Synthesis of single-walled and multi-walled carbon nanotubes in a microwave and hot-wire CVD reactor for nanobattery applications

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There is intense interest in carbon nanotubes (CNTs) because of their potential applications in strong, light-weight composite materials, nanoscale wires, interconnects and adsorbents for gas separation and storage. So the development of a simple, cost-effective and scalable method for CNT production has been a high research priority. In this work we have used a microwave assisted (MA) and hot-wire (HW) chemical vapor deposition (CVD) for the growth of single- and multi-walled CNTs.

We have grown both oriented and non-oriented on a variety of substrates CNTs using MACVD. In particular, we are interested in the growth of oriented CNTs in porous alumina substrates. The CNTs were grown using a two-step process. First, Ni catalyst was introduced into the pores using either sputtering or electrodeposition and then growth was initiated in a microwave plasma. The microwave reactor consisted of an ASTeX 5010 reactor with a 1.5 kW power supply. The CNTs grown in ~ 20 nm pores are used as electrodes in nanobatteries.

The HWCVD reactor consisted of a quartz tube with a diameter of 2” and a length of 30”. Two tungsten filaments connected in series to tantalum rods inside the reactor were used as a heat source. The pressure inside the reactor was maintained at ~150 torr and ferrocene was employed to provide a gas-phase metal catalyst. A gaseous mixture of Ar/CH_4 or H_2/CH_4 or $Ar/H_2/CH_4$ was activated over the hot filaments in the presence of the ferrocene. The reaction time was usually between 15-30 min so that all the ferrocene in the gas phase had reacted leaving a black deposition on the inside walls of the quartz tube. Often a third molybdenum wire was incorporated into the system serving as an additional heat source and possibly providing an additional gas-phase catalyst via evaporation. The material was easily scraped off the quartz tube reactor and analyzed using transmission electron microscopy (TEM). TEM indicated the presence of both single-and multi-walled carbon nanotubes with the diameters ranging from ~1-30 nm.