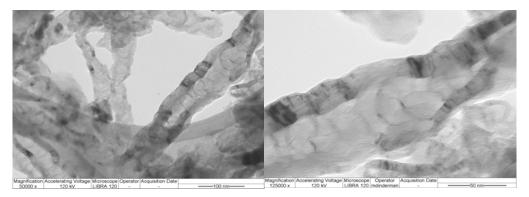
NNI Scientific Accomplishments 2009

Formation of Carbon Nanotubes in Bulk Carbonaceous Solid

The NRL has developed a unique method for the synthesis and formation of multi-walled carbon nanotubes (MWNTs) and carbon nanofibers (CNFs) in shaped, porous carbonaceous solids based on the thermal decomposition of melt-processible organometallic compounds or metal salts in the presence of an excess amount of an aromatic carbon precursor. Metal nanoparticles (any specific metal or combination thereof) can be incorporated in varying amounts into the MWNT/CNF carbonaceous solid. The principal advantage of this novel synthetic approach, relative to conventional MWNT synthesis by chemical vapor deposition (CVD), is that carbonaceous compositions can be achieved in bulk solid, moldable shapes, using relatively lowcost compounds/resins/polymers and processing equipment, thereby reducing economic barriers and production limitations that are inherent with CVD. Our highly flexible synthetic method also offers the ability to incorporate heteroatoms (N, Si, B) into the solid via the initial carbon precursors. A purification procedure (calcination) has been developed to selectively burn out the amorphous phase within the carbonaceous solid to produce a highly porous material. The surface area and porosity of the purified solid can be controlled as a function of the thermal treatment. The figure below shows bamboo-tubular structure of a Fe nanoparticle-containing nanostructured carbonaceous solid produced from pyrolysis of a 1:20 molar mixture of Fe₂(CO)₉ and a novolac epoxy resin. The nanostructured materials have been integrated into battery, superconductivity, air filtration/separation, and nanoelectronic funded programs.



TEM images of a novolac/Fe₂(CO)₉ system showing bamboo morphology

Reference: (1) T. M. Keller, M. Laskoski, M. Osofsky, and Syed B. Qadri, "Carbon Nanotube Formation Catalyzed by Ni Nanoparticles in Carbonaceous Solid", *Phys. Stat. Sol.* (*A*) **205**, 1585 (2008); (2) M. Laskoski, T. M. Keller, and S. B. Qadri, "Direct Conversion of Highly Aromatic Phthalonitrile Thermosetting Resins into Carbon Nanotube Containing Solids", *POLYMER* **48**, 7484 (2007); (3) T. M. Keller, M. Laskoski and S. B. Qadri, "Ferrocene Catalyzed Carbon Nanotube Formation in Carbonaceous Solid", *J. Phys. Chem. C* **111**, 2514-2519 (2007); and (4) M. K. Kolel-Veetil, S. B. Qadri, M. Osofsky, T. M. Keller, R. Goswami, and S. A. Wolf, "Size-Induced Effects on the Superconducting Properties of Mo₂C Nanoparticle", *J. Phys. Chem. C* **111**, 16878 (2007).

Basic 6.1 research: NRL/ONR, DTRA funded program – air filtration/separation; DARPA funded program – use of novel carbon nanotube materials in nanodevices.

Contributing Agency: DoD / NRL