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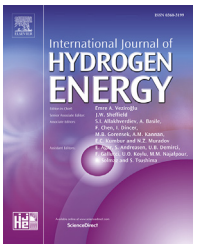

Fe-Cu coated nickel mesh usage as cathode catalyst for hydrogen evolution reaction

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ABSTRACT

Nickel mesh electrodes were used as the working electrode. Iron and copper were electrochemically deposited on the nickel mesh in different amounts. When electrochemical coatings had been carried out, different currents were passed from the circuit at different times and coatings were accumulated at constant load. The prepared electrodes called as FexCux, FexCu3x and FexCu9x and these electrodes have been used for hydrogen evolution reaction (HER). The surface morphologies were investigated by scanning electron microscopy. The HER activity is assessed by recording cathodic current–potential curves, cyclic voltammetry, electrochemical impedance spectroscopy. The results show that FexCu9x catalysts have a compact and porous structure as well as good electrocatalytic activity for the HER in alkaline media.

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Introduction

Energy is the most important notion of the 21st century [1]. With the decline of primary energy resources over time and the increase in energy demand, the search for different energy sources has increased [2]. In addition, scientists have turned to green energy sources because of the global warming and climate change that has begun in the world. Among green energy sources, hydrogen is a species with a high potential for sustainable energy [3,4]. Hydrogen is one of the most perspective fuels of energy. The basic advantages of hydrogen are high calorie value and abundant in the environment [2]. Furthermore, the fact that the energy density of hydrogen is high and it is a good energy carrier increases the preference of hydrogen [5–7]. Hydrogen can be produced by a variety of methods such as coal gasification, thermolysis, thermal water separation, hydrocarbon steam reforming, biomass pyrolysis, photolysis and electrolysis [8,9]. Because electrolysis is simple and efficient, it is the most used method to produce hydrogen [9–11]. Electrolysis is the decomposition of water into components electrochemically. The hydrogen evolution reaction (HER) formed by electrocatalytic separation of water can provide a sustainable energy support for the future. However, there are problems such as resource constraint and high cost because noble metals are used in this technique [2]. Cathodes containing platinum are often used as catalysts in the hydrogen evolution reaction. However, it is not very preferred for use due to platinum poisoning problem [12]. For this reason, easy to find and cheap materials should be developed to use as electrocatalyst to produce hydrogen. There are many studies related to electrocatalysis development [13–18]. The desired properties of the electrocatalysts are low overvoltage, high exchange current density, high surface area and long-time stability [19–22].

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Nickel and its alloys have intrinsic electrocatalytic properties for hydrogen evolution reaction with suitable corrosion resistance in alkaline environment [23–25]. Nickel-supported electrocatalysts have high activity and low cost compared to other transition metals [26]. As the electrocatalyst, different types of nickel such as Ni-based alloys, Ni-based composites, Ni foam and Ni mesh can be used. Ni mesh is produced for applications such as fuel cells, cathode ray tube, sonic control and UV filtration. In addition, the flexibility of the Ni mesh allows it to be shaped into different forms and placed closer to the anode to reduce ohmic loss [27]. The Ni mesh is used as a current collector especially in different applications [28–30]. For instance, in pyrolysis of ethane [31] and oxidation [32], acetylene pyrolysis [33], methane oxidation [34], direct ethanol fuel cell [35] and solid oxide fuel cells [36]. Iron, copper metals which have high abundance and are not expensive and these alloys, are used as electrocatalysts in different forms [37–50]. Their performance has been well studied but there is no study about nickel mesh supporting material with these alloys.

In this study, unlike the studies in the literature, we aimed to obtain a material with high catalytic properties by co-depositing iron and copper on nickel mesh at different current densities for use in HER. The surface characterization of prepared electrode was done by scanning electron microscopy (SEM) technique. The electrocatalytic activity of the developed electrode for HER is evaluated in 1.0 M KOH solution using polarization curves and the electrochemical impedance spectroscopy (EIS).

Experimental

Nickel mesh electrodes with a thickness of 1.6 mm and a width of 1 cm and a height of 1 cm were used as the based material. Nickel mesh electrode was purchased from MTI Corporation. Its density is 346 g/m^2 and 80–110 Pores per Inch. Average hole diameters about 0.25 mm. Nickel mesh electrodes were awaited in the ultrasonic bath in acetone for half an hour before the electrochemical deposition step to clean the oil and dirt layer on the surface. Then, they were kept in the ultrasonic bath in a 3.0 M HCl acid solution during 1 h to activate the surface. The prepared nickel mesh electrodes were removed from the acid and then washed with ultrapure water. They were immersed in an iron-copper bath to make electrochemical coatings.

The iron copper bath composition is as follows.

Copper bath: 27.72% $CuSO_4 \cdot 5H_2O$, 1.25% H_3BO_3 (w/w) [51].

Iron bath: 30.86% $FeSO_4 \cdot 7H_2O$ and 1.25% H_3BO_3 (w/w) [17].

For each coating process, copper and iron baths are mixed in different volumes (Fe: Cu; 50-50; 25–75 and 10–90 mL, respectively). The coating operations were carried out using the direct current source (TT Technic-YH-303D). Platinum anode with a surface area of 2 cm^2 was used as counter electrode. Iron - Copper was coated on the nickel mesh by a constant charge of 4.350 C to the electrolysis system applying different currents (29, 58, 87, 116 and 145 mA). After deposition, the electrodes were rinsed with distilled water to remove residues of bath chemicals.

The electrochemical characterizations of electrodes were analyzed by EIS, CV, and potentiodynamic polarization techniques. Ag/AgCl and Pt (2 cm^2 surface area) were used as a reference and counter electrodes, respectively. CV analysis was determined at 100 mV s^{-1} scan rate. EIS experiments were obtained by a range of frequencies from 10^6 to 0.01 Hz at 5 mV amplitude. Moreover, potentiodynamic polarization plots were analyzed at 1 mV s^{-1} scan rate at cathodic direction. All experiments were done in 1.00 M KOH solution at room temperature.

SEM (scanning electron microscopy) (FEI Quanta 650 Field Emission SEM) and EDX (energy-dispersive X-ray spectroscopy) (EDAX octane plus) analysis were performed to determine the surface morphology of the coated electrodes. Cyclic voltammetry (CV), electrochemical impedance spectroscopy (EIS) and potentiodynamic polarization curves analysis were performed with the potentiostat-galvanostat device (Gamry Interface 1000) for the electrochemical characterization of the electrodes.

Results and discussion

The SEM images of the coatings made at 87 mA for 100 s are given in Fig. 1. When the SEM images are examined, the formation of copper metal clusters is observed with the increase of the proportion of copper in the cover. The effect of iron in the 1:1 ratio coating is seen. However, with the increase in the amount of copper, iron coatings remained between the copper metals, and this increased the catalytic activity of the alloy electrode.

The EDAX analysis results are shown in Tables 1 and 2. Applying currents of different magnitudes seems to affect the proportion of metals deposited on the electrode surface. Table 2 shows that the amount of copper deposited is high, even when the amounts of copper and iron in the coating bath are the same. It is understood from the EDAX analysis that this is a competing accumulation. The movement of copper ions to the electrode surface is faster than that of iron ions. It is seen that the amount of copper deposited on the surface of the electrode increases with the increase of the amount of added copper.

Fig. 2 shows the CVs of FexCux, FexCu3x and FexCu9x on Nickel mesh support in 1.00 M KOH solution at 298 K. CV peaks are related to Fe/Fe^{2+} (−0.40 V), Cu^+/Cu^{2+} (−0.10 V) and Ni^{2+}/Ni^{3+} (0.53 V) oxidation. The peak of Ni^{2+}/Ni^{3+} transition which is occurred by Ni-mesh based material are dominated by Cu/Cu^{2+} peak at FexCu9x electrode. The cathodic peaks correspond to the Ni^{3+}/Ni^{2+}, Cu^{2+}/Cu^+ and Fe/Fe^{2+} reduction, respectively. In Fig. 2, anodic and cathodic peak intensity at the CVs of the FexCux, FexCu3x and FexCu9x electrodes increase with increasing copper composition on the electrode. Ni^{2+}/Ni^{3+}oxidation peak current value of the FexCu9x electrode is higher than other electrodes.

The Nyquist (a) and Bode (b) curves of FexCux, FexCu3x and FexCu9x electrodes in 1.0 M KOH are seen in Fig. 3. The EIS results were obtained for FexCux, FexCu3x and FexCu9x electrodes as a cathode material with regards to potential usage for hydrogen evolution reaction (HER) in alkaline

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