CARBON MATERIALS

PROJECT FACTS

UNIVERSITY OF KENTUCKY
CENTER FOR APPLIED ENERGY RESEARCH

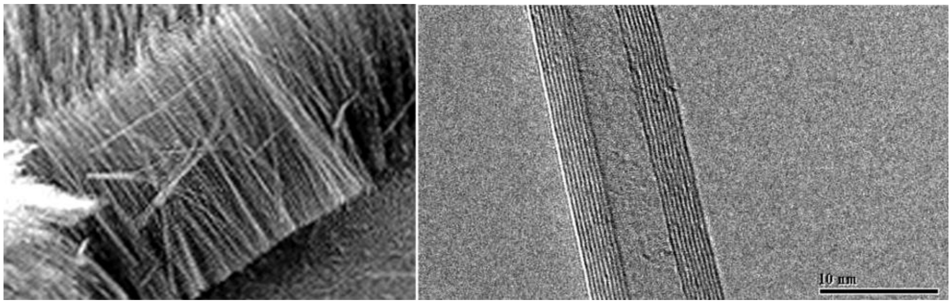
Synthesis of Multiwall Carbon Nanotubes

The remarkable properties of carbon nanotubes (CNT) have prompted intense research into a wide range of applications where their deployment could have a major impact. Their electronic properties are already being utilized in field emission applications, but it is their extraordinary mechanical properties (tensile strength and modulus) that have aroused particular interest. However, to satisfy demands for these materials, high production volumes of pure nanotubes are required. At CAER interest has focused on the synthesis of multiwall carbon nanotubes (MWNTs) by a chemical vapor deposition (CVD) process designed to produce high purity aligned nanotubes (unentangled) in bulk and at low cost. This route also benefits from its ability for scale-up to commercialization and avoids the problems associated with alternative methods, which often give low yields, poor alignment and lack the required purity. The method also obviates the need for purification which is often difficult, lowers the yield further, and is damaging to tube structure through oxidative shortening.

A synthesis process has been developed that is relatively simple, consisting of a quartz tube reactor within a multi-zone furnace into which a mixture of xylene and ferrocene is injected in a carrier gas. Decomposition of the ferrocene at temperatures in the range 500-900 °C, and at atmospheric pressure, deposits iron nanoparticles on the quartz surfaces (reactor walls and substrates). The catalytic activity of the metal promotes decomposition of volatile carbon compounds to form metastable carbides, allowing carbon to diffuse through and over the metal nanoparticles extremely rapidly, where the ordered carbon is precipitated. As a result of this growth mechanism, dense mats of aligned MWNTs are formed. To prevent the formation of amorphous carbon, the reaction is restricted to the catalyst surface through the choice of the carbon precursor, its partial pressure, and the reaction temperature. Reaction temperature is also used to control catalyst particle size and hence nanotube diameter. The larger diameter nanotubes retain the fine structure of concentric graphene cylinders, consisting of hundreds of shells, and do not transform to one of the less well-ordered carbon structures (herring-bone or platelet type nanofibers). Metal catalyst is often found at the tip of the nanotubes and also within the core in tests where high metal to carbon ratios are used. However, the root ends are usually open.

The process, initially developed as a batch system, has been scaled three times and now yields ~4g in a 3hour run. Further development is ongoing with the operation of a continuous system capable of producing ~3kg of high purity, aligned nanotubes per day.

Nanotube synthesis by CVD has many advantages over other production routes, not least among which is the high purity of the product containing virtually no amorphous carbon. However, the temperature used in the synthesis, tends to produce nanotubes with less than perfect graphene structure. This has been rectified by graphitization (1800 to 2800 °C) which anneals the structural defects and simultaneously removes the residual iron catalyst.



SEM and TEM of MWNT Product

CONTACT

David N. Jacques
UK CAER
2540 Research Park Dr
Lexington, KY 40511
Tel: (859) 257-0310
Fax: (859) 257-0220
jacquesd@caer.uky.edu

Rodney Andrews
UK CAER
2540 Research Park Dr
Lexington, KY 40511
Tel: (859) 257-0305
andrews@caer.uky.edu
Updated 7/06/04