for various catalyst/carbon precursor combinations. The nature of the grown species (number of walls, diameter, crystallinity) were also systematically examined and correlated. Ex situ Raman measurements were used to characterize the in situ synthesized samples at a large scale. [1]. Harutyunyan AR, Chen G, Paronyan TM, Pigos EM, Kuznetsov OA, Hewaparakrama K, et al. Preferential growth of single-walled carbon nanotubes with metallic conductivity. Science. 2009;326(5949):116. [2]. Chiang W, Sankaran R. Linking catalyst composition to chirality distributions of as-grown single-walled carbon nanotubes by tuning Ni_xFe_{1-x} nanoparticles. Nat Mater. 2009;8(11):882-6. [3]. Bachilo S, Balzano L, Herrera J, Pompeo F, Resasco D, Weisman R. Narrow (n, m) Distribution of Single-Walled Carbon Nanotubes Grown Using a Solid Supported Catalyst. J Am Chem Soc. 2003;125(37):11186-7. [4]. Zheng L, O'connell M, Doorn S, Liao X, Zhao Y, Akhadov E, et al. Ultralong single-wall carbon nanotubes. Nat Mater. 2004;3(10):673-6.