New insights in the synthesis and applications of carbon nanotube sea urchins

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The outstanding thermal, electrical, and mechanical properties of individual carbon nanotubes (CNTs) have been studied extensively for more than two decades. However, transferring these individual properties to macroscopic devices composed of a large number of CNTs remains a challenge, that many consider a key to the development of the next generation of nanotech products.

Carbon nanotube sea urchins (CNTSUs) are unique nanostructures, whose aerosol synthesis was pioneered by (Kim, Wang, & Zachariah, 2010). They consist of a spherical nanoparticulate core, from which CNTs are grown radially, as shown in Figure 1. As hundreds of CNTs are linked via this central focus, the issues associated with poor control of the CNT-CNT interfaces are mitigated, potentially enabling better retention of the individual CNT properties at the macroscale as compared to a bulk disordered CNT material. Moreover, the CNTSU core can be used to include additional physical or chemical functionalities in the resulting material.



Figure 1. Scanning electron microscope image of a carbon nanotube sea urchin.

In this research CNTSUs are synthesised via a continuous, industrially scalable aerosol process. An aqueous solution of aluminium and iron nitrates is nebulised in a flow of nitrogen gas. As the aerosolised droplets evaporate in a diffusion drier, the solute precipitates, forming bimetallic salt particles. These particles are then calcinated in a reducing environment - a tube furnace with hydrogen addition - so that the nitrates decompose, producing bimetallic Al-Fe nanoparticles. The addition of carbon in the form of acetylene in a second tube furnace enables the

subsequent growth of CNTs from iron catalytic sites at the surface of these cores, thereby forming CNTSUs.

We will present new characterisation of the CNTSUs at different stages of the process, which give a new insight on the physical mechanisms leading to the formation of iron catalytic sites at the surface of the cores. These characterisation methods include X-ray diffraction (XRD), X-ray photoemission spectroscopy (XPS), Raman spectroscopy, energy dispersive X-ray spectroscopy (EDX), transmission electron microscopy scanning electron microscopy (TEM), (SEM), thermogravimetric analysis (TGA), scanning mobility particle sizer (SMPS), and centrifugal particle mass analyser (CPMA). An example of XPS characterisation showing the presence of both Fe^{2+} and Fe^{3+} at the surface of the CNTSU cores is given on Figure 2.



Figure 2. XPS characterisation of the Fe 2p orbitals at the surface of the CNTSU cores.

Moreover, films of CNTSUs were deposited onto various substrates, either by direct thermophoretic deposition or by filtration followed by blade casting, and a range of physical and chemical properties of these films were tested, paving the way for a number of applications for CNTSUs.

Overall it is hoped that this work will contribute to the development of aerosol-based processes for the commercialization of nano-manufactured products.

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