INDUSTRIAL PILOT PROJECT

In operando studies of Ru(III) and Ti(III) oxidation at calcination of electrode coatings

THE INDUSTRIAL CHALLENGE

Ruthenium dioxide (RuO_2) or mixtures of RuO_2 and other metal oxides (TiO_2, IrO_2, etc.) have already for decades successfully been used as catalytic and durable electrode coatings in different industrial processes (such as chlorine, oxygen, or hydrogen gas production). The RuO_2-based electrode coating is deposited on a titanium (Ti) or nickel (Ni) support and consists of ~20 nm nanoparticles formed in a calcination process where precursor solutions of Ru(III) oxidizes in air to Ru(IV). The knowledge about the oxidation process is however very limited, especially in the view of large-scale industrial production, which prohibit R&I regarding further optimization of the calcination process in the production of efficient and energy saving electrodes. This is mainly because of experimental difficulties to study the oxidation process *in operando*.

WHY USING A LARGE SCALE FACILITY

X-ray photoelectron spectroscopy (XPS) is an element sensitive technique that can provide chemical-specific information about the probed elements in a sample. It is, thus, possible to follow the oxidation of Ru during the calcination process while it occurs. The formation of RuO_2 require oxygen gas and performing experiment in an O_2-gas mixture in turn require a high X-ray intensity that can only be obtained in a synchrotron radiation (SR) facility. In addition, the high X-ray intensity facilitates fast data acquisition necessary for in operando measurements. It is also possible to obtain the resolution needed to observe the small binding energy shifts in the XPS spectra during the calcination process. Hence, this study would be impossible using conventional XPS.

HOW THE WORK WAS DONE

An initial study was performed using a conventional XPS system, which provided information needed to select suitable samples prepared at the Permascand research lab. The *in operando* study was part performed at the ambient pressure XPS beamline HIPPIE at MAX IV in Lund.

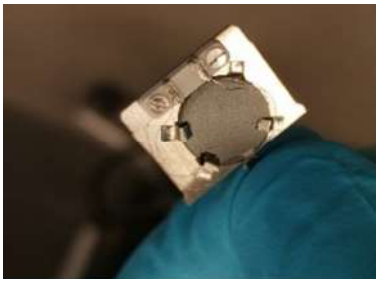

Figure 1. A circular coated electrode mounted in a sample holder, ready for SR-XPS at MAX IV.

THE RESULTS AND EXPECTED IMPACT

As displayed in Figure 2, RuO_2 is formed at 370 °C. However, a temperature of 475 °C was needed to obtain an electrocatalytic active coating. The experience has provided new perspectives on the calcination process and valuable inputs for the optimization process.

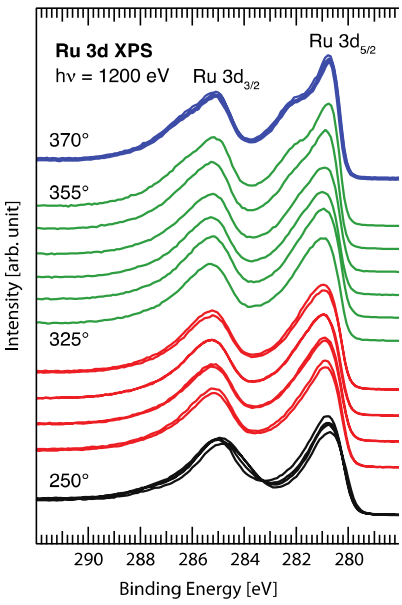

Figure 2. Ru 3d XPS of the transition between Ru(III) and Ru(IV) forming RuO_2-based coating at 325-355 °C (green spectra).

"We see a great value in the results for our future product development"
/Fredrik Herlitz, CTO, Permascand AB

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