Elaboration and optimization of oxygen DSA of the type Ta / IrO_2

Patrice Palau, Pierre Taxil and André Savall*

Laboratoire de Génie Chimique, CNRS, UMR 5503, Université Paul-Sabatier,
118, route de Narbonne
31062 Toulouse Cedex 04, France.
* savall@chimie.ups-tlse.fr

Iridium dioxide is used as catalyst in Dimensionally Stable Anodes (DSA) for oxygen evolution. The experimental conditions to obtain the layer of iridium dioxide by thermal decomposition of the precursor influence strongly the behaviour of a Ta/IrO_2 electrode. In this work a detailed investigation was carried out on the influence of the temperature of preparation of the IrO_2 layer on its structural properties. IrO_2 was prepared by thermal decomposition of $IrCl_4$ in an alcoholic solution applied on a tantalum layer electrodeposited on a substrate of steel in molten salt.[1,2] The temperature range explored was comprised between 400 and 500°C. In a preliminary part, it was shown that IrO2's layer deposited on tantalum should have a minimum thickness of 1,1 µm to give reproducible service life.

The morphology of the layers prepared at various temperatures was observed by SEMicroscopy and DRX analysis. Data obtained on the crystallization of the oxide layer were correlate with the pyrolysis temperature. Rutherford spectroscopy (RBS) measurements evidence the presence of tantalum oxide into the electrocatalyst layer. Measurements by Nuclear Reactive Analysis (NRA) of the carbon and oxygen contents in the electrocatalyst layer prepared for different temperatures, allow to optimize the temperature range to obtain pure IrO_2 free from organic compounds.

The influence of the temperature of pyrolysis was examined in the overall work. For too low temperatures (340-380°C), the layer was composed of amorphous and hydrated IrO_2 with impurities in the lattice of the compound. Consequently, the electrodes elaborated at this temperature have the most important specific area and then displays highest activity. However, their stability under anodic polarisation is too low, and they are rapidly deactivated during water electrolysis.

For a better stability of the electrodes, the temperature of pyrolysis should be increased in order to obtain a crystallized structure, with a large size of the crystals, low inner porosity and to eliminate impurities by a complete decomposition of the precursor. However, the increase of the temperature above 420°C favours the oxidation of the tantalum substrate in an insulating oxide Ta_2O_5. When the temperature is above 500°C, the content in this oxide is too high and causes the passivation of the electrode. Because of these two antagonist effects of the increase of the temperature a compromise was found in the 420-500°C range; the optimized value, determined by comparing the stability in polarisation conditions of the electrodes prepared at various temperatures, was 460°C which is the temperature corresponding to the maximum of the service life of the electrodes (15.8 $h.m^2.g^{-1}$). Correlatively, the inner porosity of the layer is minimum at this value.

Acknowledgement

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Reference

1. P. Chamelot, P. Palau, L. Massot, A. Savall, and P. Taxil, *Electrochim. Acta* **47** (2002) 3423.
2. Patent Nr 01401628.1-2119, Electricité de france, Véronique Reid, Olivier Bulteau, Alain Hita, André Savall, Pierre Taxil, Patrice Palau.