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Electrodeposition of nano-nickel in deep eutectic solvents for hydrogen evolution reaction in alkaline solution

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ABSTRACT

The electrodeposition of nano-Ni films on a Cu substrate from NiCl₂ precursors in choline chloride (ChCl)/urea or ethylene glycol (EG) based deep eutectic solvent (DES) was illustrated. Voltammetric study demonstrates that the electrodeposition of Ni(II) in both ChCl/urea and ChCl/EG systems is a simple one-step reaction process, and the electrochemical process is a typical diffusion-controlled irreversible process. The obtained Ni films can be controlled to form nanostructures depending on the DES and the electrodeposition conditions. The nanostructured Ni films were investigated as electrodes for hydrogen evolution reaction (HER) in an alkaline medium. The enhanced catalytic activity toward the HER was confirmed, which is mainly attributed to the formation of needles morphology and the improved electrochemical surface area. The result reveals that the electrodeposition process may provide a promising strategy for the production of nano-Ni at low temperature for HER.

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Introduction

Hydrogen has received tremendous attention because of its bright future in the application of replacing traditional fossil fuels [1–3]. Among hydrogen production techniques including water electrolysis, natural gas reforming, coal gasification etc. [4–6], water splitting has been recognized as one of the most promising methods to promote highly pure hydrogen from

sustainable water sources without any pollution [7]. However, this method is restricted directly by its high cost and energy consumption of production. Therefore, searching for low-cost electrocatalysts with good catalytic performance for hydrogen production to reduce energy consumption is crucial for this technique [8]. Several excellent reviews suggested that noble platinum (Pt)-group metals are the most active electrocatalysts for producing hydrogen by water electrolysis which is called hydrogen evolution reaction (HER) as well [9–11].

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Nevertheless, the pervasive application for HER is limited by their high costs and low abundance. Hence, the great challenge in HER is unquestionably that finding earth-abundant non-noble metal electrocatalysts with high catalytic activity replaces Pt-group metals. Much work so far has focus on some non-noble metals/alloys [12,13], nitrides [14], carbides [15], borides [16], phosphides [17], sulfides [18], selenides [19], etc. for HER which have considerable electrocatalytic activity.

Nickel and nickel-based alloy electrodes continue to be a great impetus to the HER due to their relatively low cost, accessibility and relatively high catalytic activity superior to other non-precious electrode materials [20–24], which stands out from these materials. Even so, it should be pointed out that the catalytic properties of Ni for the HER still need to be optimized. Starting from geometric factors, increasing the active surface area of electrode by fabricating nanoporous structure is to improve the electrocatalytic activity of Ni for HER, which is one of most effective methods [25]. It is commonly known that the electrodeposition of Ni has been widely fabricated in aqueous plating systems [26–28]. In recent years, for the electrodeposition of Ni, ionic liquids (ILs) provide an ideal electrolyte since ILs have many advantages such as wide electrochemical windows, high ionic conductivity, extremely low vapor pressure, thermal stability and high solubility of metal salts [29]. Deep eutectic solvents (DESs), initially synthesized and characterized by Abbott et al. [30] in 2003, are considered as a promising media to produce metals due to relatively low cost, stability in air and water, nontoxicity and environmental protection in contrast to most of ILs.

In the early 21st century, the electrodeposition of Ni from choline chloride/urea or ethylene glycol based ILs have been examined by Abbott et al. [31]. The deposition of kinetics, thermodynamics and morphologies are typically different from the aqueous processes. However, there is not detailed information to further study the reduction process of Ni(II). Subsequently, Srivastava et al. [32] also studied the Ni coatings prepared from choline chloride based ILs and from the aqueous solutions. The investigation showed that the corrosion behavior of nickel deposits has nothing to do with the electrolyte type used for deposition. Amanda S. C. Urcezino et al. [33] had reported on the influence of the temperature and of DESs nature on the nucleation and growth processes of Ni coating onto Cu surface in two different systems ChCl:2EG and ChCl:2Urea containing NiCl₂·6H₂O. Then, the corrosion performance of the Ni deposits was researched by linear polarization and electrochemical impedance spectroscopy. Moreover, Kityk A. A. et al. [34] investigated that the density, surface tension, viscosity and conductivity of ethaline by changing Ni(II) salt content in the temperature range of 25–80 °C. Little is known, however, about the detailed comparison of the effects of two ionic liquids (choline chloride/urea or ethylene glycol based ILs) on nickel deposition. In addition, Cao Y et al. [35] reported a procedure to prepare highly-porous Ni film electrode by a facile vertical template-assisted composite electrodeposition method using polystyrene (PS) microspheres templates. The results show that this method increases the electrochemical surface area, which improves the electrocatalytic activity for HER. In spite of this, the two-steps progress of this method is cumbersome and complicated. To sum up, on the one hand, the comparison

of electrodeposited nano-nickel in the two media (choline chloride/urea or ethylene glycol based ILs) has not yet been studied in detail. On the other hand, less research about nanostructured Ni electrodeposited for HER in DESs currently.

In this study, the electrodeposition of nanostructured Ni films from NiCl₂·6H₂O in DESs has been systematically investigated. Furthermore, we examined the comparison between the two media by voltammetric measurements. Then, the as-prepared nano-Ni films were studied as the electrodes for HER in an alkaline medium at room temperature, and the results exhibit promising properties for HER.

Experimental

Electrolyte preparation

To remove the water residue, Choline chloride [HOC₂H₄N(CH₃)₃Cl] (ChCl) (Aldrich, 99%), urea [NH₂CONH₂] (Aldrich, >99%), ethylene glycol (EG) (Aldrich, >99%) were dried under vacuum at 343–363 K for 6 h before use. Moreover, NiCl₂·6H₂O (Aldrich, >99%) and KOH (Aldrich, >99%) were used as received. The eutectic solvents were obtained by stirring 1:2 molar ratio of ChCl and urea (1:2CU) or EG (1:2CE) at 343 K, which is a achromatous, homogeneous ionic liquid finally. And, 0.2 M NiCl₂·6H₂O was respectively added into the two kind of ionic liquids at 343 K to get diaphanous, uniform green liquids later. More details can be found in our previous work [36,37].

Fabrication of Ni coatings

All electrochemical experiments were performed on a Bio-Logic HCP-803 electrochemical workstation. The chronoamperometry (CA) and cyclic voltammetry (CV) experiments were conducted in a 100 mL of cylindrical glass breaker used as the electrolytic cell with a three-electrode system. Before the electrochemical experiments, all the electrodes should require pretreatment process that includes leaching in 5% H₂SO₄ for 1 min, rinsing with distilled water and then washing with anhydrous alcohol and drying finally. During the electrodeposition experiment, a Cu sheet (10 × 10 mm²) was used as working electrode in CA experiments while in CV experiments a Pt wire (0.5 mm in diameter) was used as working electrode, a platinum flake (10 × 10 mm²) was served as counter electrode and a Ag wire (2 mm in diameter) was employed as reference electrode. Fig. 1 shows the schematic illustration of the electrodeposition of Ni films on a Cu substrate from NiCl₂·6H₂O precursor in 1:2CU or 1:2CE ionic liquid. The as-prepared samples were washed with distilled water and anhydrous alcohol to get rid of the residues on the surface, and dried quickly after electrodeposition. Relative to a silver wire quasi-reference electrode, all potentials of electrodeposition were measured.

The structure of 1:2CU, 1:2CU with NiCl₂·6H₂O (0.2 M), 1:2CE and 1:2CE with NiCl₂·6H₂O (0.2 M) were examined by Fourier transform infrared spectroscopy (FTIR, AVATAR 370). The surface morphology the Ni coatings was characterized by scanning electron microscope (SEM, JEOL JSM-6700F, Japan) accompanied by the EDS, and the composition of the Ni

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