



## Improved Method of Purifying Carbon Nanotubes

Unwanted material is removed without damaging the nanotubes.

Ames Research Center, Moffett Field, California

An improved method of removing the residues of fabrication from carbon nanotubes has been invented. These residues comprise amorphous carbon and metal particles that are produced during the growth process. Prior methods of removing the residues include a variety of processes that involved the use of halogens, oxygen, or air in both thermal and plasma processes. Each of the prior methods entails one or more disadvantages, including non-selectivity (removal or damage of nanotubes in addition to removal of the residues), the need to dispose of toxic wastes, and/or processing times as long as 24 hours or

more. In contrast, the process described here does not include the use of toxic chemicals, the generation of toxic wastes, causes little or no damage to the carbon nanotubes, and involves processing times of less than 1 hour.

In the improved method, purification is accomplished by flowing water vapor through the reaction chamber at elevated temperatures and ambient pressures. The impurities are converted to gaseous waste products by the selective hydrogenation and hydroxylation by the water in a reaction chamber. This process could be performed either immediately after growth or in a post-

growth purification process. The water used needs to be substantially free of oxygen and can be obtained by a repeated freeze-pump-thaw process. The presence of oxygen will non-selectively attach the carbon nanotubes in addition to the amorphous carbon.

*This work was done by Lance D. Delzeit of Ames Research Center and Clement Delzeit. Further information is contained in a TSP (see page 1).*

*Inquiries concerning rights for the commercial use of this invention should be addressed to the Patent Counsel, Ames Research Center, (650) 604-5104. Refer to ARC-14733.*

## Patterned Growth of Carbon Nanotubes or Nanofibers

Numerous parameters of deposition conditions, structures, and compositions affect what is grown.

Ames Research Center, Moffett Field, California

A method and apparatus for the growth of carbon nanotubes or nanofibers in a desired pattern has been invented. The essence of the method is to grow the nanotubes or nanofibers by chemical vapor deposition (CVD) onto a patterned catalyst supported by a substrate.

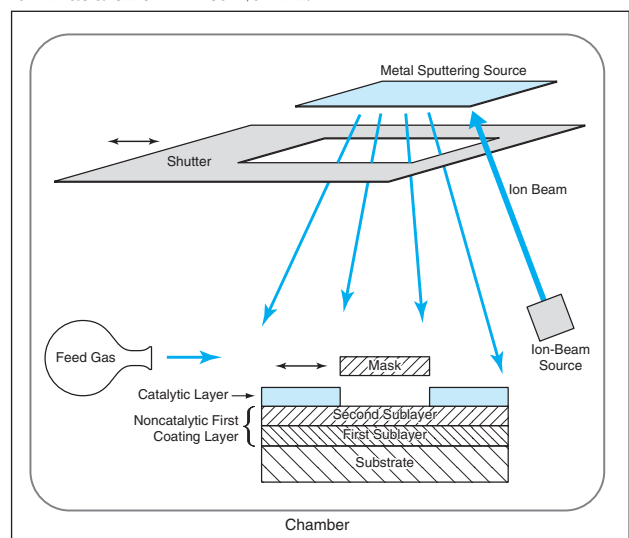
The figure schematically depicts salient aspects of the method and apparatus in a typical application. A substrate is placed in a chamber that contains both ion-beam sputtering and CVD equipment. The substrate can be made of any of a variety of materials that include several forms of silicon or carbon, and selected polymers, metals, ceramics, and even some natural minerals and similar materials. Optionally, the substrate is first coated with a non-catalytic metal layer (which could be a single layer or could comprise multiple different sublayers) by ion-beam sputtering. The choice of metal(s) and thickness(es) of the first layer (if any) and its sublayers (if any) depends on the chemical and electrical properties required for subsequent deposition of

the catalyst and the subsequent CVD of the carbon nanotubes.

A typical first-sublayer metal is Pt, Pd, Cr, Mo, Ti, W, or an alloy of two or more of these elements. A typical metal for the second sublayer or for an undivided first layer is Al at a thickness  $\geq 1$  nm or Ir at a thickness  $\geq 5$  nm. Proper choice of the metal for a second sublayer of a first layer makes it possible to use a catalyst that is chemically incompatible with the substrate.

In the next step, a mask having holes in the desired pattern is placed over the coated substrate. The catalyst is then deposited on the coated substrate by ion-beam sputtering through the mask. Optionally, the catalyst could be deposited by a technique other than sputtering and/or pat-

terned by use of photolithography, electron-beam lithography, or another suitable technique. The catalytic metal can be Fe, Co, Ni, or an alloy of two or more of these elements, deposited to a typical thickness in the range from 0.1 to 20 nm.



This Schematic Diagram represents a composite of the apparatus as configured for the catalyst-patterning and CVD steps of the method.

Following deposition of the patterned catalyst, a shutter is moved into place to protect the sputtering equipment against CVD of carbon, then a hydrocarbon feed gas (primarily CH<sub>4</sub>, C<sub>2</sub>H<sub>2</sub>, or C<sub>2</sub>H<sub>4</sub>) heated to a suitable temperature is admitted into the chamber. Optionally, the feed gas can be part of a mixture that includes an inert carrier gas. The heated feed gas decomposes into hydrogen and carbon, with deposition of the carbon on the cata-

lyst. Whether what are grown are single-walled nanotubes (SWNTs), multi-walled nanotubes (MWNTs), or nanofibers depends primarily on the feed gas and temperature used in CVD and secondarily on the structures and compositions of the first layer and the patterned catalytic second layer. For example, for growing SWNTs, the preferred gas is CH<sub>4</sub> and the preferred temperature is ≈900 °C. For growing MWNTs, the preferred gas is C<sub>2</sub>H<sub>2</sub> or

C<sub>2</sub>H<sub>4</sub>, the preferred temperature is ≈750 °C. For growing nanofibers, it is preferable to form a plasma discharge in the chamber and to maintain the temperature between 400 and 700 °C.

*This work was done by Lance D. Delzeit of Ames Research Center. Further information is contained in a TSP (see page 1).*

*Inquiries concerning rights for the commercial use of this invention should be addressed to the Patent Counsel, Ames Research Center, (650) 604-5104. Refer to ARC-14613.*

---

## **Lightweight, Rack-Mountable Composite Cold Plate/Shelves**

*Lyndon B. Johnson Space Center, Houston, Texas*

Rack-mountable composite-material structural components that would serve as both shelves and cold plates for removing heat from electronic or other equipment mounted on the shelves have been proposed as lightweight alternatives to all-metal cold plate/shelves now in use. A proposed cold plate/shelf would include a highly thermally conductive face sheet containing oriented graphite fibers bonded to an aluminum honeycomb core, plus an extruded stainless-steel substructure containing optimized flow passages for a cooling fluid, and an inlet and outlet that could be

connected to standard manifold sections. To maximize heat-transfer efficiency, the extruded stainless-steel substructure would be connected directly to the face sheet. On the basis of a tentative design, the proposed composite cold plate/shelf would weigh about 38 percent less than does an all-aluminum cold plate in use or planned for use in some spacecraft and possibly aircraft. Although weight is a primary consideration, the tentative design offers the additional benefit of reduction of thickness to half that of the all-aluminum version.

*This work was done by Kathryn M. Hurl-*

*bert of Johnson Space Center and Warren Ruennele, Hai D. Nguyen, Kambiz K. Andish, and Sean McCalley of Lockheed Martin.*

*Title to this invention has been waived under the provisions of the National Aeronautics and Space Act {42 U.S.C. 2457(f)}, to Lockheed Martin Space Operations. Inquiries concerning licenses for its commercial development should be addressed to:*

*Lockheed Martin Space Operations*

*2400 NASA Road 1*

*Nassau Bay, TX 77058*

*Refer to MSC-23160, volume and number of this NASA Tech Briefs issue, and the page number.*