

Effect of Thermal Treatment on Anode Catalyst for PEM Water Electrolysis

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INTRODUCTION AND EXPERIMENTAL

Anode catalyst in PEM systems can be prepared by different methods. In this work, a spraying method has been chosen, and the catalysts therefore have to be prepared as powders. Oxide particles of Ir are prepared by the Adams fusion method, which consists of heating $\text{H}_2\text{IrCl}_6 \cdot 6\text{H}_2\text{O}$ (Alfa Aesar) in excess of sodium nitrate[1].

The membrane electrode assemblies (MEAs) were prepared by spraying a slurry of catalyst particles, Nafion[®] ionomer and solvent on to a Nafion[®] 115 membrane. The reference $\text{Hg}/\text{Hg}_2\text{SO}_4$ electrode was made from a glass capillary filled with wet Nafion membrane material. The specimens were examined in a Jeol 2010 TEM operating at 200 keV.

RESULTS AND DISCUSSION

TEM- and diffraction images show that the average particle size of not-annealed sample was in the range 5-20 nm and for samples annealed at 490°C about 20-100 nm. When comparing the TEM images of the not-annealed particles and the annealed particles it is observed that crystal growth has occurred and the annealed particles consists of small crystallites. The diffraction image of the not-annealed sample indicates an amorphous phase. Crystalline particles were found only in a few small particles (< 10 nm) for the not-annealed samples, whereas for the annealed sample also the large particles had become crystalline.

The current-potential behaviour of electrodes made from the different catalyst samples, is shown in figure 1. The potential contains part of the IR-drop in the membrane, the lateral IR-drop in the catalyst layer and the oxygen overpotential. The catalyst annealed at 490°C exhibits the lowest potentials and, thus, the best performance. The samples annealed at 510°C and 540°C and 8 hours shows decreasing performance by increasing temperature.

From the voltammetric curves we observed that the not-annealed samples exhibited the largest area within the curve in the oxygen region. This points to a high electrocatalytic activity and/or a high surface area. These samples however correspond to the highest potentials in the polarization curves. This can be explained by the impedance curves in fig. 2. The not-annealed sample exhibits the highest ohmic resistance, defined by the high frequency intercept on the real axis and the highest total polarization resistance, defined by the low frequency intercept. On the other hand, the diameter of the semi circle is the lowest, indicating the highest catalytic activity.

The reason for this behaviour must be explained by two processes occurring during the annealing stage; particle growth and crystallization and by the fact that

small particles crystallize easier than large particles. Lodi *et al.* [2] studied the microstructure and electrical properties of IrO_2 and found that the electrical resistivity decreased by increasing annealing time and temperature and that the particle size increased by increasing temperature. The electrical resistance was believed to depend on both crystallinity and particle size (smaller particles provide more particle boundaries). At 490°C, a low ohmic resistance has been obtained without sacrificing active surface area by particle coarsening, as indicated by the AC-impedance and the area of the voltammograms. The good total performance here indicates a reasonable compromise between surface area and ohmic resistance.

REFERENCES

1. R. Adams and R. L. Shriner, *The Journal of the American Chemical Society*, **45**, p. 2171 (1923)
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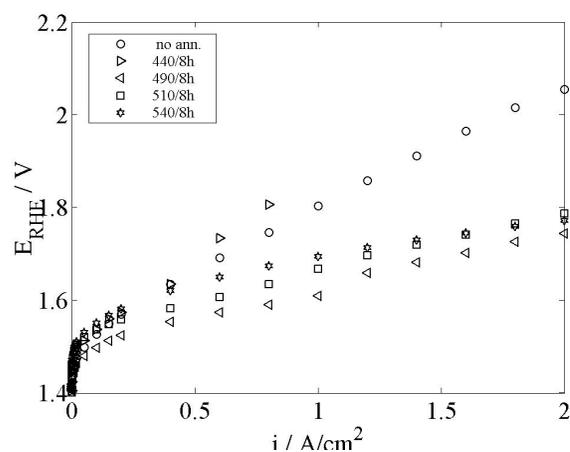


Figure 1. Steady state polarization curves.

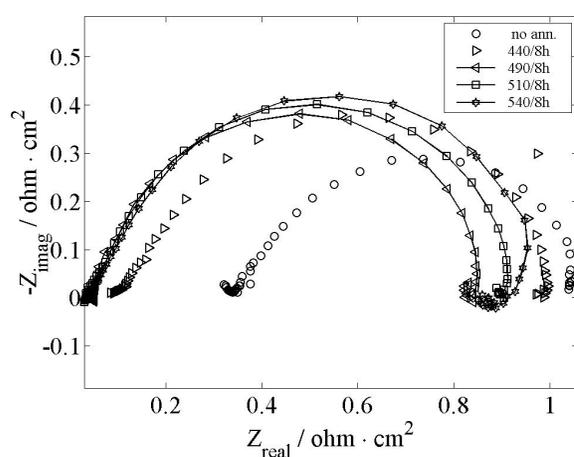


Figure 2. AC impedance.