Surface Analysis of Isolated Nanoobjects by XPS

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X-ray photoelectron spectroscopy (XPS) is a very efficient and still progressing surface analysis technique. However, when applied to nanoobjects, this technique faces drawbacks due to interactions with the substrate and sample charging effects[1]. We present a new experimental approach to XPS[2] based on coupling soft x-ray synchrotron radiation with an in vacuum beam of free nanoparticles, focused by an aerodynamic lens system (Fig 1a). Two examples of experiments performed on the PLEIADES beamline at the SOLEIL Synchrotron facility are presented to illustrate the effectiveness of this approach.

In the first example, the structure of the Si/SiO_2 interface is probed on isolated silicon nanocrystals previously oxidized with ambient air[1] (Fig. 1b) or by heat treatment under air. Full characterization of the surface has been achieved for different sizes of nanocrystals between 4 and 80 nm and with different oxidizing treatments. The technique allows probing the presence of various oxidation states at the interface and to deduce therefrom a thickness of the oxide layer. For the smaller and more oxidized nanoparticles, a relatively abrupt interface including Si = O double bond is highlighted.

In the second example, the adsorption of water on the surface of TiO_2 nanoparticles is investigated in the gas phase[3]. TiO_2 free aerosols are exposed to a controlled pressure of water vapor before being analyzed directly by XPS. The technique allows here the observation of a predominantly dissociative adsorption of water on the surface of TiO_2 in its first stage, highlighting a largely covered surface by OH groups (Fig. 1c).

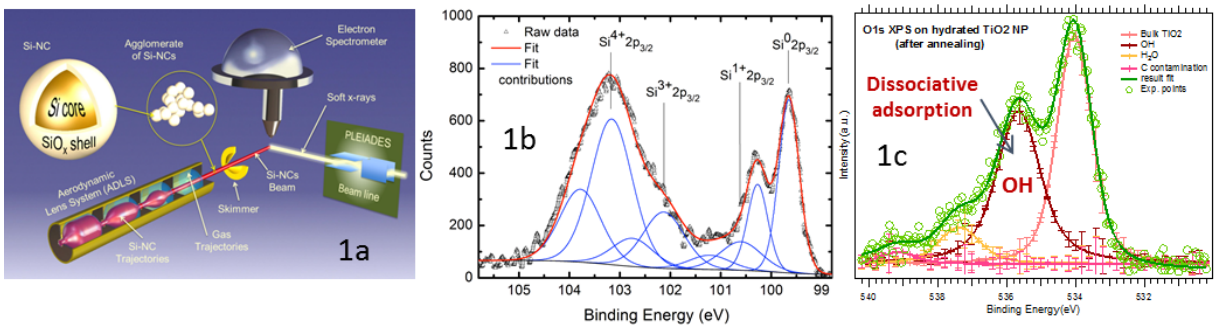

Fig. 1a. XPS of isolated nanoparticles. **Fig1b.** Si2p XPS spectrum of isolated silicon nanocrystals. **Fig 1c.** O1s XPS spectrum of hydrated TiO_2 nanoparticles.

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Acknowledgements

The experiments were performed at the PLEIADES beamline at the SOLEIL Synchrotron (Proposals No. 20110423 and 20130466). The development of the MPSC has received funding from the Agence Nationale de la Recherche (ANR) under Grant No. ANR-07-NANO-0031.