CVD Synthesis of Carbon Nanotubes as Active Materials for Electrochemical Capacitors

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In this work, results are reported on a parametric experimental approach to synthesize multi-walled carbon nanotubes (MWNTs) using a chemical vapor deposition (CVD) method. The purpose of this work was to optimize a process for carbon nanotube growth specifically targeted for active materials in electrochemical capacitors.

A metallocene catalyst (iron, nickel, or cobalt) was dissolved in xylene and injected via a syringe pump into a heated volatilization zone in a small furnace. The liquid metallocene-catalyst mixture was volatilized and swept into a multi-zone quartz tube reactor with an argon/hydrogen gas, where the nanotubes are grown. Metal atoms deposit along the wall of the quartz tube, as the metallocene/xylene mixture decomposes. As the catalyst mixture reaches the reaction zone, it is heated to a specified temperature for nanotube growth. A schematic of the reactor used to grow MWNTs is shown in Figure 1.

The metal atoms are catalytic sites for nanotube growth while the decomposed xylene is the carbon source. An alternate carbon containing gas, such as ethylene or carbon monoxide, may also be introduced as part of the experiment to grow nanotubes.

A series of experiments were performed to examine the effect of various process parameters on nanotube yield and quality. Some of the experimental variables investigated include:

1. Growth temperature
2. Sweep gas flow rate
3. Metallocene type
4. Reaction Time
5. Catalyst/carbon atom ratio
6. Catalyst/xylene feed rate

The electrochemical properties of as-grown carbon nanotubes were evaluated in sulfuric acid and potassium hydroxide electrolytes using electrochemical impedance spectroscopy (EIS) and cyclic voltammetry (CV) in symmetric carbon-carbon capacitors.

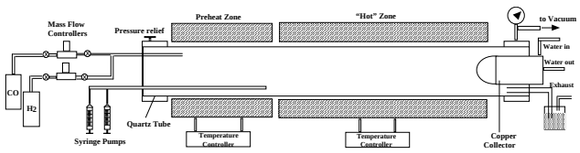

Figure 1. Schematic of CVD nanotube reactor.