

Experiment 08-01-1051, BM08

Title: Iron oxide layers of nanopetals for photocatalytic water treatment: investigation of Fe site at different steps of nanopetal formation.

Participants: Chiara Maurizio, Borid Kalinic

Experimental conditions: XAS at Fe K-edge.

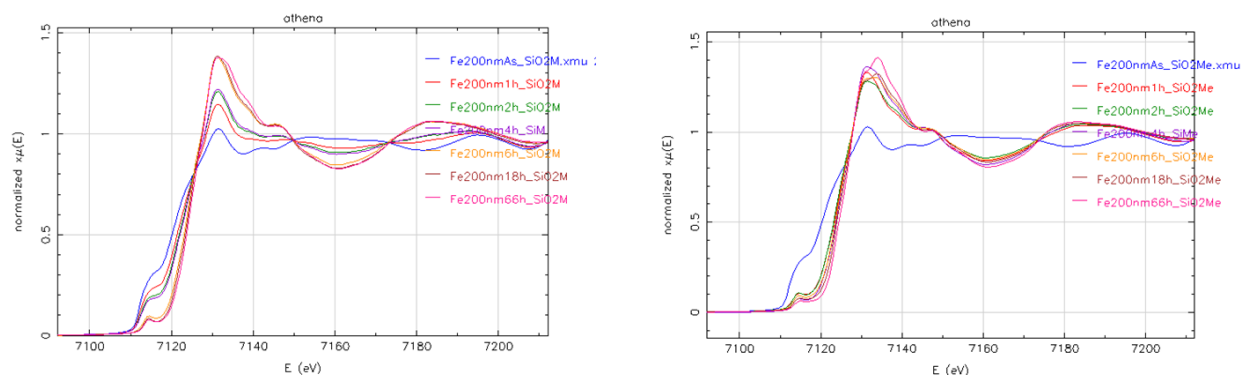
Samples:

The samples were Fe films where the growth of nanowires was induced upon suitable oxidizing annealing. The growth, that takes hours at 300 C, was stopped at different times, in order to monitor the local order changes during the oxidation process. Fe K-edge XAS spectra of the films were recorded in fluorescence mode and in total electron yield (TEY) mode, in order to gain a partial surface sensitivity.

Report

We measured the Fe K-edge XAS spectra of Fe films of different thickness after annealing for 0, 1, 2, 4, 6, 18h in air.

In the figure the spectra obtained in fluorescence yield (left) and in total electron yield (right) are reported for a Fe film 200 nm-thick (data labels are *Fe200nm-AnnealingDuration-substrate*). The as-deposited film corresponds to the blue curves. Data of the same quality were obtained for films of lower thickness.



As visible in the figure, for the same time duration the oxidation is much more evident close to the film surface (total electron yield). An interesting point is that the XANES spectrum is much more similar to the one of Fe₃O₄ (fluorescence yield) even after 66h of annealing, condition in which the TEY gives a spectrum Fe₂O₃-like. This is interesting, since in this condition TEY takes contribution essentially from the nanowires grown on the surface while the fluorescence yield give information especially on the underneath layer. This comparison also shows that the fraction of Fe involved in the nanowires is minimum. The following picture compares for the 66h-annealed film the XANES spectra recorded in fluorescence mode (blue), in TEY mode (red) and fluorescence mode recorded on nanowires peeled off from the substrate, confirming the Fe₂O₃ nature of the nanowires.

The analysis is in progress. Coupled with XRD, and scanning electron microscopy, they will allow to model the mechanism of nanowire formation.

