Synthesis of Single-Wall Carbon Nanotubes in a High Frequency Furnace

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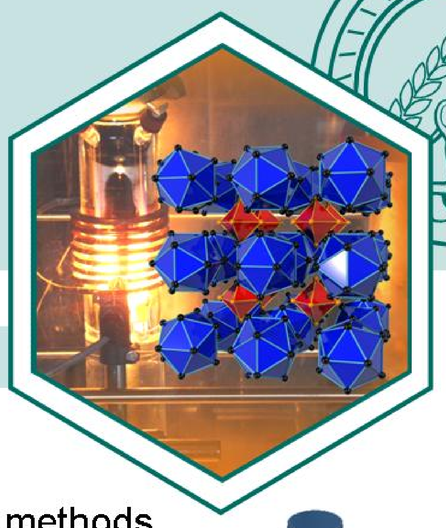

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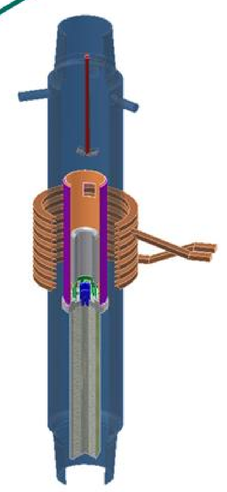
Objectives / Introduction

Carbon nanotubes can be synthesized by various methods which can be classified in high temperature methods (arc-discharge, laser vaporization), which are based on evaporation of a solid carbon source, and "low temperature" methods (chemical vapour deposition), which are based on decomposition of a carbonaceous gas.

The high frequency furnace was found to be an effective method for fullerene as well as endohedral fullerene synthesis. In this work the synthesis of carbon nanotubes by direct co - evaporation of a solid carbon body and a metal catalyst in a high frequency furnace was investigated.

Raman Spectroscopy, SEM and TEM were used for characterization.



Experimental setup: HF - Furnace

As susceptor as well as carbon source for the CNT synthesis, highly pure isostatically pressed graphite in the shape of hollow cylinders was used.

The graphite susceptor is surrounded by two pyrolytic boron nitride (PBN) shields. Those protect the surrounding quartz tube and secondly insulate the reaction space around the graphite susceptor from heat loss. The working coils are arranged around the quartz tube.

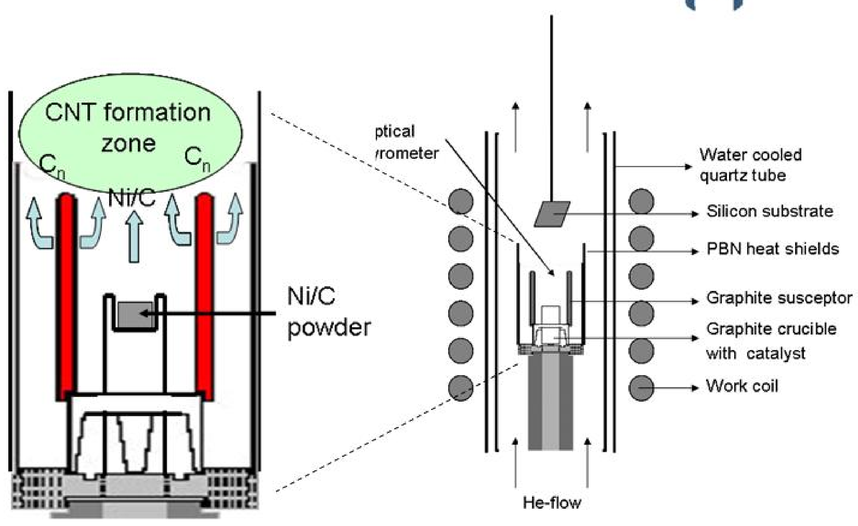
The temperature is monitored at the top of the inner side of the graphite body where the temperature is highest using an optical pyrometer. The onset of carbon evaporation from the graphite body (carbon source) was observed at about 2400 ° C.

The metal catalyst was positioned in a graphite crucible in the inside of the graphite body. The crucible can be height adjusted.

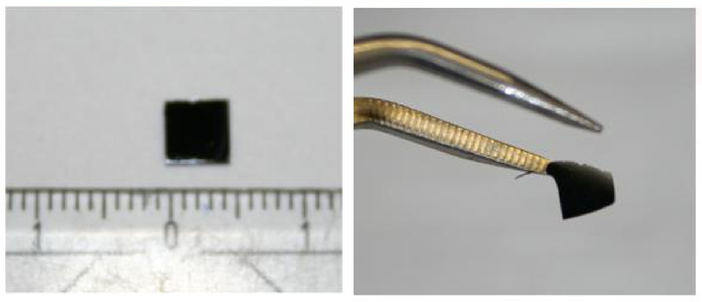
A substrate can be inserted with a special sample holder at any horizontal position for in situ sample collection.

Ni was used as catalyst metal.

The setup provides good control over synthesis parameters and design of the reaction chamber.



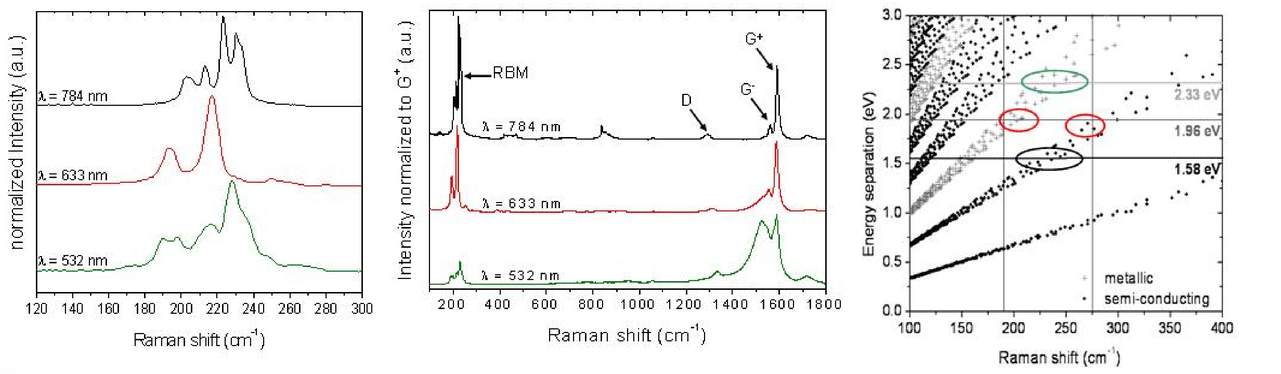
Results



A black deposit formed during synthesis on the silicon substrate and the furnace walls.

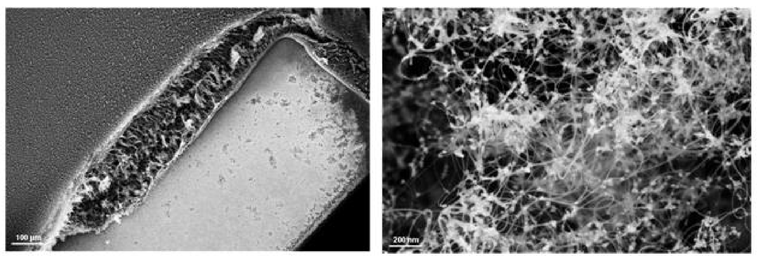
Raman Spectroscopy

Raman spectroscopy was used for bulk characterization of the as produced soot.



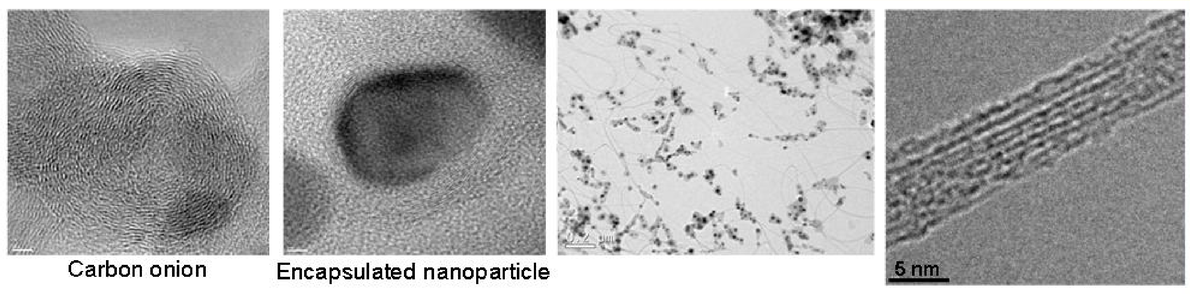
According to Raman spectra the CNT diameter lies between 1.26 – 0.84 nm (estimation from equation ω_{RBM} = 224 cm^{-1}/d + 12 cm^{-1}). Both metallic as well as semi-conducting SWCNTs form during synthesis. This is illustrated with a Kataura plot. It can also be deduced from the G^- -peak shape which shows typical shape of semi-conducting SWCNTs for the spectrum taken with 1.58 eV excitation energy and a Breit-Wigner-Fano peak shape (typical for metallic SWCNTs) for the spectrum taken with 2.33 eV excitation energy. The 1.96 eV spectrum shows contribution from both SWCNT types. For all three excitation energies a small D-band is observed which indicates "good quality" SWCNT material with low amount of defects.

SEM



SEM images of black synthesis deposit on the silicon substrate. The deposit consists of highly entangled bundles of SWCNTs and synthesis byproducts. No MWCNTs were observed. The deposit is very uniform over the whole substrate regarding thickness and morphology.

TEM



TEM confirms the formation of single-wall carbon nanotubes which were found to be in a state of bundles. Carbonaceous byproducts like cabron onions and carbon encapsulated metal nanoparticles were also observed. The bundle diameter ranges from about 14 nm down to individual SWCNTs in rare cases.

Conclusions

- SWCNTs have been synthesized in a high frequency furnace by direct co-evaporation of graphite and a metal catalyst
- The SWCNTs obtained exhibit a diameter distribution between 1.26 – 0.84 nm and were found in a state of several µm long bundles
- Both semi-conducting as well as metallic CNTs were formed
- No MWCNTs were observed in the substrate deposit

Future Development

As it is possible to insert a substrate directly over the evaporation zone and to control the substrate's temperature by its vertical position in the furnace it seems to be possible to grow carbon nanotubes in a "CVD"-like mode using predeposited catalyst particles on the substrate as nucleation seeds.

Different catalyst systems and their influence on the CNT product will be investigated as well.

References

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