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Synthesis and characterization of single-walled carbon nanotube via low-pressure chemical vapor deposition from FeMo catalyst

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Abstract :

Materials science of carbon nanotubes (CNTs) lies at the intersection of various paradigms from fundamental to applied physics and chemistry. However, understanding the mechanism of CNTs formation through different concepts remains a considerable challenge. Currently, to be able to use CNTs for specific applications, the synthesis of CNTs with the desired diameter and chirality remains one of the most critical challenges for nanotube science. To face this challenge, the understanding of growth mechanisms of CNTs has to improve and complete, thereby developing highly selective synthesis methods. Previous studies suggest that the use of catalysis in carbon tube growth significantly enhances understanding of the very early stage of CNT nucleation and is a promising method for the selective synthesis of CNTs through the chirality control. A well-controlled synthesis of catalyst nanoparticles (composition, morphology, size) appears to be a mandatory condition for controlling the characteristics of the as-synthesized nanotubes. This study aims to complement the well-established ex-situ TEM observations of SWCNTs growth with Raman spectroscopic analysis and scanning electron microscopy (SEM) when Fe-Mo catalyst is used. The synthesis was carried out in a homemade hot filament chemical vapor deposition (HFCVD) reactor. This CVD system allows conducting studies on the influence of various parameters in synthesizing SWCNTs such as temperature, pressure, gas flow and catalyst. The unique feature of this system is the ability to reduce the reaction pressure to a maximum of 10^{-4}mbar allowing studies of the SWCNTs synthesis at low pressure.

As applied to different new and controlled bimetallic nanoalloy catalysts types, such as Fe-Mo catalyst, the emphasis is to understand the role played by the catalyst and put forward a growth mechanism essential to realized SWCNTs with specific and targeted chirality. In particular, we will discuss the role of the growth pressure on the final SWCNTs structure, In the view of experimental results, we observed a strong

influence of Mo when it acts as a co-catalyst with Fe. The SWCNTs growth mechanism is “tip-mode”, in which the catalyst nanoparticles reside at the top of CNTs. Mo acts as a co-catalyst to improve the ability of small Fe catalyst nanoparticles to stay active among conditions of high carbon flux with CH4, thereby contributing to an increase in the number of synthesized SWCNTs. With 0.5 nm of Fe thickness, a sufficient amount of added - Mo could enhance the quality of SWCNTs and increase the number of SWCNTs. However, the effect of Mo gradually decreases when a large amount of Mo is used as a co-catalyst.

This study will be a prototype for the in-situ study in the environmental transmission electron microscope (ETEM) implemented with a CVD gas source. Special thanks to the ANR’s funding for the project GIANT (number: ANR-18-CE09-0014)