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## Physical and mechanical properties of thick self-standing layers of multiwall carbon nanotubes

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## Abstract

Massive samples of self-standing, well packed, vertically aligned and very long multiwall carbon nanotubes (average diameter 50 nm; length exceeding 3 mm) were synthesized on uncoated silicon by a very efficient thermal chemical vapor deposition (CVD) process. The deposition involves the co-evaporation of camphor and ferrocene in a nitrogen atmosphere and subsequently the pyrolysis of the precursors at 850 °C. The average growth rate was 500 nm/s (1.8 mm/h), with a conversion of approximately 30% in weight of the total hydrocarbon feedstock.

The morphology and physical properties of the nanotube forest were characterized by electron microscopy and spectroscopy techniques, thermo-gravimetric analysis and mechanical tests. The analysis showed the presence of negligible amount of carbonaceous by-products and about 6% in weight of ferromagnetic iron clusters. Moreover, the as-grown carpet showed good thermal stability, very interesting mechanical properties, such as elasticity and resistance under compression, and was fully hydrophobic. © 2006 Elsevier B.V. All rights reserved.

Keywords: Multiwall carbon nanotubes; CVD; Hydrophobicity; Mechanical properties

## 1. Introduction

Carbon nanotubes have attracted an enormous interest in recent years because of their outstanding properties and the various applications that have been proposed [1]. Lightweight, high-strength application have been proposed as well [2] but a growth method that, at the same time, is simple, economical and that has a high conversion/deposition rate, for potential future industrial applications has yet to be developed [3–5]. Chemical vapor deposition (CVD) methods have attracted much attention because of their advantages in the light of these requirements, but a real breakthrough has not yet occurred.

In this work, we report about a CVD system that allows a rapid growth of self-standing large area millimeters-thick carpets of multiwall carbon nanotubes (MWNTs) by catalytic decomposition of a ferrocene–camphor mixture. This process is simple, economic, very effective, versatile (e.g. varying growth parameters lead to the achievement of different kind of carbon materials, such as carbon fibers and nanographite) and can be easily scaled up and used for continuous or semi-continuous production [6,7]. The growth rate reached 500 nm/s and the mass production rate exceeds 5 g/h, with a conversion rate of reagents over 30% in weight; carpet thickness can reach 3 mm in 90 min.

The availability of self-standing large area blocks of carbon nanotubes allowed us to perform a series of macroscopic investigations of the mechanical and physical properties of the material, avoiding the drawbacks suffered by recent works in which measurements are on the microscopic scale and give little information on what can be expected for large-scale systems [8].

Moreover, MWNT carpet showed high purity, excellent mechanical properties and impressive hydrophobic behavior previously reported for functionalized non-self-standing nanotubes only [9].

## 2. Experimental

The thick layers of MWNTs were grown in a CVD reactor by a method similar to that reported in Refs. [6] and [10], with some improvements in process parameters in order to reach the best growth conditions.

The vapor mixture of camphor (carbon precursor) and ferrocene (catalyst precursor) was carried by a nitrogen gas flow to the deposition region, where at a substrate temperature of

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