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Macroscopic growth of carbon nanotube mats and their mechanical properties

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Since carbon nanotubes have been recognized as a ultrastrong material, both by simulations and experimental measurements [1], various applications have been proposed for their use in thermal and electrical enhancement [2,3] and in the area of strength and toughness as reinforcement for composite materials [4]. Nevertheless, significant progresses in developing carbon nanotube-composites technology are limited by the lack of a simple, economical and high conversion/deposition rate production method for carbon nanotubes. Because of the several advantages of the CVD techniques [5,6], much efforts have been devoted to the optimization of this method, though a real breakthrough in this field has yet to be achieved. Here we report the growth and the properties of self standing large area millimeters-thick sheets of as-deposited multiwall carbon nanotubes (MWCNTs). Such sheets were grown by catalytical chemical vapor deposition (CVD) at a growth rate up to 0.5 $\mu m\ s^{-1}$ over an area limited only by the deposition system size (up to 100 cm^2 at present). With this method curved surfaces can be straightforwardly coated. The sheets showed good thermal stability and interesting mechanical properties such as elasticity and resistance under compression and were totally hydrophobic. These results show that the route towards the use of carbon nanotubes as a structural material is now open.

The thick layers (over 2 mm) of MWCNTs, termed "mats" from here on, were grown in a CVD reactor formed by a horizontal quartz tube housed in a cylindrical furnace in which a constant nitrogen gas flow rate was maintained at pressures just above atmospheric [7]. The carbon (camphor) and the catalyst (ferrocene) sources were mixed in the ratio 20:1 in a pyrex flask. The precursors solid mixture, which contained no solvents, was melted and evaporated directly from the flask by heating it at 220 °C with a heater plate. The flask connection between the quartz tube and the nitrogen gas source was realized with a T-joint. The gas evaporated from the flask was injected into the nitrogen flux, that carried it to the substrate region. Deposition of the MWCNTs took place on non pre-etched bare silicon substrates. The temperature in the deposition region was kept at 850 °C. The reactor quartz tube internal diameter was 4.2 cm, and the length of the uniform temperature region of about 30 cm. The size of the substrate housed in such a tube was therefore 4×30 cm (substrate divided in two pieces 4×15 each, due to the size of commercial silicon wafers). The temperature gradient present at the edges of the uniform temperature region caused small gas turbulences reducing the uniform deposition area to 4×25 cm (100 cm^2). As the current version of the system does not allow a continuous feed of the reagents, the process time lasted two hours at most. At the end of the process the substrates (up to 100 cm^2 in total) were covered by a few millimeters thick uniform MWCNT mat. A consistent deposition of MWCNTs occurred also on every curved surface of the silicon substrate (see Fig. 1b) and on the inner surface of the quartz tube, although on the latter the deposition is in the form of non ordered compact and irregular tangles. The deposition rate reached 0.5 $\mu m\ s^{-1}$ and the mass production rate (considering only the mats grown on Si substrate) exceeded 1 $g\ h^{-1}$, while the total

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