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Novel photocatalytic set-up for ambient pressure X-ray photoelectron spectroscopy

Content

Processes driven by solar radiation play a crucial role in life on Earth. Nowadays,replacing fossil fuels with clean and renewable energy sources is one of the most important challenges for modern human civilization. Natural photoassisted solar reactions can convert solar energy into usable chemical energy by driving reduction and oxidation processes via generation of electrons and holes, respectively, upon irradiation of the photocatalyst. Photo-assisted atomic layer deposition (Photo-ALD) is a less studied approach to facilitating selective area ALD. ALD is an extremely versatile thin film deposition process that is currently used in many steps within the microelectronics process flow and is also attracting increasing interest in other industrial areas.

The APXPS endstation at SPECIES beamline at MAX IV is designed to perform *in-situ* and *operando* XPS experiments in a controlled atmosphere at pressures up to 20 mbar. Research areas of interest include catalysis, material and surface sciences, environmental chemistry, and atomic layer deposition. However, there is a lack of APXPS studies that pair synchrotron light with external light sources. Here we will introduce and discuss the use of solar irradiance with APXPS cells and provide a series of scientific case studies that serve to illustrate the usefulness of the concept.

One such scientific case is hydrogen production via water splitting sunlight is used to induce a photocatalytic reaction at a photoactive surface. We study pristine Ni@NiO/NiCO3 core-shell nanostructures as photocatalysts and characterize their post-annealing variants. Thorough structural (XRD) and spectroscopic (XPS, TEM/EELS) investigations revealed a core Ni nanoparticle decorated with a 3-5 nm thick composite shell of crystalline NiO and amorphous NiCO3. During illumination, it was observed that the metallic Ni peak vanishes, and the new peak appeared that we've assigned to NiOOH (Fig. 1). However, no changes were observed in the Ni 2p peaks from NiO and/or NiCO3.

Another example lies in an under-utilized technique called photo-ALD which allows spatially selective ALD to be carried out. Herein, we tested the set-up using the titanium tetraisopropoxide precursor to deposited TiO2 onto a Si wafer. Throughout the deposition, XPS was recorded with time-resolved snapshot mode tracking O 1s, Ti 2p, C 1s, Si 2p. Upon exposure to the UV light, time-resolved data for the Ti 2p core-level showed significant changes in intensity of Ti3+ vs. Ti4+ correlating to prolonged UV radiation exposure.

In summary, the APXPS endstation at SPECIES beamline has two ambient pressure cells, a standard cell for catalysis, redox chemistry, etc. and an ALD cell for ALD research. Solar simulator and UV-lamp can be used with both cells, as we've demonstrated. These combinations provide a unique platform to conduct experiments to study the electronic structure of matter under IR/visible/UV radiation and at different pressure ranges and gas mixtures.

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