Iron nanoparticles deposited on ozone pre-treated carbon nanotubes by Fluidized Bed Metal Organic Chemical Vapor Deposition

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Abstract

Composite materials are used in the structure of planes to make them lighter, greener and more economical in fuel consumption. The aeronautic field aims now to reduce the mass of on-board electronic equipment packaging, which requires developing innovative composite materials able to evacuate thermal and electric charges while keeping appropriate mechanical features. Due to their unique physical properties, metal-coated carbon nanotubes combined with a polymer matrix represent a promising solution to face this challenge.

The present work aims to uniformly deposit iron nanoparticles on the surface of Graphistrength® multi-walled carbon nanotubes (MWCNTs) tangled in balls of 400 µm in mean diameter, by Fluidized Bed Metal Organic Chemical Vapor Deposition (FB-MOCVD). A dry-mode pre-treatment by ozone and a mixture of ozone/water vapor in fluidized bed is first applied, in order to increase the surface reactivity of the MWCNTs. Three atmospheres are studied during iron deposition from ferrocene ($FeC_{10}H_{10}$), involving nitrogen, hydrogen or water vapor, in order to exalt pure iron deposit. The obtained O_3 or O_3/H_2O pre-treated MWCNTs and Fe-MWCNTs are analyzed by High Resolution Transmission Electron Microscopy (HRTEM), Thermo-Gravimetric Analysis (TGA), X-Ray Diffraction (XRD), and Scanning Electron Microscopy equipped with a Field Emission Gun and an Energy Dispersive X-ray detector (FEG-SEM-EDX).

The O_3/H_2O pre-treatment applied between 1 h and 20 h has allowed grafting oxygen containing groups (mainly hydroxyl and carboxyl bonds) on the nanotube surface much more efficiently than using O_3 alone. The amount of damaged and even opened MWCNT walls (Fig. 1) increases with the treatment duration.

Whatever the atmospheres tested, the deposit from ferrocene occurs under the form of Fe_3C/Fe nanoparticles. Except in presence of hydrogen, the iron based nanoparticles are able to catalyze the formation of nanofibers or of large nanotubes. The pre-treatment increases the nucleation of iron-based nanoparticles (Fig. 2b) in comparison with non-treated MWCNTs (Fig. 2a) and then the surface reactivity of MWCNTs towards ferrocene and its gaseous products of decomposition.

In comparison with the wet methods, a main advantage of dry mode pre-treatments in fluidized bed and of the fluidized bed CVD process is that they can treat large amounts of carbon nanotubes, i.e. 100 g at the lab-scale in our case and tons at the industrial scale, opening the way for a mass production of decorated MWCNTs for applications like nano-fillers of innovative multi-functional composite materials.

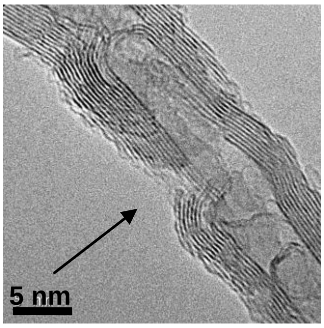

Figure 1: HRTEM view of a MWCNT walls after 12 h of O_3/H_2O pre-treatment

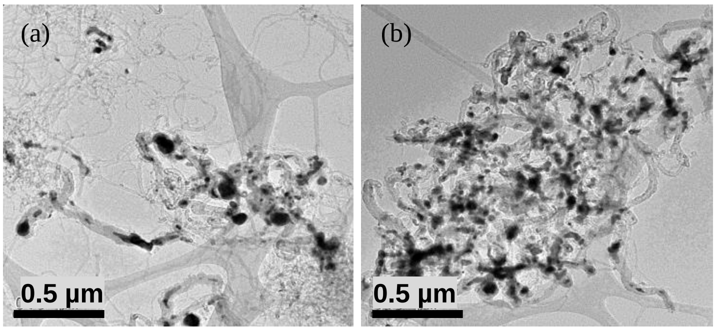

Figure 2: TEM views of MWCNTs after iron deposition (a) without and (b) with pre-treatment