EELS investigation of carbon nanotubes and other carbon modifications

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Electron energy loss spectrometry is a powerful tool for the investigation of the electronic structure of materials. The low energy part of the EELS spectrum gives access to the same kind of information as optical absorption measurements while core loss edges mirror the site- and angular-momentum projected density of unoccupied states and gives information similar to that obtained from X-Ray absorption spectrometry.

In the following we will show how the different regions of the EELS spectrum can be used for the characterization of carbon-based modifications. Such materials show up in a wide range of arrangements leading to very different electronic structures in spite of their same chemical composition. They are therefore an excellent test case for EELS investigations. Moreover, the carbon K edge has very pronounced fine structures extremely sensitive to the electronic structure of the material. In addition, the relatively high ionization cross section for the excitation of the 1s electrons and the low density of the material guarantees a high signal/background ratio at the C-K edge.

First we will show that the low loss spectrum is very sensitive to small changes made to the structure. The investigated materials are bundles of single wall carbon nanotubes. Some of them are filled with organic molecules, while the other are pristine tubes. It is possible to detect faint changes in the plasmon loss upon filling of the tubes. Figure 1 shows a typical low loss EELS of a bundle of carbon nanotubes. The two main features can be fitted with Gaussians and their positions determined by the position of the maximum of the Gaussian. When the tubes are filled with organic molecules, the peaks are slightly shifted to higher energies.

In the second part, we will address the core loss spectrum. The carbon K edge can be used to characterize the sp2/sp3 ratio in the carbon materials, thus giving access to the local hybridization of the material. For this purpose, it is mandatory to work under the so-called "magic angle conditions". This particular setup of the acquisition conditions (convergence and collection angles) guarantees that the changes in the fine structures are only due to the binding of carbon atoms and not to different orientations of the strongly anisotropic material with respect to the electron beam. The spectra are recorded under magic angle conditions[1]. Three Gaussian are used to fit the first 15eV of the spectrum, two of them are attributed to the transitions to σ states and one to transitions to the π states (figure 2). Higly oriented pyrolitic Graphite (HOPG) is used as a reference materials for the pure sp2 carbon. Even if the absolute value of the sp2/sp3 ratio should be taken carefully, faint changes in this ratio can be detected. Figure 3 shows the changes in the sp2/sp3 ration for different carbon modifications used as catalysts before and after reaction[2]

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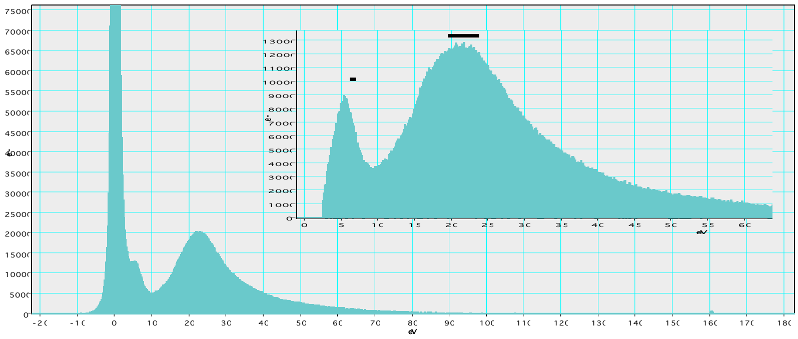

Figure 1. Low loss spectrum of a bundle of single wall carbon nanotubes.

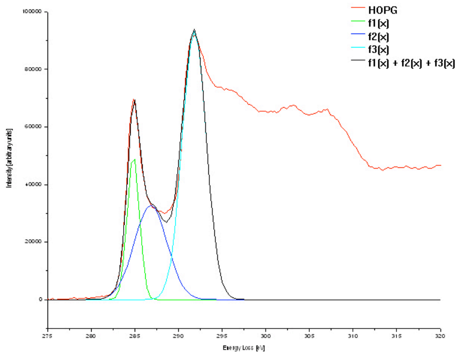

Figure 2. Carbon K edge of HOPG recorded under magic angle conditions and the three Gaussians used to fit the fine structure of the edge.

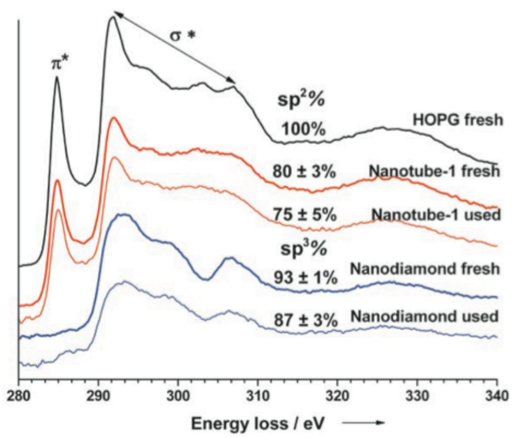

Figure 3. Carbon K edge obtained from different carbon modifications before and after use as catalysts. The sp2/sp3 ratio was determined from many measurements made on different places on each sample. From [2]

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