HARVESTING OF PROCESSED CARBON NANOTUBES

CROSS-REFERENCE TO RELATED PATENT APPLICATIONS

[0001] This application claims the benefit of priority of provisional application 60/973,287 filed in the U.S. Patent & Trademark Office on Sep. 18, 2007, the disclosure of which is incorporated herein by reference.

ORIGIN OF THE INVENTION

[0002] The invention described herein was made by both employees of the United States Government and United States Government Contractors. The United States Government has an ownership interest in this invention.

FIELD OF THE INVENTION

[0003] The present invention relates to harvesting processed carbon nanotubes, and more particularly processes that involve the descaling, debundling, catalyst removal and/or size-sorting of carbon nanotubes.

BACKGROUND OF THE INVENTION

[0004] Carbon nanotubes (CNT) are nanoscale structures that have attracted much interest throughout various technologies for their exceptional electrical, magnetic, mechanical, and thermal properties. CNTs are particularly strong materials with high tensile strength and elastic modulus. Aligned CNTs are good thermal conductors along their axial length and good insulators lateral to their axes.

[0005] Given the unique combination of properties possessed by CNTs, there have been an ever increasing number of potential applications for CNTs. Electronic, optic and optoelectronic applications include, for example, field emission devices (FEDs), composites for electromagnetic interference shielding and electrostatic discharge protection, additives for battery and supercapacitor electrodes, and fuel cell electrodes.

[0006] Generally, a CNT is a hexagonal network or lattice of carbon atoms that appears as an elongated sheet rolled into an elongated, seamless cylinder. The cylinder may have a diameter, for example, on the order of a few nanometers, and a length up to several micrometers. The cylinder may consist of a single wall or multiple concentric walls or shells tightly packed with one another and attracted to one another by van der Waals forces. These CNTs are respectively referred to as single-wall CNTs (or SWCNTs) and multi-wall CNTs (or MWCNTs).

[0007] There are a variety of different methods for fabricating CNTs. A common feature of these methods is the use of a transition metal-based nanoparticle that serves as a catalyst or seed for the growth of the carbon cylindrical body. The nanoparticle seeds are exposed to gaseous carbon or a hydrocarbon source which deposits carbon on the particle surface and builds upon itself to generate the carbon tubular body. The particular technique employed for generating and depositing the carbon varies. Known techniques include chemical vapor deposition, pulsed laser vaporization, radio frequency plasma, and electric arc discharge. The characterization of the carbon deposition as either a mono- or multi-walled structure will depend largely on the size of the transition metal-based nanoparticle. Larger nanoparticles tend to favor MWCNTs. It should be understood that the present invention is not limited to, and may be practiced in combination with, a variety of these synthesis techniques and nanostructures.

[0008] Conventional CNT synthesis techniques as described above do not produce discrete, catalyst-free tubes. Rather, the resulting nanostructure usually is a bundle of tubes intertwined together and connected to their respective metal catalysts. Further, amorphous carbon shells coat the outside of the tubes. Many of the commercial and potential applications discussed above require that the carbon nanotubes be cleaned of their amorphous carbon shell, debundled from other intertwined tubes, separated from their metallic catalysts and/or shortened. The separation of the tubes from their metallic catalysts and the removal of the amorphous carbon, in particular on large scales, are significant barriers to harvesting a relatively pure population of CNTs suitable for large scale applications.

[0009] The current state of the art for purifying CNTs is to use combinations of organic dispersants, high power ultra sonication, strong mineral acids, and/or oxidizers to treat the CNTS. These purification processes can destroy and/or cause excessive damage to the CNTS. In particular, it has been estimated using these state-of-the-art techniques to debundle, purify and shorten CNTs may damage or destroy up to 90 percent of the CNTs, In this regard, chiral semi-conducting CNTs and/or shortened tubes (<500 nm long) are particularly sensitive. High-power sonification, for example, has been found to blow-out the side walls of the CNTS. Strong acids (e.g., nitric and sulfuric acids), oxidizers (e.g., hydrogen peroxide), and organic solvents (e.g., tetrahydrofuran, methyl ethyl ketone, and methyl chloride) produce significant harmful waste, raise worker safety and toxicity concerns, and in some instances have been found to chemically alter the CNTs. An alternative mechanical-based approach that applies a "ball-milling" process to debundle and shorten CNTs has also been reported but has not to date been successfully implemented as a practical, scalable method. This is because ball milling has been found to be one of the most destructive methods for processing CNTs. Because of the above-described problems, established purifying methodologies are not scalable to high industrial rates.

[0010] Even after the CNTs have been debundled and debulked to extricate the individual tubes from their intertwined state, the CNTs must be separated from their catalysts and amorphous carbon before being suitable for use in many commercial and theoretical applications. Current state of the art separation methods include high-speed centrifugation, filtration (paper, glass, filter, membrane, gel), and chromatography (column and HPLC). Common problems plaguing these state-of-the-art techniques are limited throughput capability and scalability due to CNT aggregation and filter clogging.

[0011] In view of the increasing demand for the large-scale production of processed CNTs so as to satisfy the various potential applications of CNTs, there is a need in the art for a process for harvesting CNTs in a manner that much more robust and scalable while being much less destructive to the CNTs than the above-discussed known techniques.

SUMMARY OF THE INVENTION

[0012] In accordance with the purposes of the invention as embodied and broadly described herein, a first aspect of the invention provides a method of harvesting carbon nanotubes (CNTs). According to this method, CNT bundles, comprising CNTs associated with metallic catalysts and having amor-