Production of well dispersible single-wall carbon nanotubes via a “floating catalyst”-method

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Single-wall carbon nanotubes (SWCNT) exhibit unique physical and mechanical properties for the use as composite material in many different applications. We present a unique process chain for the production of SWCNTs and identify the relationship between the process parameters in gas phase production and the properties of nanotubes. Therefore, a solution of ferrocene in ethanol was used for the production of SWCNTs by a floating catalyst method in a vertical three stage heating system.

The optimized riser reactor design reduces undesired wall-sticking of the catalyst particles and SWCNTs. Homogenous precursor distribution is achieved by an aerosol inlet with 10 mm diameter which is surrounded by a shield gas flow through a sinter plate with the same diameter as the quartz tube. As the rising precursor undergoes decomposition to subsequently form the catalyst particles, the diffusion towards the reactor wall is suppressed by the surrounding inert gas. Production rates in the range from 5 to 20 mg/h were achieved in a quartz tube with 1 m length. SWCNTs have been separated from the outlet gas flow by a PTFE membrane.

The influence of initial precursor concentration on remaining iron content has been investigated. In combination with a short residence time in the reactor, a product with an iron catalyst content of less than 30 wt.% has yielded.

TEM analysis show smaller catalyst particles with decreasing precursor concentration. Raman analyses of the SWCNT Radial breathing mode with different excitation wavelengths and UV-Vis spectroscopy indicate that the diameter distribution of SWCNTs is attributed to the range from 0.6 to 2.0 nm. Moreover, Statistical Raman analysis enabled in-depth investigation of the purity and electronic properties of the product. Processing conditions, as such precursor concentration, residence time, etc. were found to have an impact on the mean geometric SWCNT diameter and standard deviation.

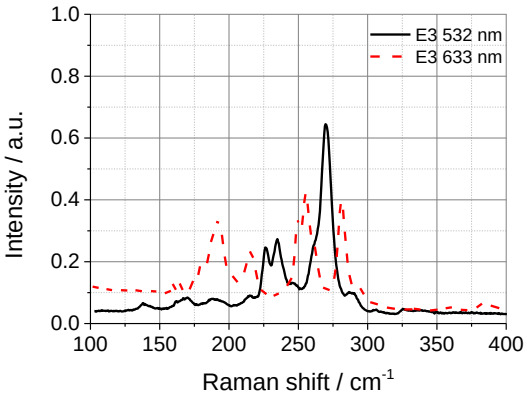

Figure 1. Raman spectra of SWCNT buckypaper at excitation wavelengths of 532 nm and 633 nm, normalized with G-Band intensity.

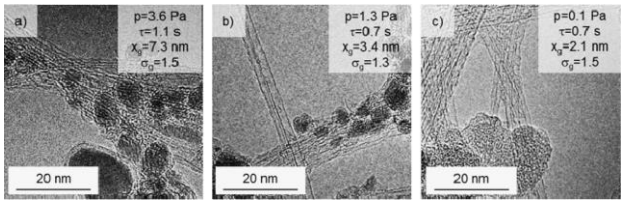

Figure 2. TEM images for SWCNTs produced at different precursor concentrations.

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