

Electrochemical impedance spectroscopy & Solid oxide electrolysis

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Description

Water electrolysis appears to be as one the foremost technologies for a sustainable production of hydrogen from renewable energy to feed next generation fuel cell vehicles. This technology is characterized by the ability to rapidly follow the intermittent production of renewable energy and, thus, can provide efficient energy storage and grid-balancing service in power-to-gas processes. These characteristics will become very important in the next future because of the increasing impact of renewable sources on the grid and the differences in supply and demand for energy generation and consumption. Accordingly, it is important to get a better understanding of the reaction process and performance limiting phenomena in membrane-electrode assemblies which are the core part of a PEM electrolysis system. In this regard, electrochemical impedance spectroscopy appears an excellent diagnostic tool for electrochemical processes and devices especially when there is no possibility of using an internal reference electrode to help in deconvoluting different phenomena [1]. Ac-impedance analysis is frequently used to separate the contribution of the different mechanisms influencing the polarisation characteristics. Electrochemical Impedance Spectroscopy (EIS) allows to discern between the various phenomena on the basis of the different relaxation times of the single processes and the variation of relevant components of the equivalent circuits upon a change in the operating parameters such as potential, current density, temperature and MEA characteristics, e.g., catalyst loading.

Few Layers of Graphene (FLG) films were grown by Chemical Vapour Deposition (CVD) of ethanol on Cu substrates at 1070°C. The details relative to the CVD growth based on ethanol vapor precursor, to the graphene transfer and to the steps involved in the fabrication of the G/n-Si cells have been previously reported by the authors.

Resumes the cells fabrication process. The starting substrates were commercial n-type Si wafers, 1 Ω cm, with a thermal SiO₂ layer (300 nm) on both sides. The wafers were patterned through HF etching of SiO₂, and an active area of 0.76 cm² was obtained. The back contact was realized by evaporating Al on the back side of the wafer. The graphene films were transferred onto the cells by a cyclododecane (CD)-supported transfer. Two kinds of top contacts were realized. The first was made by standard gold deposition by e-gun evaporation; the second was obtained through a colloidal graphitic glue, spread over the graphene outside the active area [2].

Unsupported IrRuOx catalyst (70% at. Ir: 30% at. Ru), prepared by using a modified Adams process, was used as anode electrocatalyst. A 40% Pt/Vulcan XC-72 was synthesized according to the sulphite complex process and utilised at the cathode.

A chemically stabilised Aquivion® short-side chain extruded film (E98-09S), with a thickness of 90 μm and an equivalent weight (EW) of 980 g/eq developed by Solvay Specialty Polymers, specifically for water electrolysis applications, was used as membrane separator. An ionomer dispersion (830 g/eq equivalent weight-based Aquivion® ionomer—D83-06A) was used as ionic conductor in both the composite catalytic inks.

Membrane-Electrode Assemblies

Membrane-Electrode Assemblies (MEAs) were manufactured by using a catalyst-coated membrane (CCM) preparation procedure. Separate catalyst slurries of IrRuOx and Pt/C with the Aquivion® ionomer dispersion (D83-06A) were sprayed in sequence onto the different faces of the Aquivion® membrane. The ionomer content in the catalytic layers was 15 wt. % at the anode and 28 wt. % at the cathode. The properties of the catalytic inks were examined by using a combination of transmission electron microscopy and X-ray diffraction (XRD) [3]. Structural analysis was carried out by a Panalytical X-Pert diffractometer using a CuKα as radiation source operating at 40 kV and 20 mA. The diffraction patterns were interpreted using the Joint Committee on Powder Diffraction Standards (JCPDS). The morphology of the catalytic layers was investigated by Transmission Electron Microscopy (TEM) using a FEI CM12 instrument.

The macroscopic and microscopic appearance of the different types of titanium substrates studied, fully dense and porous samples. Furthermore, the density values and more important porosity parameters obtained (PT, Pi, and size range of pores), using the Archimedes method and image analysis.

Archimedes Methods

The comparison of the porosity obtained for the three studied substrates: fully-dense as a reference and two designed with a 40 vol. % of spacer and two differences size ranges, manufactured according to the conditions described. First, the macrographs reveal a micro and macropores distribution homogenous on the titanium samples. The microstructure of the titanium substrates and allow the evaluating of the porosity

features, in terms of content, size and morphology. These features are consistent with the values of total porosity and equivalent diameter, in both ranges of spacer sizes studied. Finally topography of the surface of the samples. In summarizes the characterization values performed by the Archimedes method and by IA techniques. These techniques allow the determination of the porous density value, total and interconnected porosity, as well as the equivalent diameter of pores and the pore shape factor while the roughness characterization of inside of pores and on flat surface (arithmetical mean deviation) was obtained with the confocal-laser microscopy technique [4].

This research reports a simple label-free aptasensor for the detection of lysozyme. The electrochemical grafting using carbodiimide chemistry has been a good choice for the immobilization due to its ease of realization, and for the possibility of electrical addressing. With the use of EIS technique we can evaluate the recognition event, and check the rest of the processes modifying the electron transfer kinetics of the redox probe at the electrode interface. In addition, the described aptasensor showed a reasonable range of response (from 1.67 μM to 5 μM) with a Lower Detection Limit (LOD) of 1.67 μM [5]. The calculated LOD is lower than the maximum amount allowed to be added in wine as prescribed by the International Organization of Vine and Wine (OIV), allowing the determination in this field. The interference produced by BSA showed distorted signal that probably may be solved using stronger blocking

agent. Unfortunately, thermal wet regeneration of the biosensor was not effective for more than twice, impeding its repeated use. Finally, the aptasensor developed in this research could be applied for the detection of lysozyme in a complex matrix such as wine, providing satisfactory recovery yields of about 77%, showing the great potential of the proposed methodology for detecting Lys in other food matrices.

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