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Surface Chemical Structure of Isolated Aerosols by X-ray Photoelectron Spectroscopy

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X-ray photoelectron spectroscopy (XPS) is a powerful tool to investigate the surface chemical structure of any material. However, when applied to nanoobjects, this technique faces drawbacks due to interactions with a substrate, on which nanoobjects have to be deposited, and sample charging effects. We present a new experimental approach to XPS¹ based on coupling soft x-ray synchrotron radiation with an in vacuum beam of free nanoaerosols, 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/SiO₂ interface is probed on isolated silicon nanocrystals previously oxidized with ambient air¹ (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 bonds is highlighted.

In the second example, the adsorption of water on the surface of TiO₂ nanoparticles is investigated in the gas phase³. TiO₂ 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 TiO₂ in its very first stage, highlighting a largely covered surface by OH groups (Fig. 1c).

[1] Baer, D. R. et al (2010) *Chem.*, **396**, 983.

[2] Sublemontier, O. et al (2014) *J. Phys. Chem. Lett.*, **5**, 3399.

[3] Benkoula, S. et al (2015) *Sci. Rep.*, **5**, 15088.

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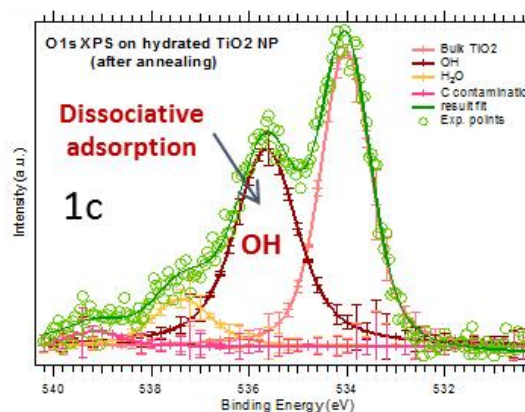
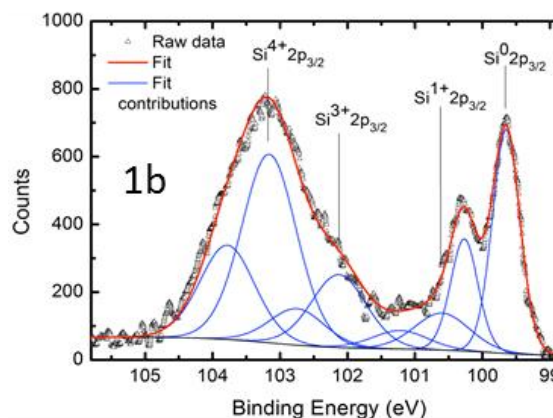
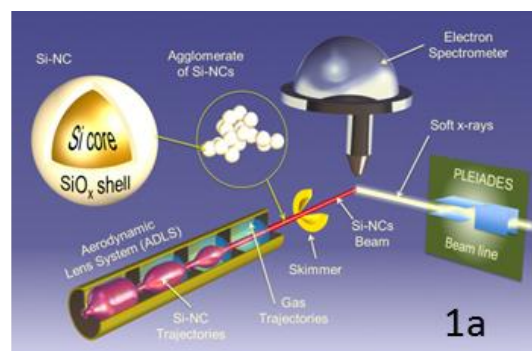


Figure 1a. XPS of isolated nanoparticles. **Figure 1b.** Si2p XPS spectrum of isolated silicon nanocrystals. **Figure 1c.** O1s XPS spectrum of hydrated TiO₂ nanoparticles.