Low Dimensional Materials and Catalysts

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Systematic characterization of dynamics of transition metals inside SWNT by in-situ aberration-corrected HRTEM at low acceleration voltages between 20 and 80 kV

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Single-walled carbon nanotubes (SWNTs) attract great attention for their aptitude as nano-test tubes and have proven to be an ideal confinement for atomically resolved imaging of dynamics and interactions of sub-nanometer sized molecules. Apart from a matrix free stabilization of molecules, SWNT offer protection against undesired beam damage resulting from ionization or chemical etching under a highly energetic electron beam. Moreover SWNT provide a unique environment for the investigation of carbon-based reactions at the atomic scale. This unique combination make it a precious test-system for the study of interaction mechanisms between the specimen and the energetic electron-beam by nature omnipresent in transmission electron microscopy (TEM) observations.

In this study we present in-situ irradiation experiments of sub-nanometer sized *d*-element metal-nanoclusters enclosed in SWNTs by means of low voltage aberration-corrected high-resolution transmission electron microscopy (AC-HRTEM).[2,3] The transition metals are varied systematically along Groups as well as Periods of the Periodic Table of Elements in order to characterize their properties and track their interactions with the electron beam and the carbon special environment. In these experiments the particular technique of HRTEM combines imaging tool and irradiation source in one integral experiment while further benefiting from the SWNTs low and regular contrast combined with minimized susceptibility to head-on collisions of the electrons with the carbon atoms nuclei (knock-on damage) while operating with electron energies less or equal 80 keV.[4] In our experiments we pursue the methodology of varying both, the metal type and the electron acceleration voltage (between 20 kV, 40 kV and 80 kV). Thus we are able to separate the influence of the electron beam from that of the specimen which is a necessary condition for the detailed and atomically resolved study of sample or irradiation induced structural SWNT-modifications. Finally it is our aim to explicitly describe the interaction mechanisms between carbon and the different under the influence of the electron beam. While irradiating, in order to answer the demand of comparability, time series of interactions are recorded until reaching the same total electron dose of 10^{10} e^-/nm^2. Experiments are conducted using a Cs-corrected FEI Titan 80-300 operated at 80 kV and a monochromated/Cs-corrected SALVE (Sub-Ångstrøm Low-Voltage Electron Microscopy) Zeiss LIBRA prototype microscope operated at 20, 40 and 80 kV.

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2. A. Chuvilin, S. Roth, U. Kaiser, Angew. Chem. Int., (2010)
3. U. Kaiser, A. N. Khlobystov, G. Benner, UM (2011)
4. J. Meyer, F. Eder, U. Kaiser, Phys. Rev. Lett. 108 (2012)

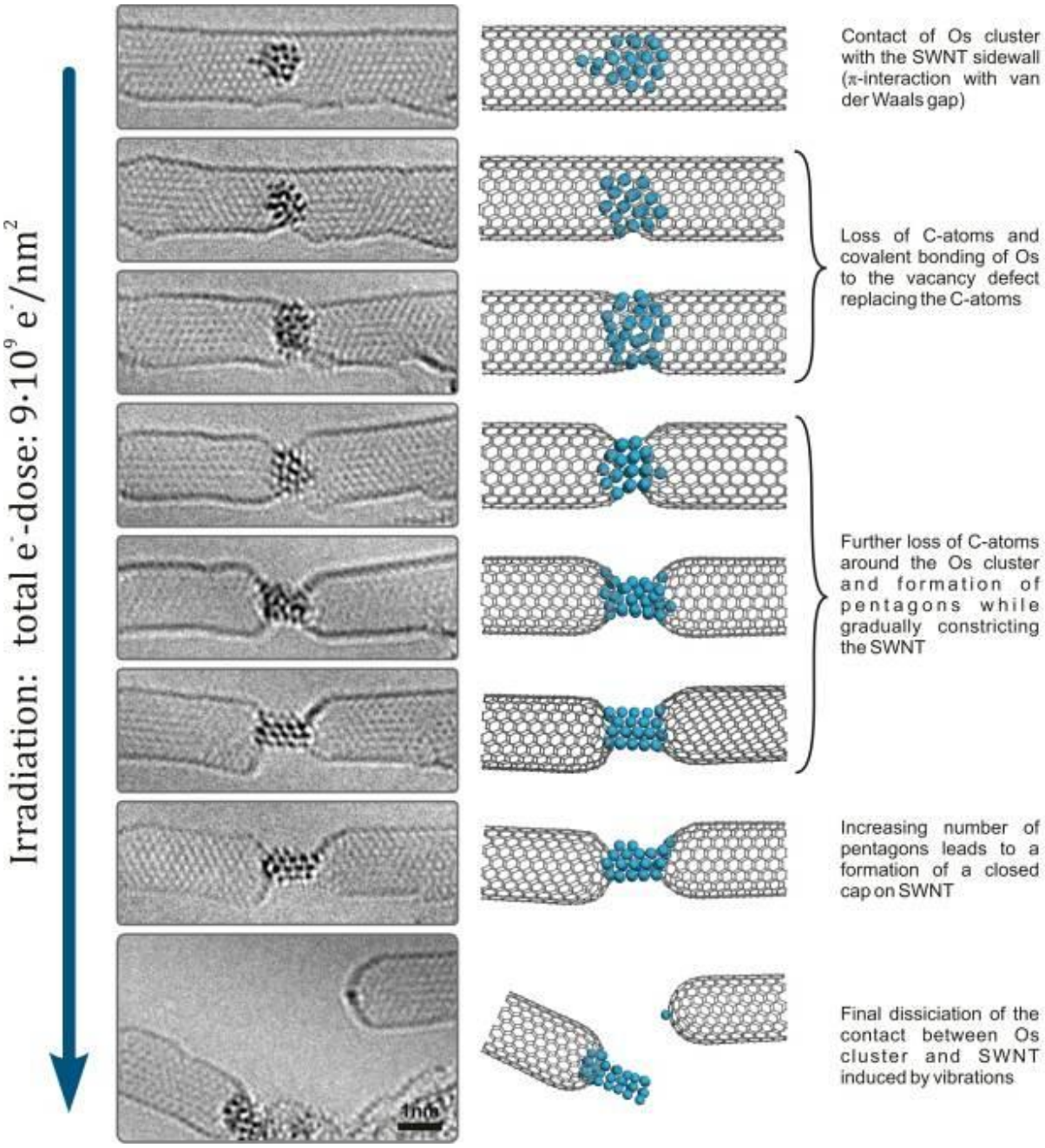

Figure 1. The time-series shows the stages of the interaction between an Os nanocluster and SWNT inner sidewall induced by the electron-beam imaged by AC-HRTEM at 80 keV. On the right column are the corresponding structural diagrams.