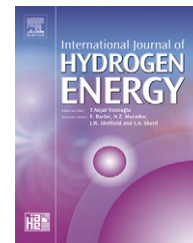


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Electrochemical preparation of a novel, effective and low cost catalytic surface for hydrogen evolution reaction

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ABSTRACT

The formation of platinum nucleus on the freshly polished aluminum (Al) and anodized aluminum electrodes ($\text{Al}_2\text{O}_3/\text{Al}$) was studied by cyclic voltammetry. Results showed that the deposition of platinum on freshly polished aluminum from an aqueous 0.5M phosphate buffer solution containing H_2PtCl_6 takes place rapidly through the electro-reduction of dissolved Pt (IV) ions. At shorter deposition times, small particles of platinum crystals were formed on the aluminum and the surface coverage was imperfect. At longer deposition times, the size of the platinum crystals increases while their number decreases due to the coalescence and agglomeration processes. The electrodeposition of Pt on the Al electrode was conveniently carried out over the $\text{Al}_2\text{O}_3/\text{Al}$ electrode. The electrochemical and catalytic activities of the Pt/Al and Pt/ $\text{Al}_2\text{O}_3/\text{Al}$ electrodes were studied in 0.1M H_2SO_4 solution. In cyclic voltammetry, the two pair symmetric peaks appeared in 0.1M H_2SO_4 solution which was attributed to the formation of strongly (H_s) and weakly bounded hydrogen (H_w). The occurrence of the third anodic hydrogen peak (H_{3rd}) was revealed at low scan rate and in high concentration of H_2SO_4 . At potentials more negative than -0.3V vs. SCE, the current is mainly due to hydrogen evolution reaction. The influence of the various parameters such as deposition method and amount of platinum, sulfuric acid concentration and medium temperature on the hydrogen evolution reaction is described. Finally the kinetic of the hydrogen evolution reaction is also discussed on the Pt/Al electrode.

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1. Introduction

The hydrogen evolution reaction (HER) is an electrochemical process that has received wide attention because of its importance in both fundamental and technological electrochemistry such as fuel cell technology [1–4]. For development of the hydrogen-based fuel cell systems as power sources for emission-free electric vehicles, preparation of an active electrocatalyst is still the main subject. The nature of the electrocatalytic surface plays a decisive role in the kinetics and mechanism of this reaction.

Although platinum presents the highest activity for the HER, the high cost of this material is often very prohibitive. The search for new and less expensive alternative materials for HER has been a topic of current interest [5–17]. Deposition of Pt layers on the less expensive metal materials such as titanium [18], tantalum [19], tungsten [20] or deposition of fine platinum particles on high surface area carbon supports [21,22] and dispersion of the platinum micro particles in polymer matrix [23–29] are one of the workhorses of electrochemical technology that reduced the cost of anode materials in the industrial applications. Several methods have been

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reported for the preparation of platinum-based electrode [30]. Electrodeposition of small clusters of platinum is a simple and relatively cheap method to produce efficient electrocatalysts.

Recently, we have reported that, metallic nickel, cobalt and palladium can easily be cementated on the freshly polished Al [31–33] and Zn [34] electrodes. Subsequently, some transition metal cyanoferrate-based modified Al electrodes were prepared and used for electrocatalysis purposes. However, it is surprising that only minor attention has been devoted to use this very low price substrate for deposition of Pt particles and application of Pt-coated Al electrode in electrocatalysis. G. Kokkinidis and coworkers [35] reported that the platinum plating of aluminum substrate from acidic solution of PtCl_4^{2-} is not possible, because the plating reaction followed by the very fast chemical reaction between aluminum and hydrogen ions and as a result the aluminum layer supporting the platinum film is dissolved in the solution. In the previous paper [36], we have reported that platinum can be deposited on a freshly polished aluminum surface from an aqueous solution of H_2PtCl_6 in phosphate buffer solution saturated with NaF as a result of an electroless reaction taking place at the open-circuit potential. The methanol electrooxidation reaction was used as a probe reaction for testing both the catalytic activity and deposition conditions. The results provided some evidences that the current density of the methanol electrooxidation is affected by the morphology and the size of the platinum particles. In the same paper we described that the deposition of fine platinum particle on the aluminum surface may be achieved by electrolytic procedure.

In the present article we report the study of the HER on electrodeposited platinum on aluminum electrode as a low cost electrocatalyst in sulfuric acid solutions. The influence of various parameters such as deposition method and amount of platinum, sulfuric acid concentration and temperature is studied.

2. Experimental

2.1. Chemicals and reagents

The aluminum bar with purity of 99.9% (purchased from Tabriz Wire & Cable MFC. Co, Simcat, Iran) was used as substrate for the working electrodes construction. H_2PtCl_6 , H_2SO_4 and other chemicals were of analytical grade purchased from Merck. The phosphate buffer solutions were made up from NaH_2PO_4 and adjusted to pH 7 by adding 1M NaOH solution. All solutions were prepared with doubly distilled water.

2.2. Instrumentation

The electrochemical experiments were carried out using an AUTOLAB PGSTAT-30 potentiostat/galvanostat equipped with an USB electrochemical interface and a personal computer. A conventional three-electrode cell was used. The temperature of the cell was varied from 25 to 75 °C by circulation of thermostated water, using a heated water circulator (EYELA-

DIGITAL UNI ACE UA-10). The working electrode was a Pt/Al or Pt/ Al_2O_3 /Al modified electrode (with a diameter of 3 mm). A saturated calomel electrode (SCE) and platinum wire were used as reference and auxiliary electrodes, respectively. The scanning electron microscopy (SEM) was a LIO-40 oxford instrument.

2.3. Procedures

2.3.1. Electrodes preparation

A cylindrical aluminum bar of 90 mm in length and 12 mm in diameter was used for preparation of the aluminum disk electrode as follows:

One end of the Al bar was threaded by means of a suitable screw tap in order to connect it to an available holder or rotating disk system. From other end of the bar, one part (30 mm in length) was ground until its diameter reaches to 3 mm, then was fitted into the hole which was previously made in a Teflon rod of 30 mm in length and 12 mm in diameter [37]. The aluminum surface fitted in Teflon was mechanically polished successively to a mirror finish, first with 200, 600, 1000 and 1500 emery papers and then with 0.05 μm alumina powder. The polished surface was cleaned by dipping in concentrated HCl for about 1 min and rinsed twice with distilled water.

2.3.2. Deposition of platinum particles on the aluminum

In this work the electrolytic deposition of the Pt particles on the Al electrode and characterization of its catalytic activity were carried out as follows:

Two electrochemical cells were used: cell (1) for electrodeposition of Pt particles on a polished Al electrode and cell (2) for testing the catalytic activity of the platinum particles for the HER. The plating solution (cell 1) was an aqueous solution containing 0.5M phosphate buffer + 10^{-3} M H_2PtCl_6 and testing solution (cell 2) was an aqueous solution of 0.1M H_2SO_4 . After polishing and washing, the Al electrode was transferred to the cell (1) to deposit platinum. Electrodeposition of platinum was carried out by a single potential step ($E = -0.6$ V) in nitrogen purged and stirred plating solution. After platinum deposition, the electrode was washed immediately and transferred to cell (2) in order to record the cyclic voltammograms (CVs) corresponding to the Pt-particles and HER which may indicate the presence and catalytic activity of Pt particles.

For electrodeposition of Pt particles on an anodized aluminum (Al_2O_3 /Al) electrode, a similar procedure was utilized as follows: After obtaining the reproducible CV of Al in a cell containing only phosphate buffer solution, the potential was switched to the upper limit ($E = 1.2$ V) for 10 s to grow a thin oxide film. Then, the Al electrode was transferred to the cell (1) without polishing. The next steps were followed as described previously.

The quantity of metallic platinum plated on the Al and Al_2O_3 electrodes surface which depends on the deposition potential and its duration in the potentiostatic method was calculated from electric charge consumed.

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