One-step CVD synthesis of nanometric carbon rings and junctions

Néstor Perea-López¹, Emilio Muñoz-Sandoval¹, Ana L. Elías-Arriaga¹, Humberto Terrones¹, Benji Maruyama² and Mauricio Terrones¹

 ¹ Laboratory for Nanoscience and Nanotechnology Research (LINAN) and Advanced Materials Department, IPICYT, Camino a la Presa San Jose 2055, Lomas 4a. Seccion, San Luis Potosi 78216, Mexico.
² Air Force Research Laboratory, Materials and Manufacturing Directorate, AFRL/RX, Wright-Patterson Air Force Base, Ohio 45433, USA

Abstract

Carbon nanotube architectures like rings, Y and X junctions were obtained from a one-step catalytic chemical vapor deposition (CVD) process. The catalyst consisted of cobalt nanoparticles (NPs) deposited on $AlO_x/Si(100)$ by DC magnetron sputtering in a multiple target system. First, The AlO_x was fabricated by depositing a 20 nm thick Al thin film on Si and then it was oxidized inside the deposition chamber by increasing the substrate temperature to 500 °C and introducing oxygen at 200 mTorr for 30 min. Secondly, after the AlO_x layer was formed and cooled to room temperature, the NPs were deposited from a cobalt target with 2 and 4 nm thicknesses to obtain samples with different NPs density.

The nanorings and junctions were formed by thermolizing ethanol on the catalyst substrates placed in a 1 inch diameter tubular furnace CVD set-up. The ethanol was vaporized with an ultrasonic nebulizer and Ar+H₂ gas mixture was used to drag the micro droplets into the furnaces at 2.5 l/min flow rate, the reaction temperature was 950 °C during the 30 minutes CVD growth process. However, shorter CVD periods (1, 5 and 10 minutes) were made in order to explore the initial steps of formation and the growth mechanism. The catalyst substrates morphology was studied by atomic force microscopy and the CVD grown nanostructures were studied by scanning transmission, scanning electron microscopies (STEM and SEM) and high resolution TEM.

From the microscopy observations, we found that the substrates made to study the initial formation steps contained low surface density of multiwalled carbon nanotubes (MWCNTs), ~10 nm in diameter and tens of microns length. Nevertheless, the final nanostructures, it is the rings and junctions, are made of very thick CO_x MWCNTs, ~200 nm diameter. The proposed mechanism of formation is related to a thickening of the initial MWCNTs, which could have coiled or formed networks, and when the walls of those structures thicken, the initial MWCNTs are fully covered by thick CO_x layers. In the network scenario the thickening by incoming carbon species welds the MWCNTs to each other creating a junction. On the other hand, in the case of a coil the threads stick to each other creating a single loop. The phenomenon described above is possible considering that the NPs density is low and the active catalyst particles are used in the initial moments of the CVD to form MWCNTs, after that there are no more catalytic centers, hence the incoming species of pyrolized ethanol and H₂ only have the MWCNT walls to stick to.

This work was supported in part by CONACYT-Mexico Grant: 56787 (Laboratory for Nanoscience and Nanotechnology Research-LINAN) and the US-Air Force, grant FA9550-08-1-0204.