RESEARCH PAPER

Nanosized IrO$_2$ electrocatalysts for oxygen evolution reaction in an SPE electrolyzer

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Abstract Nanosized IrO$_2$ electrocatalysts ($d \sim$ 7–9 nm) with specific surface area up to 100 m^2 g^{-1} were synthesized and characterized for the oxygen evolution reaction in a solid polymer electrolyte (SPE) electrolyzer. The catalysts were prepared by a colloidal method in aqueous solution and a subsequent thermal treatment. An iridium hydroxide hydrate precursor was obtained at ~ 100 °C, which was, successively, calcined at different temperatures from 200 to 500 °C. The physico-chemical characterization was carried out by X-ray diffraction (XRD), thermogravimetry–differential scanning calorimetry (TG–DSC) and transmission electron microscopy (TEM). IrO$_2$ catalysts were sprayed onto a Nafion 115 membrane up to a loading of 3 mg cm^{-2}. A Pt catalyst was used at the cathode compartment with a loading of 0.6 mg cm^{-2}. The electrochemical activity for water electrolysis of the membrane-electrode assemblies (MEAs) was investigated in a single cell SPE electrolyzer by steady-state polarization curves, impedance spectroscopy and chrono-amperometric measurements. A maximum current density of 1.3 A cm^{-2} was obtained at 1.8 V and 80 °C for the IrO$_2$ catalyst calcined at 400 °C for 1 h. A stable performance was recorded in single cell for this anode catalyst at 80 °C. The suitable catalytic activity and stability of the most performing catalyst were interpreted in terms of proper combination between nanostructure and suitable morphology.

Keywords Oxygen evolution reaction · Water electrolysis · IrO$_2$ catalyst · SPE electrolyzer · Synthesis

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Introduction

Water electrolysis is one of the few processes where hydrogen can be produced from renewable energy sources such as the photovoltaic or wind energy without evolution of CO$_2$. In particular, a solid polymer electrolyte (SPE) electrolyzer is considered as a promising methodology for producing hydrogen as an alternative to the conventional alkaline water electrolysis. A PEM electrolyzer possesses certain advantages compared with the classical alkaline process in terms of simplicity, high energy efficiency

and specific production capacity. This system utilizes the well known technology of fuel cells based on proton conducting solid electrolytes. Unfortunately, electrochemical water splitting is associated with substantial energy loss, mainly due to the high over-potentials at the oxygen-evolving anode. It is therefore important to find the optimal oxygen-evolving electro-catalyst in order to minimize the energy loss.

Typically, platinum is used at the cathode for the hydrogen evolution reaction (HER) and iridium or ruthenium oxides are used at the anode for the oxygen evolution reaction (OER) (Ardizzone et al. 1990; Da Silva et al. 1997). These metal oxides are required, compared to the metallic platinum, because they offer a high activity, a better long-term stability and less efficiency losses due to corrosion or poisoning (Andolfatto et al. 1994; Millet et al. 1996; Yamaguchi et al. 1997; Ledjeff et al. 1995; Rasten et al. 2003; Slavcheva et al. 2007; Hu et al. 2004; Song et al. 2008; Nanni et al. 1999; De Oliveira-Sousa et al. 2000).

A high surface area is required for the anode electrocatalyst to increase the number of catalytic sites involved in the reaction. Accordingly, most preparation methods aim at obtaining nanosized primary particles. Moreover, being the oxygen evolution process a structure sensitive reaction, it is important to tailor the structural properties which determine the turnover frequency of the catalytic process.

This work is mainly addressed to the synthesis and characterization of nanosized IrO_2 catalysts. Various methods are reported in the literature for the synthesis of noble metal based oxides. The Adams fusion method (Adams and Shriner 1923) has been widely used to produce fine noble metal oxide powders (Rasten 2001; Rasten et al. 1991; Marshall et al. 2003; Hutchings et al. 1984; Song et al. 2008); it is based on the oxidation of metal precursors in a molten nitrate melt. Sol–gel methods have also proved useful in producing noble metal based oxides (Murakami et al. 1994a, b; Ito et al. 1994; Kameyama et al. 1993; Lassali et al. 2000). A very interesting approach was proposed by Marshall et al. (2005, 2006); it was based on a modified polyol method in which a glycol-precursor solution was heated under nitrogen atmosphere to the refluxing temperature in order to obtain a colloid. Also Song et al. (2008) used this approach to synthesize IrO_2 catalysts with a

specific surface area of $203 \ m^2 \ g^{-1}$. In our work, nanosized IrO_2 catalysts were prepared from $H_2IrCl_6 \cdot nH_2O$ using a similar colloidal process; yet, in order to make it economically competitive and easily applicable, water was used as the solvent instead of ethylene glycol (Marshall et al. 2005, 2006) and the process was simply carried out under air atmosphere at 100 °C. The resulting hydroxides were then calcined at various temperatures to tailor the structural properties. By using this approach nanosized crystalline particles in the range of 7–9 nm were obtained, allowing the attainment of a good compromise between catalytic activity and stability (Ma et al. 2006). We focused our attention on the effect of thermal treatments on the crystallographic structure and particle size of IrO_2 catalysts and how these properties may influence the performance of oxygen evolution electrode. Electrochemical characterizations were carried out by polarization curves, impedance spectroscopy and chrono-amperometric measurements.

Experimental

Preparation of IrO_2 electrocatalysts

An $H_2IrCl_6 \cdot nH_2O$ (STREM Chemicals, Inc.) powder was first dissolved in deionised water. The aqueous solution was then heated (100 °C) under air atmosphere and magnetically stirred for 45 min. A sodium hydroxide solution (1 M) was added in order to favour the formation of an Ir-hydroxide. Afterwards, the solution was placed in a centrifuge for 10 min and filtered. The precipitate was washed with deionised water to remove the chlorides. The Ir-hydroxide was dried for 5 h at 80 °C. The dry powder was subjected to a thermal analysis, in order to investigate the processes occurring for the as-prepared catalyst as a function of the temperature. Afterwards, it was calcined in air at different temperatures, 200, 300, 400 and 500 °C for 1 h, using a heating ramp of 5 °C min^{-1}. The resulting powders were characterized by X-ray diffraction (XRD) to determine the crystallographic structure and particle size. Catalyst morphology and porosity were investigated by transmission electron microscopy (TEM) and BET measurements, respectively.

Physico-chemical characterization

XRD was performed on the dry electrocatalytic powders using a Philips X-Pert diffractometer that used as radiation source the Kα line of copper (Cu Kα). This diffractometer operated at 40 kV and 20 mA, step time of 0.5 2θ min^{-1}, angular resolution of 0.005° 2θ. The diffraction patterns were fitted to Joint Committee on Powder Diffraction Standards (JCPDS) and crystalline size was calculated using line broadening analysis (LBA). The TG/DSC analysis was carried out in an STA 409C of NETZSCH—Gerätebau GmbH thermal analysis. The sample was heated from room temperature up to 550 °C at a heating rate of 5 °C min^{-1} under air atmosphere.

The morphology of the in-house prepared catalyst was investigated by TEM using a Philips CM12 instrument and compared to a commercial catalyst (IrO$_2$ from SPECTRUM). Specimens were prepared by ultrasonic dispersion of the catalysts in isopropyl alcohol and depositing a drop of suspension on a carbon-coated grid.

BET surface area, pore size distribution and pore volume characteristics for the in-house prepared catalyst were measured by a Thermoquest 1990 series Sorptomatic. The Barrett–Joiner–Halenda (BJH) method ($C = 0.8$) was used for the determination of cumulative area and pore size. Since the aim of this work was to obtain a nanosized catalyst characterized by high surface area, these properties were compared to those of a commercial catalyst to assess the progress beyond the state of art.

Preparation of membrane-electrode assembly

A Nafion 115 (Ion Power) membrane was used as the SPE. The IrO$_2$ catalysts were directly deposited onto one side of the Nafion 115 by a spray technique. Inks were composed of aqueous dispersions of catalyst, deionised water, Nafion® solution (5% Aldrich) and ethanol (Carlo Erba); the anode catalyst loading was 3 mg cm^{-2}. A Ti/Pt (95:5) grid was used as backing layer. A commercial 30% Pt/Vulcan XC-72 (E-TEK, PEMEAS, Boston, USA) was used as the catalyst for the H$_2$ evolution. The cathode electrode was prepared by directly mixing in an ultrasonic bath a suspension of Nafion ionomer in water with the catalyst powder. The obtained paste was spread on carbon cloth backings (GDL ELAT from E-TEK) with a Pt loading of

0.6 mg cm^{-2}. The ionomer content in both electrodes was 33 wt% in the catalytic layer after drying.

Membrane-electrode assemblies (MEAs) (5 cm^2 geometrical area) were directly prepared in the cell housing by tightening at 9 N m using a dynamometric wrench.

Electrochemical characterization of MEA

The SPE electrolyzer performance was evaluated at 80 °C under atmospheric pressure. Heated deionised water, which was circulated by a pump at a flow rate of 2 mL min^{-1}, was supplied to the anode compartment. The water temperature was maintained at the same cell temperature. Steady-state polarization measurements under potentiostatic control, electrochemical impedance spectroscopy (EIS) and stability tests at 1.6 V and 80 °C were carried out by an Autolab PGSTAT 302 Potentiostat/Galvanostat equipped with a 20 A booster (Metrohm) and an FRA. The impedance measurements were performed under potentiostatic control in a frequency range between 20 kHz and 0.1 Hz by frequency sweeping in the single sine mode. The amplitude of the sinusoidal excitation signal was 0.01 V. The series resistance was determined from the high frequency intercept on the real axis in the Nyquist plot. The charge transfer resistance was taken as the difference between the extrapolated low frequency intercept and the high frequency intercept on the real axis.

Results and discussion

In order to investigate the influence of temperature on the precursor modifications, a thermal analysis was carried out. The results of TGA and DSC measurements are shown in Fig. 1.

The first weight loss (I) was assigned to physical dehydration (below 140 °C); whereas, the second one (II) was attributed to chemical dehydration (in the range 140–240 °C). A phase transition of IrO$_2$ from amorphous to crystalline structure (III) was observed in the range 240–480 °C as confirmed by XRD. This was also accompanied by a slight weight loss due to the decomposition of hydrophilic groups of the amorphous material. Above 480 °C (IV) a slight oxygen loss was recorded, possibly forming a
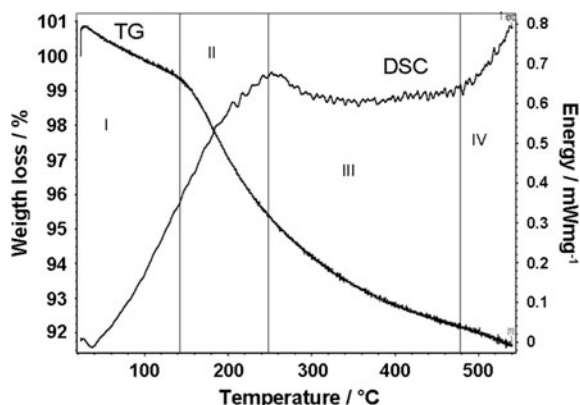
Fig. 1 TGA and DSC curves of the as-synthesized IrO$_x$ precursor

substoichiometric oxide. Total weight losses of 8 wt% were observed after the treatment at 550 °C.

Figure 2 shows XRD patterns of the precursor powder before and after calcination at 200, 300, 400 and 500 °C. The precursor and samples calcined at 200 and 300 °C were characterized by an amorphous phase whereas those calcined at 400 and 500 °C showed a crystalline phase with a tetragonal structure. The mean crystallite size for the powder calcined at 400 and 500 °C was estimated from the broadening of main peaks by the Debye-Scherrer equation; it was 7 and 9 nm, respectively. No presence of metallic Ir was found in all samples. Previous studies carried out for RuO$_2$ showed that this range of crystallite size is appropriate in terms of stability (Ma et al. 2006).

All calcined samples were investigated electrochemically for the OER in an SPE electrolyzer. A commercial IrO$_2$ (SPECTRUM) catalyst was also

studied for comparison. This catalyst is mainly characterized by an amorphous structure with the presence of a small amount of metallic Ir, as reported elsewhere (D'Urso et al. 2007; Di Blasi et al. 2009). Being the hydrogen evolution at the Pt/C electrode quite faster than the oxygen evolution at the IrO$_2$ catalyst (Antonucci et al. 2008), the latter represents the rate determining step of the overall electrochemical process. Furthermore, the same Pt/C cathode was used in all experiments.

Figure 3 shows I–V curves for the in-house prepared and commercial anode catalysts. The best performance at high current densities for the OER at 80 °C was obtained for IrO$_2$ powder calcined at 400 °C. The cell based on the commercial Iridium oxide showed a performance similar to the best catalyst up to 1.6 V. The maximum current density reached at 1.8 V with the sample calcined at 400 °C was 1.32 A cm^{-2}. The high performance obtained with this catalyst calcined at 400 °C was probably due to the simultaneous presence of an amorphous and crystalline structure as an optimal compromise between surface area and specific activity (see Fig. 2). A different behaviour in terms of the slope of the I–V curves was also observed for the various samples. To investigate more in depth the electrochemical properties of these materials, impedance spectroscopy measurements were carried out on the different cells at 1.5 V (Fig. 4). From this analysis, it appears that the sample calcined at 400 °C showed both low series (Rs) and charge transfer (Rct) resistance compared to the other samples; this justify the best performance of the cell based on this catalyst.

Fig. 2 X-ray diffraction patterns of IrO$_2$ powders, as-synthesized and calcined at various temperatures
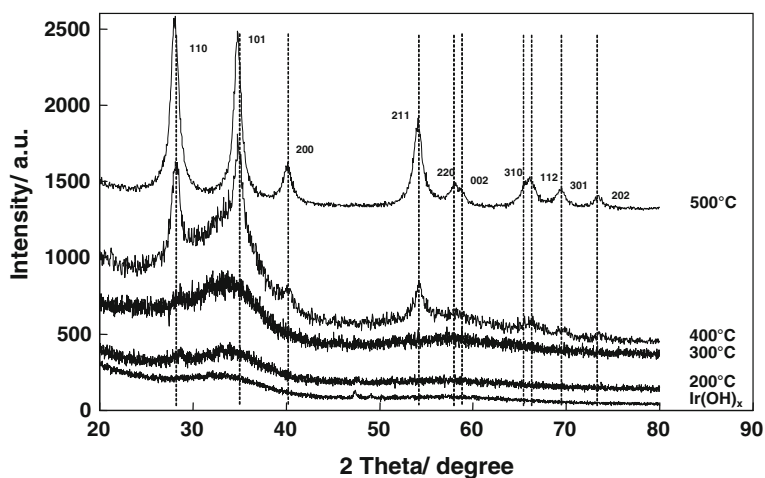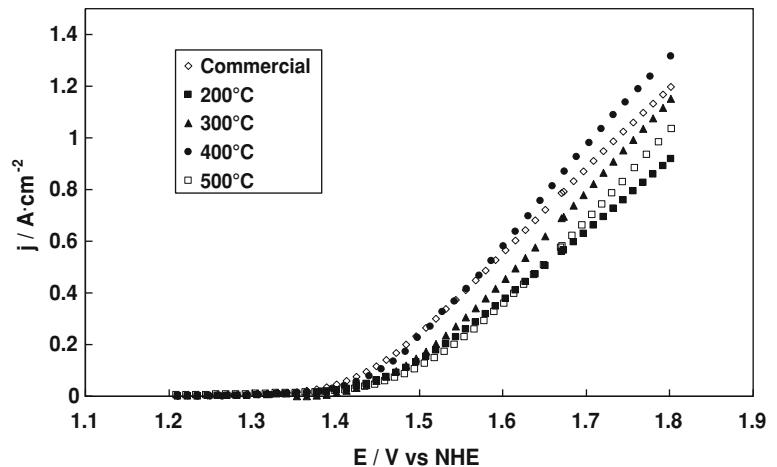
Fig. 3 Polarization curves at 80 °C for the SPE electrolyzer based on IrO_2 powders, commercial and calcined at different temperatures

The cells based on the other in-house prepared catalysts showed a high Rct, indicating a low electrocatalytic activity for the oxygen evolution process. As discussed above, this was interpreted in terms of poor intrinsic activity or low active surface area as related to the structure and particle size. The cell based on the commercial catalyst showed lower charge transfer resistance compared to the sample calcined at 400 °C, but larger series resistance possibly due to the amorphous structure similar to the samples treated at 200 and 300 °C. Notably, an increase of series resistance is observed for the samples with crystalline structure treated at 500 °C. The thermal analysis indicated for this sample an additional loss of oxygen and the onset of an exothermic peak above 450 °C indicating the occurrence of structural changes at high temperatures.

As observed from the I–V curves, the onset potential in terms of oxygen evolution for the commercial catalyst is slightly lower than that obtained for the best in-house catalyst. Yet, the latter showed lower series resistance than commercial catalyst. This explains the best performance of the in-house catalyst, in particular at high current densities. In fact, the slope of the I–V curves at high currents for the two catalysts reflects the differences in ohmic drop (Rs). Moreover, from the polarization curves of Fig. 3, higher diffusion constraints were observed for the commercial catalyst. This could be due to the different morphology of the two catalysts.

A TEM analysis was carried out on these samples (Fig. 5). The commercial catalyst (Fig. 5a) showed the presence of agglomerates composed of fine

particles attributed to the amorphous IrO_2 and some big particles probably due to the presence of metallic Ir. The BET surface area for this catalyst was about 23 m^2 g^{-1}. This catalyst appears characterized by the occurrence of micro-pores as confirmed by the porosity measurements reported in Fig. 6a. On the contrary of the commercial catalyst, the in-house prepared catalyst calcined at 400 °C (Fig. 5b) showed larger particles and the prevailing occurrence of meso- and macro-pores (Fig. 6b). This may explain the lower mass transfer polarization of the cell based on this catalyst. The BET surface area for the in-house prepared catalyst calcined at 400 °C was about 100 m^2 g^{-1}. This was significantly larger than that measured for the commercial one (23 m^2 g^{-1}),
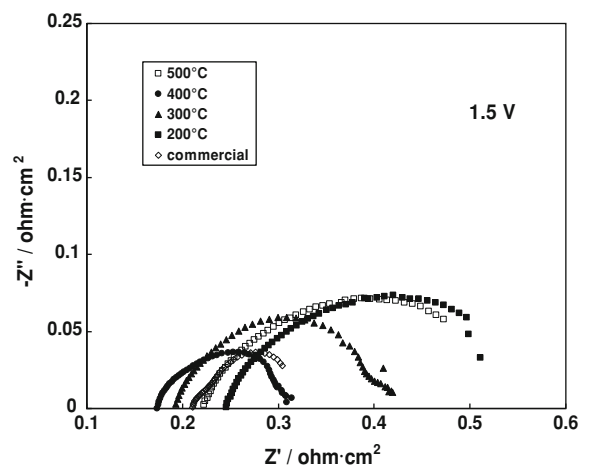

Fig. 4 Impedance spectroscopy at 80 °C and 1.5 V of the SPE electrolyzer based on IrO_2 electrocatalysts, commercial and calcined at different temperatures
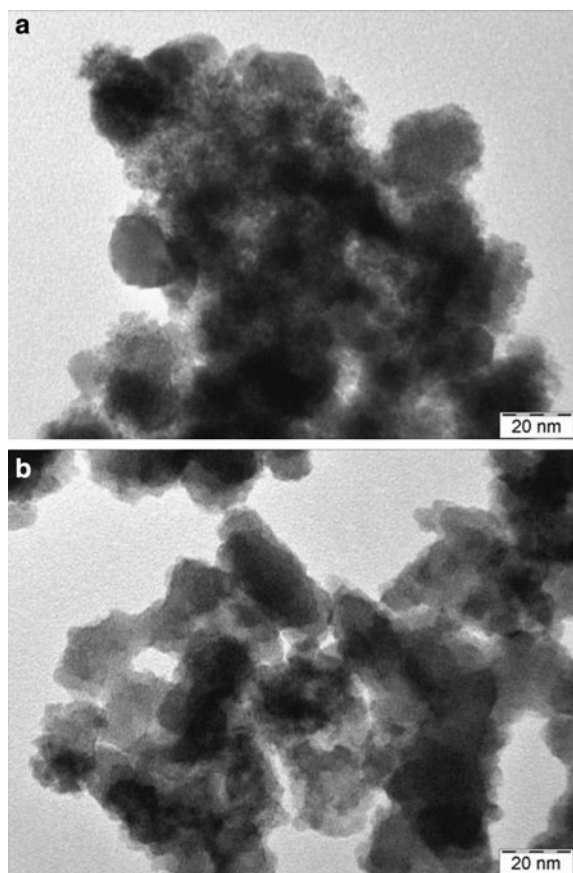
Fig. 5 TEM images of **a** IrO$_2$ calcined at 400 °C and **b** commercial IrO$_2$

Fig. 6 Pore size distribution and cumulative pore volume of **a** commercial IrO$_2$ and **b** IrO$_2$ calcined at 400 °C

although it was lower than that reported by Song et al. (2008) concerning their IrO$_2$ catalyst obtained by a colloidal method. As well known, the specific surface area is determined by both particle size and dispersion. The occurrence of very fine IrO$_2$ particles as well as accessible pores for the OER is indicative of a large number of catalytic sites in this sample. This confirms the paramount influence of the nanostructure and morphology of the anode catalyst in determining the performance of SPE electrolysers.

The Tafel plots related to the OER at the IrO$_2$ catalyst calcined at 400 °C and commercial catalyst are shown in Fig. 7. These curves were corrected for the ohmic drop (iR) to extract electro-kinetic parameters. The same Tafel slopes were identified for the two catalysts. The value was about 80 mV dec^{-1}, which reflects a Temkin adsorption condition (Aricò et al. 1993).

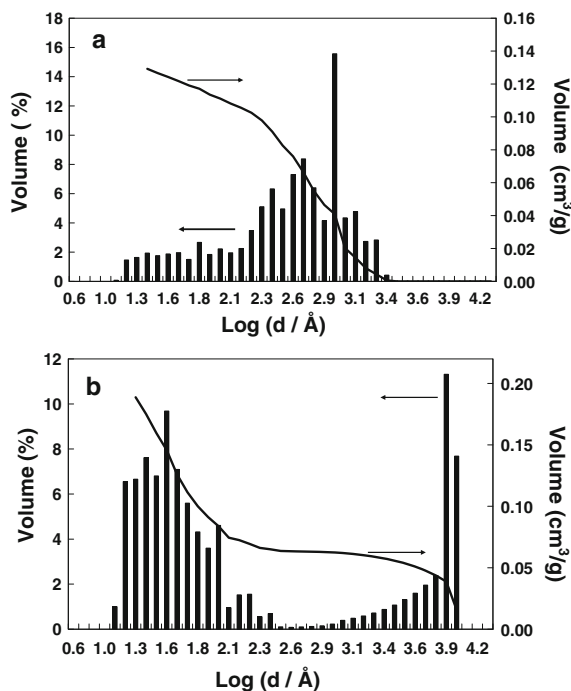As envisaged from the ac-impedance results, the ohmic resistance at low overpotentials (1.5 V)

contributes to more than 50% to the overall process for the best performing in-house catalyst. This aspect is more significant at high currents. On such basis, appropriate increase in performance at practical current densities can be achieved by using thinner or better proton conducting membranes than conventional ones.

Figure 8 is related to the chrono-amperometric measurement carried out at 80 °C and atmospheric pressure for the IrO$_2$ catalyst calcined at 400 °C. The test was performed at a potential of 1.6 V for a period of 20 h. The current density remained constant throughout the test, at 460 mA cm^{-2}, indicating a stable behaviour of the in-house prepared IrO$_2$ electrocatalyst.

Conclusions

A cheap, simple and fast low temperature colloidal method was used for obtaining nanosized IrO$_2$ catalysts characterized by a good compromise between catalytic activity and electrochemical stability. These properties were achieved by a proper combination of nanostructure and morphology. A mean crystallite size

Fig. 7 Tafel plots for oxygen evolution reaction in an SPE electrolyzer at 80 °C for the IrO_2 powder calcined at 400 °C (1 h) and the commercial one
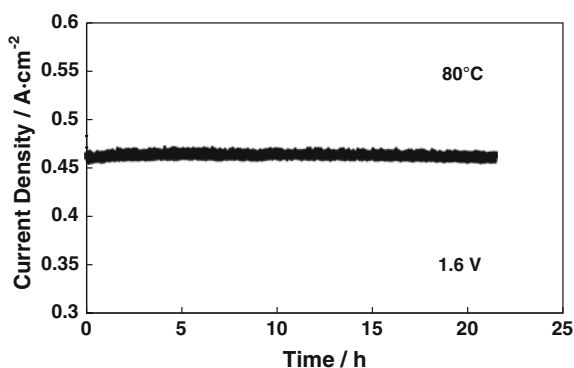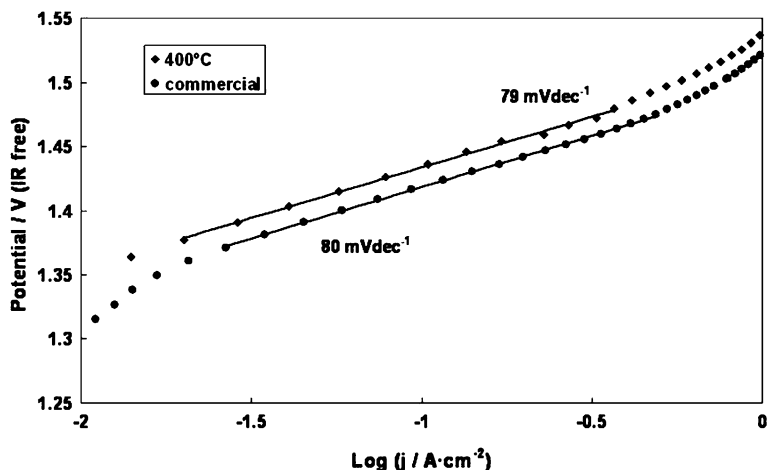

Fig. 8 Chrono-amperometric measurement of the SPE electrolyzer based on IrO_2 calcined at 400 °C/1 h

of 7 nm, a suitable morphology and a BET surface area of 100 m^2 g^{-1} were obtained for the best electrocatalyst. The preparation method consisted in a colloidal process carried out in aqueous solution at ~ 100 °C and subsequent calcination at temperatures varying from 200 to 500 °C. The largest current density in an SPE electrolyzer at practical voltages (above 1.6 V) was obtained for the IrO_2 calcined at 400 °C for 1 h. In terms of electrochemical activity, the best anode catalyst achieved 1.32 A cm^{-2} at 1.8 V in an SPE electrolyzer. Chrono-amperometric measurements demonstrated a suitable stability of the in-house prepared Iridium oxide catalyst for the OER.

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