Microwave Synthesis of Ru_3Pd_6Pt as Cathode in PEM Fuel Cells

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Abstract

In this work is presented a synthesis and characterization of a nanometric and highly dispersed material. The Ru_3Pd_6Pt was produced by microwaves assisted, using the polyol method. The particle size was approximately 9 nm, obtained by xrd data and estimated by tem micrographics. The kinetic parameters obtained shown that a Ru_3Pd_6Pt is an acceptable candidate by using in PEM fuel cells.

Introduction

Historically, the synthesis methods have involved high temperatures and relative high pressures, a long time for synthesis, was used organic solvents that requires an extraction and purification steps. To date synthesis arises as promising as being able to finalize the process in minutes, achieving an efficient way greatly reduce environmental impact. Because microwave energy can be transferred directly to the reactive species, the reactions can be carried out in shorter times than would normally be by employing other methods.

Experimental

Electrocatalyst preparation

The trimetallic electrocatalyst was produced on a conventional microwave oven by polyol method at 190°C by the reduction of corresponding metallic salts $RuCl_3$ (Aldrich), $PdCl_2$ (Aldrich), and H_2PtCl_6 (Aldrich) in ethylene glycol (EG/Aldrich), as reported in literature.[1-5] The system employed its shown in the *Figure 1*. The reaction product was washed several times with acetone and deionized water. Afterwards, the powders was dried at 50°C for 16h and kept in a closed vessel. The resulted dark-fine powder was used for electrochemical measurements [6].

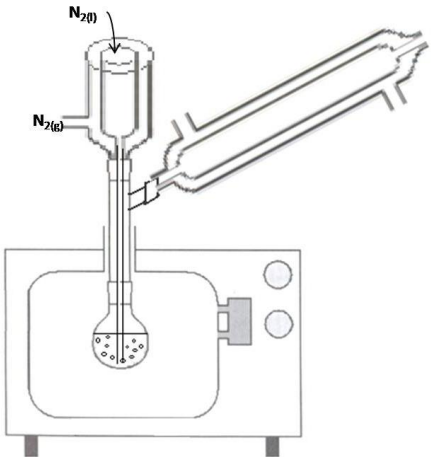

Figure 1. System employed in the synthesis of Ru_3Pd_6Pt

Electrochemical Characterization

Diverse techniques were used to determine the kinetic parameters of the orr on Ru_3Pd_6Pt. Was used the common half cell with three electrodes as working electrode a glassy carbon disc electrode, as counter electrode a platinum mesh and a $Hg_2/Hg_2SO_4/H_2SO_4$ 0.5 M (0.68 V/NHE) as reference electrode. Al measurements are referenced to NHE. The electrochemical surface was determined by the CO_2 stripping technique, at 0.1 V/NHE by the saturation with CO_2 forward by the saturation with N_2. Then rde technique was applied. The hydrodynamic experiments were recorded in the rotation rate range of 100–1600 rpm at 5 mV s^{-1}. All the experiments were performed at room temperature (24°C). Afterward, the corresponding treatment was performed to known the kinetic parameters.

Results and discussion

Physical Characterization

The X-ray diffraction of the Ru_3Pd_6Pt catalyst is shown in *Figure 2.* The powder electrocatalyst showed five diffraction peaks good definite at about 2θ=40°, 47°, 67°, 81° and 87° identified as single hexagonal fcc phase of palladium and in the same range for the single hexagonal fcc platinum. The experimental pattern matches well with the standard crystallographic tables JCPDS cards 01-065-2867 and JCPD 01-065-2868 for Pd and Pt, respectively. The pick on the 43° is attributed to Ru presence. Using Topas Academic Software a particle size was determined around 9 nm.

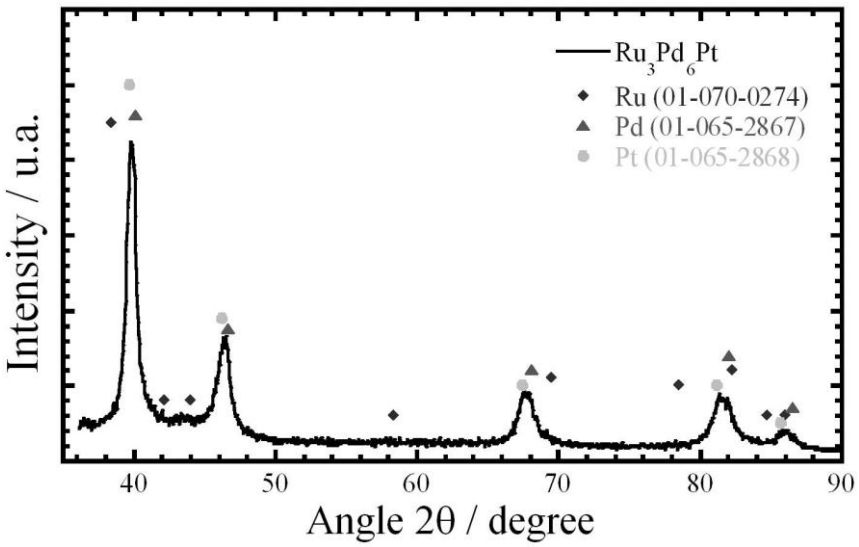

Figure 2. XRD pattern of nanosized particles of Ru_3Pd_6Pt

A transmission electronic micrograph image was shown in a *Figure 3*. A crystalline form immerse in an amorphous part. Analyzing the image in Digital Micrograph 5.0 an estimate of a particle size was determinate around 7 nm with spherical form.

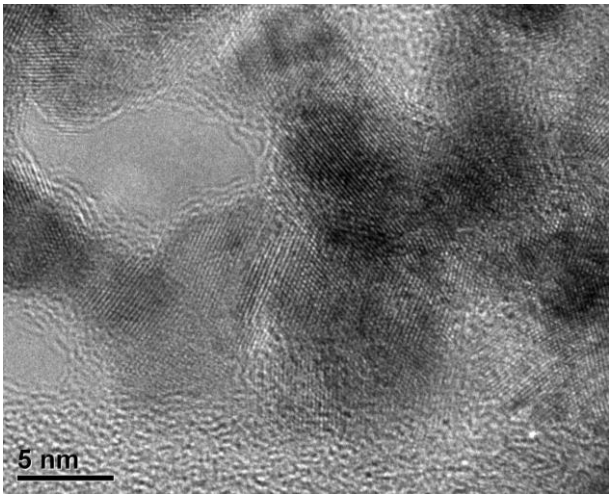

Figure 3. TEM image for Ru_3Pd_6Pt

Electrochemical Characterization

The cyclic voltammetry characterization of the Ru_3Pd_6Pt electrode in the supporting electrolyte was performed in a nitrogen purged H_2SO_4 0.5 M solution, at $100mVs^{-1}$ scan rate. In this experiment the electrode was submitted to 30 cycles in order to obtain a reproducible voltammogram. *Figure 4* presents a corresponding

voltammograms on Ru_3Pd_6Pt oxygen atmosphere that no shows good-definite peaks associated with adsorption/desorption of hydrogen characteristic in polycrystalline noble metals.

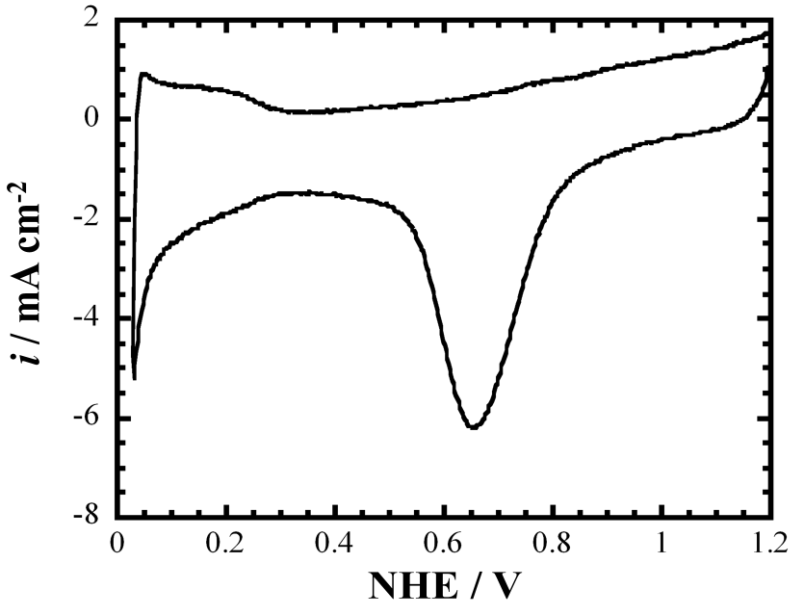

Figure 4. Voltammogram on Ru_3Pd_6Pt in H_2SO_4 0.5 M at room temperature, in oxygenated atmosphere. *v*=100 $mVs^{-1.}$

The CO_2 stripping on Ru_3Pd_6Pt as shown in a *figure 5*. Obtained at 0.78 V/NHE the scan rate was 20 mV s^{-1}. On the *figure 6* is presented a voltammograms obtained after and before de CO_2 stripping technique. Is observed that the characteristic shape no change, that is a good indicator that the material is CO_2 resistant. The area was estimated around a 1.445 cm^2 with a R_f=11.5.

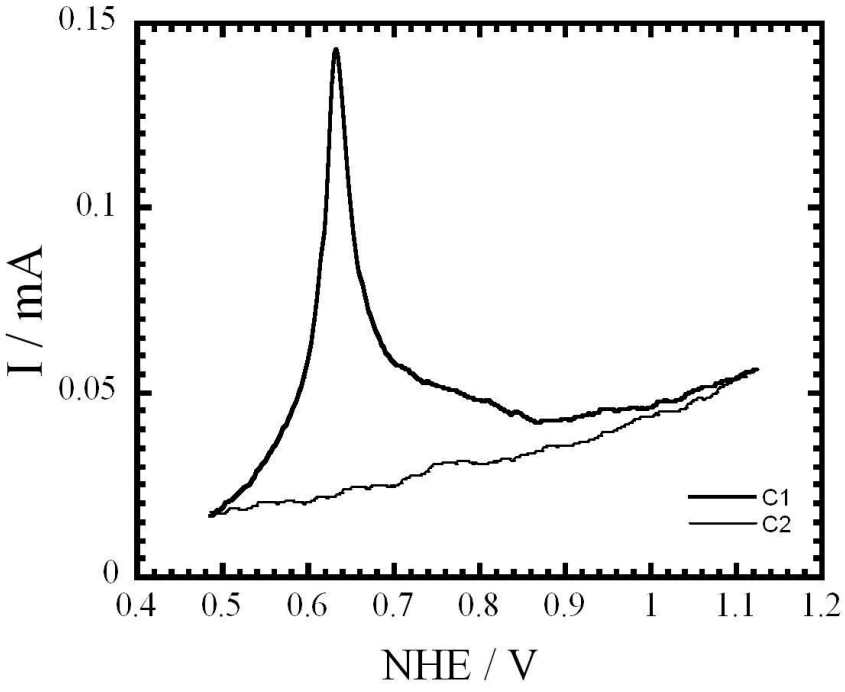

Figure 5. CO_2 stripping on Ru_3Pd_6Pt at room temperature, E=0.78 V. v=20mv s^{-1}.

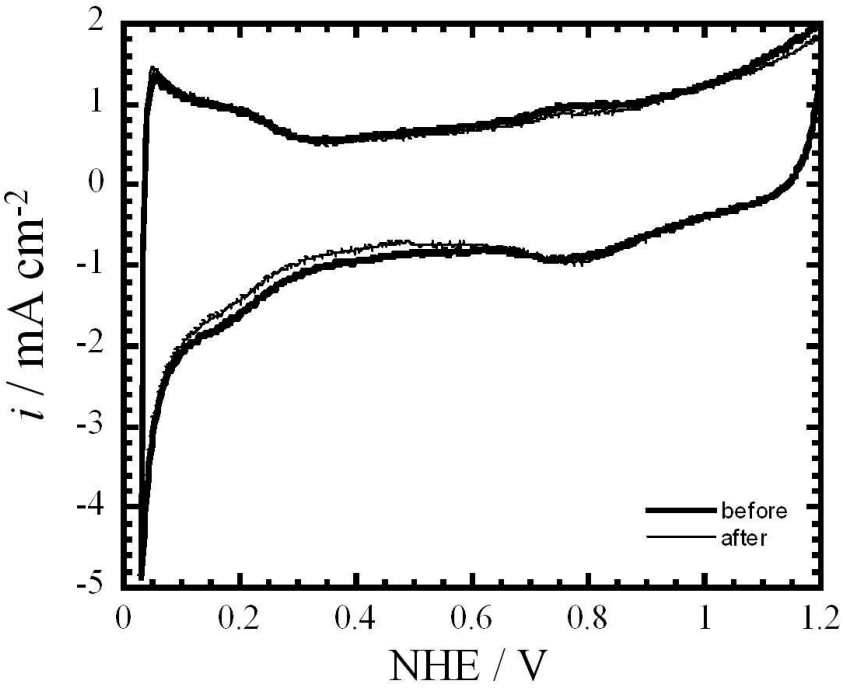

Figure 6. Ciclic Voltammograms on Ru_3Pd_6Pt, in N_2 , before and after the CO_2 stripping. v=100 mv s^{-1}.

Afterwards, the edr was obtained and is shown in the Figure 7. The clasic shape are present in this graphics, the kinetic zone, the difussional and the mixed control was observed. The *figure 8* presents a tafel graph obtained from the analysis of rde. Is observed that the material exhibit a good activity to catalyze the orr on acid media.

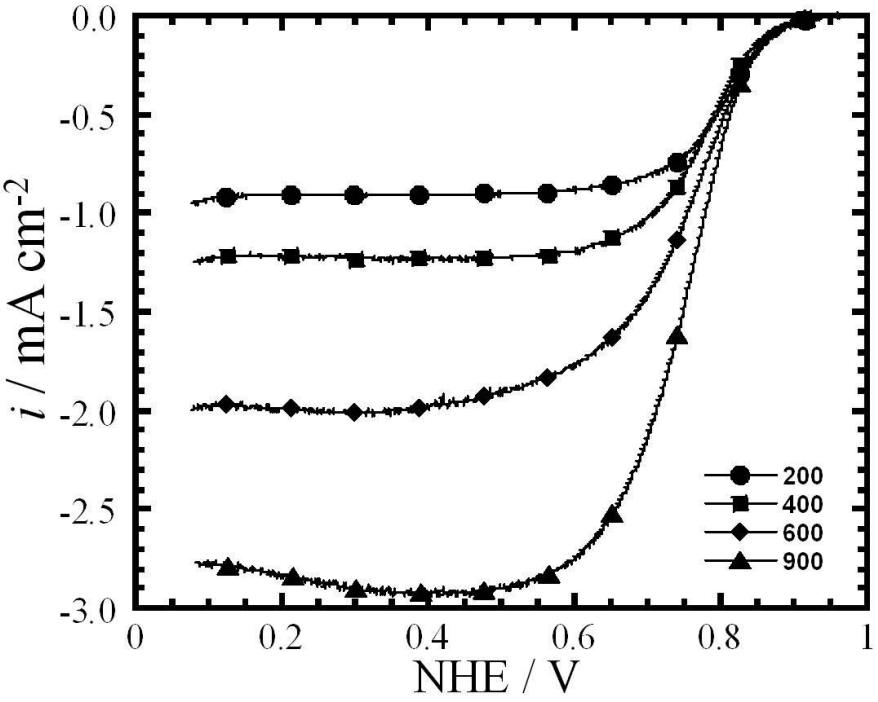

Figure 7. rde graph for orr on Ru_3Pd_6Pt, at room temperature. v =5 mv s^{-1}.

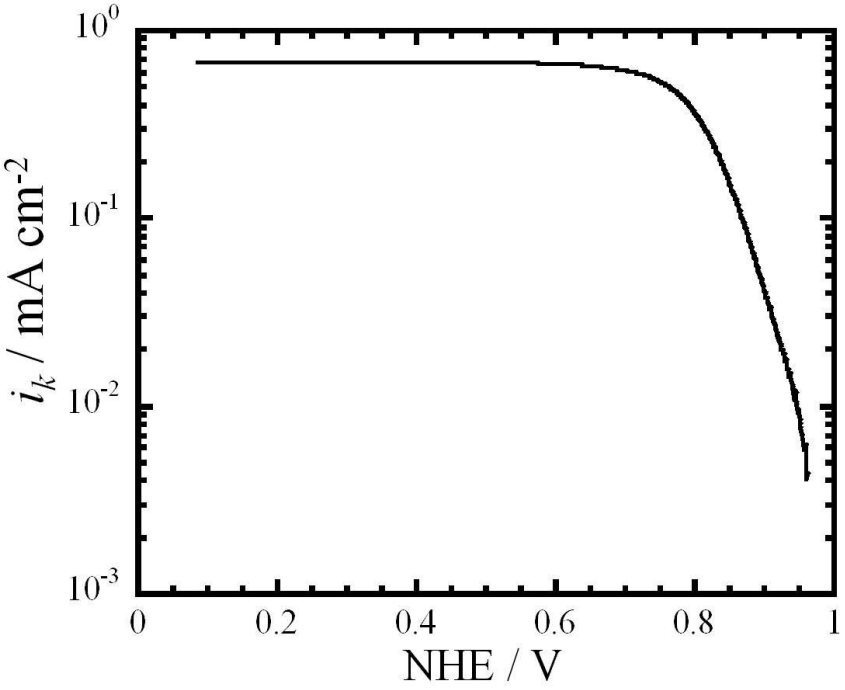

Figure 8. A Tafel plot of Ru_3Pd_6Pt for the orr on H_2SO_4 0.5 M at room temperature.

Conclusions

The kinetic parameters were obtained from the tafel plot. The open circuit potential that exhibit this material was 0.95 ± 0.02. the area calculated from the CO_2 stripping determined from the figure 5 was 1.445 cm^2, tafel slope (-*b*), current density exchange (i_0) and α parameter, was 0.56 ± 0.09 mV dec^{-1}, (9.07 ±1.3) x10^{-5} mA cm-^{2}, respectively. The Ru_3Pd_6Pt material is candidate to be considered as cathodic catalyst for polymer electrolyte membrane fuel cell (pemfc).

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References

[1] Y. N. Wu, *J. Power Sources,* **194** (2009) 805–810.

[2]W. Hong, D. Rui, C. Hsin, L. Ching, C. Yui; *Mater. Lett*., **61** (2007) 830.

[3]G. Zhao, J. He, C. Zhang, J. Zhou, X. Chen, T. Wang; *J. Phys. Chem. C,* **112** (2008) 1028.

[4]J. Zhao, P. Wang, W. Chen, R. Liu, X. Li, Q. Nie; *J. Power Sources*, **160** (2006) 563.

[5] P. Ochal, *J. Electroanalytical Chemistry,* **655** (2011) 140.

[6] D.C. Martínez, O. Solorza, *J. Power Sources,* **196** (2011) 4468.