



Fabrication of thermally annealed NiFeS ternary alloy for green hydrogen production

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Significance and Relevance

In this work, we have studied and optimized the fabrication of a ternary alloy of Ni, Fe, and S for H₂ evolution reaction in an alkaline electrolyzer. Particularly, the alloy was fabricated using a template electrosynthesis method in a deposition containing different concentrations of Fe, Ni ions, and S precursor. The composition of the deposition bath was optimized, as well as the deposition parameters (such as potential and deposition time), to obtain a stable and durable alloy. Moreover, the effect of thermal annealing on alloy stability and performance was studied. The optimized electrode has been tested as both anode and cathode. Preliminary results showed a constant potential of -0.4 and 1.6 V vs RHE by applying a constant current density of $\pm 50 \text{ mA cm}^{-2}$.

Preferred and 2nd choice for the topic: H₂ storage and transportation, green H₂ production, hydrogen vectors, sustainable and clean energy production and transport

Preferred presentation: Oral preferred or Short Oral

Introduction and Motivations

In recent years, the use of renewable energy sources has increased more and more¹. The main drawback of this kind of energy source is the lack of predictability that makes an energy storage system mandatory. The production of green hydrogen using water electrolysis can solve this problem².

In this work, we have studied the performance of a novel catalyst for both H₂ and O₂ production in an alkaline electrolyzer. The catalyst was fabricated with nanostructured morphology using cheap and highly active materials, such as iron, nickel, and sulphur³.

Materials and Methods

The alloy was prepared by template electrosynthesis, using a polycarbonate membrane as a template⁴. To make it conductive, a thin gold layer was sputtered. In the same membrane face, a nickel current collector was electrodeposited. Then, the NWS were deposited into the other membrane face using a Watt's bath modified with FeSO₄·7 H₂O and Na₂S₂O₃·5H₂O using the pulsed electrodeposition method. The effect of different concentrations of S precursor and Fe ions in the deposition bath was studied. Finally, the polycarbonate membrane has been dissolved using pure dichloromethane. The annealing process was carried out in a N₂ saturated environment at different temperatures and for different time. The electrodes were characterized using SEM, EDS and XRD analysis. The performances of the electrode for both O₂ and H₂ evolution were evaluated using in a solution of 30% KOH.

Results and Discussion

The electrode fabrication was optimized to obtain a stable array of vertically standing NWs. This kind of morphology allows the obtaining of extremely high surface area and, thus, higher current density for both H₂ and O₂ production. As mentioned in the materials and method section, the concentration of Fe, S, and Ni in the NWs was optimized to obtain this goal. Particularly, the best results have been obtained using a deposition bath made of 0.44 M of Fe ions, 15 g/L of S precursor in the Ni Watt's bath. The annealing process was optimized as well, and the best results were obtained at 500°C for 3 minutes. Figure 1a shows the SEM image of the electrode after the annealing process, and the electrode shows the presence of vertically standing NWs with an average length of about 15 μm . The EDS analysis (Figure 1b) confirmed the deposition of the ternary alloy. The formation of the alloy was also confirmed using XRD analysis.

The optimized electrodes have been tested as both anode and cathode in an alkaline electrolyzer. The counter electrode was a Pt mesh, and a Hg/HgO as a reference electrode. The electrochemical surface area was evaluated by carrying out CVs at different scan rates in a potential range where non-faradic processes occur. These experiments were carried out using the optimized electrode and, for comparison, also a planar Ni strip. Results showed that the proposed electrode has a 10 time higher capacitance compared to the planar one. The same electrodes (NiFeS NWs and Ni strip) were used in a quasi-steady state polarization and results were fitted with Tafel regression. Results showed that the modification of the NiFe alloy with sulfur leads to an improvement in both O₂ and H₂ evolution reactions with a much more remarkable effect on the O₂ evolution reaction.

Finally, the electrodes were used in a short time polarization (6h) using a fixed current density of $\pm 50 \text{ mA cm}^{-2}$. Excellent preliminary results were obtained, with an outstanding stability of potential over time which is around 1.62 and -0.38V vs RHE for O₂ and H₂ production, respectively.

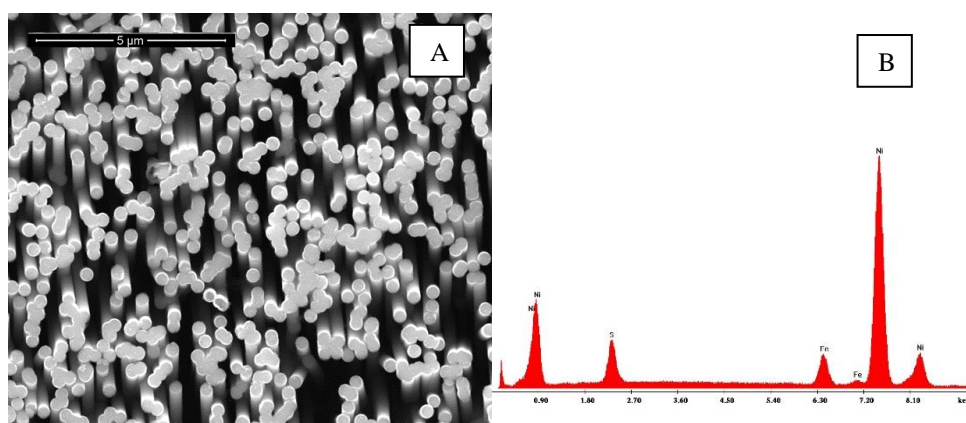


Figure 1 a) SEM image of NiFeS NWs after annealing and corresponding (b) EDS analysis.

Concluding, in the present work we have optimized the fabrication process of a ternary alloy of NiFeS NWs. The electrode was used for O₂ and H₂ production, showing excellent results. Indeed, compared to planar electrodes, the proposed one has a 10 times higher surface area and preliminary galvanostatic experiments showed a very stable potential over 6h of production.

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