

The synthesis and characterization of fluorinated single-walled carbon nanotubes

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Single-walled carbon nanotubes (SWCNTs) were treated with fluorine at fluorine pressure over 0.8 to 2.3 bar range and temperature over 140 to 345°C range. The following methods were applied: transmitting electron microscopy, gas absorption, ¹³C and ¹⁹F NMR, FTIR spectroscopy, X-ray absorption and photoelectron spectroscopy, thermal stability (analysis of gaseous products by mass-spectrometry). The limiting stage of fluorination of SWCNTs (in a form of powder or paper) is the diffusion of fluorine inside bundles and nanotube microcrystals. At optimum conditions ($p_F=0.8$ bar, temperature 190°C, treatment duration 5 hours) structure of SWCNTs remains unchanged up to composition CF_x , $x=0.436$, close to theoretical limit $x=0.5$. Thermal destruction of fluorinated SWCNTs is negligible when treatment temperature does not exceed 190°C (fluorination duration is equal to 5 hours), but become important when temperature exceeds 220°C. Above 220°C partial gasification of fluorinated SWCNTs is accompanied with amorphysation and bundles aggregation. Below 190°C degree of fluorination in SWCNT bundles falls with depth and decreases markedly at points placed 1.5 nm from the SWCNT surface. Fluorine atoms are covalently attached to SWCNT surface and to carbon atoms at open nanotube ends. The internal nanotube channels became inaccessible even for small size molecules such as N₂.