

Laser-Engineered Cobalt-Deposited Nickel Foam Electrode for Alkaline Hydrogen Evolution

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ABSTRACT

The development of low-cost, high-performance electrocatalysts is critical for enabling sustainable hydrogen production via water electrolysis. Herein, we present a rapid, scalable, and energy-efficient strategy for fabricating highly active hydrogen evolution reaction (HER) electrodes by combining solvothermal deposition with subsequent laser processing on nickel foam (NF). The laser treatment induces an in situ transformation of the deposited cobalt precursor into catalytically active cobalt phases under ambient conditions, eliminating the need for conventional high-temperature thermal processing. The resulting Co-modified NF exhibits a substantially enlarged electrochemically active surface area and a high density of readily accessible active sites compared with pristine NF. Consequently, the electrode delivers markedly enhanced HER activity, requiring an overpotential of only 242 mV_{RHE} to achieve a current density of 50 mA cm⁻², compared with 351 mV_{RHE} for bare NF. The catalyst also demonstrates excellent electrochemical durability during prolonged operation. The enhanced catalytic performance is attributed to the synergistic effects of laser-induced surface reconstruction, increased exposure of catalytically active sites, and accelerated charge-transfer kinetics. These findings establish laser-assisted processing as a rapid, scalable, and versatile route for the fabrication of low-cost, high-performance electrocatalysts for efficient hydrogen production.

Keywords: renewable energy technologies, water electrolysis, clean hydrogen production, sustainable electrocatalysis, affordable and clean energy

1. INTRODUCTION

Designing efficient hydrogen production processes is central to the transition toward sustainable energy systems, with water electrolysis representing a key pathway for carbon-neutral H₂ generation in these systems^{1,2}. Yet, large-scale implementation faces intrinsically slow interfacial charge transfer and proton-electron coupling kinetics at the cathode in these systems. This leads to substantial energy losses, particularly under industrially relevant current densities^{3,4}. Consequently, the HER remains a key kinetic bottleneck that is further exacerbated in alkaline electrolyte, where water dissociation provides an extra step^{1,5-7}. Moreover, a persistent tradeoff between catalytic efficiency and economic feasibility continues to limit large-scale implementation of HER technologies⁸. Optimal HER activity requires near-thermoneutral hydrogen adsorption ($\Delta G_{H^*} \approx 0$ eV), yet most non-noble catalysts do not meet this requirement⁹. Although Pt exhibits near-ideal adsorption energetics and a minimal overpotential, its scarcity, high cost, susceptibility to poisoning, and long-term degradation severely limit its scalability¹⁰⁻¹². Overcoming these fundamental limitations remains a critical challenge for the realization of economically viable large-scale hydrogen production technologies.

Transition-metal-based materials, particularly Co-containing compounds, have emerged as promising HER electrocatalysts.

Their tunable electronic structures and d-band center optimize hydrogen adsorption and facilitate efficient charge transfer¹³⁻¹⁵. Further performance enhancements have been achieved by engineering metal-metal interfaces, where synergistic interactions improve electrical conductivity and increase active site density^{16,17}. Additionally, supporting these Co-based compounds on interconnected porous architectures mitigates diffusion limitations and promotes rapid gas-bubble release, which is vital for sustaining catalytic activity at high currents¹⁸. Therefore, numerous studies employ three-dimensional Ni foam substrates that impart excellent electrical conductivity, efficient mass-transport pathways, and a large surface area^{19,20}. Unfortunately, traditional synthesis strategies for the deposition of materials on Ni foams typically have limited scalability and allow poor structural control, hindering reproducibility and practical implementation²¹. Conventional fabrication methods also typically rely on multistep, energy-intensive procedures that increase production costs^{22,23}.

Conventional wet-chemistry synthesis inherently struggles with a tradeoff between catalytic performance and scalability²⁴. Laser-processing technologies offer a promising solution to this limitation. By utilizing rapid localized heating and chemical transformation, this technique modifies materials with high precision and minimal thermal damage to generate highly electrochemically active surfaces¹. The ultrafast heating-cooling

cycles inherent to laser processing enable nonequilibrium structural evolution and rapid surface modification, producing nanostructured materials with enhanced electrocatalytic activity^{2,25}. In our previous work, we used a similar methodology to synthesize a trimetallic carbide (NiFeCoC) catalyst requiring a low overpotential of 197 mV_{RHE} at 10 mA cm⁻² for the oxygen evolution reaction in alkaline media²⁶. Laser fabrication bypasses the diffusion limitations of bulk synthesis, ensures robust chemical bonding at the interface between the catalytically active layer and substrate, and reduces production time in relation to traditional multistep methods^{2,3}. Reducing the processing time is necessary to narrow the gap between laboratory research and industrial adoption²⁷.

Building on the unique capacity of laser treatment to rapidly engineer surface morphology and porosity, in this study, a laser-processed Co-functionalized Ni foam electrocatalyst is fabricated and its performance for the alkaline HER is evaluated in comparison with that of a bare Ni foam. By directly forming the catalytic layer on a porous conductive substrate, this approach increases the electrochemically active surface area (ECSA), while modulating the electronic structure of the Co-based active sites. Finally, X-ray diffraction (XRD) and scanning electron microscopy (SEM) were conducted to track changes in the surface states before and after prolonged electrolysis.

2. MATERIALS AND METHODS

2.1. Materials. All reagents were of analytical grade and used as received without further purification. Cobalt(II) acetate tetrahydrate (Co(CH₃COO)₂ · 4H₂O), ethanol (≥99.5%), ethylene glycol, urea, and polyvinyl alcohol (PVA, 99-100% hydrolyzed) were purchased from Sigma Aldrich. The Ni foam, obtained from MTI Corp. with 1.6 mm thickness, was cut into 1 cm × 2 cm pieces and ultrasonically cleaned in ethanol prior to use.

2.2. CO₂-Laser-Assisted Fabrication of Co-Deposited Ni Foam. Figure 1 provides a schematic of the fabrication of Co-based materials on the Ni foam. First, a precursor solution was obtained via dissolution of 0.05 g of polyvinyl alcohol in 18 mL of ethanol and stirring the mixture for 10 min. Second,

2 mL of ethylene glycol was added to the solution, followed by 0.12 g of urea and 0.249 g of cobalt(II) acetate tetrahydrate. In this system, ethanol served as a volatile solvent to promote uniform coating, while ethylene glycol served as both a coordinating agent and carbon source during the subsequent laser-induced decomposition.

The resulting mixture was heated to 90 °C and stirred at 300 rpm until it formed a homogeneous pink solution, indicating the complete dissolution of the Co salt and stabilizing additives. Pre-cleaned Ni foam substrates were immersed in the solution, and the entire mixture was transferred to a Teflon-lined stainless-steel autoclave. The autoclave was then sealed and subjected to hydrothermal treatment at 180 °C for 24 h. Next, the coated Ni foam pieces were removed and rinsed with ethanol to eliminate loosely bound residues. The collected sample was dried at 60 °C to stabilize the precursor layer before laser treatment.

Then, the precursor-coated substrate was subjected to laser treatment (Universal Laser Systems CO₂ laser (wavelength of 10.6 μm)), which induces rapid localized heating, promoting precursor decomposition and facilitating interactions between the Co species and the available carbon and oxygen sources (ethylene glycol and ethanol). This rapid thermal process drives the formation of catalytically active Co-based surface species. To produce the Co-deposited Ni foam electrodes, the CO₂ laser was operated at 30 W through an objective lens to treat the samples. To ensure a homogeneous dispersion of Co particles all over the surface, both sides of the substrates were irradiated. Following laser treatment, the samples were used directly without additional washing steps.

2.3. Material Characterization. X-ray diffraction (XRD; Rigaku Ultima IV) was used to characterize the crystalline structure over a 2θ range of 10°–80° at a scan rate of 5° min⁻¹. Surface morphology and elemental composition were examined via scanning electron microscopy (SEM; JEOL JSM-5800LV) equipped with energy-dispersive X-ray spectroscopy (EDX).

2.4. Electrochemical Measurements. All electrochemical measurements were performed in 1 M KOH using a Biologic SP-300 electrochemical workstation with a standard three-electrode configuration. The laser-treated, Co-deposited Ni foam was employed as the working electrode, with a Pt wire and an

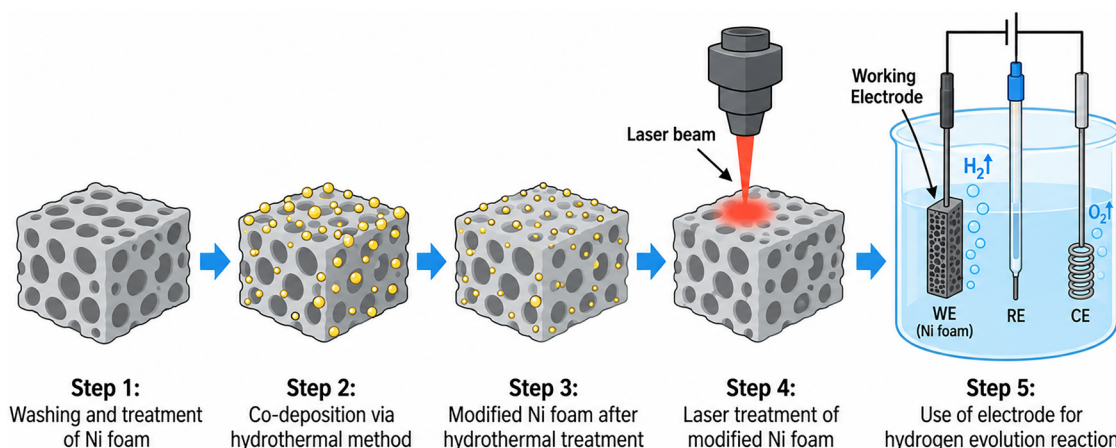


Figure 1. Fabrication process for the laser-treated Co-deposited Ni foam electrocatalyst, including precursor deposition, laser-induced structural transformation, and application in the HER

Ag/AgCl electrode serving as the counter and reference electrodes, respectively. All measured potentials were referenced to the reversible hydrogen electrode (RHE) scale by applying the Nernst equation: $E_{RHE} = E_{Ag/AgCl} + 0.197 + 0.059 \times \text{pH}$.

The catalytic activity of the electrodes was evaluated by linear sweep voltammetry (LSV) at a scan rate of 5 mV s^{-1} . Electrochemical impedance spectroscopy (EIS) was performed over a frequency range of 10^5 to 0.01 Hz to probe the reaction kinetics, and Tafel slopes were derived from the corresponding polarization curves. The ECSA of both the bare and laser-treated Co-deposited Ni foam samples were determined by cyclic voltammetry (CV) in the non-Faradaic region at varying scan rates. Chronoamperometry was carried out at a fixed potential to assess the long-term stability of the electrodes, followed by post-stability polarization measurements to monitor any degradation in electrocatalytic performance.

3. RESULTS AND DISCUSSION

3.1. Crystalline Phases of the As-Prepared Electrodes.

Figure 2 presents the XRD patterns of the as-prepared bare Ni and laser-treated Co-deposited Ni foam samples. The bare Ni foam substrate exhibits characteristic diffraction maxima at $2\theta = 44.5^\circ$, 51.8° , and 76.4° were readily indexed to the (111), (200), and (220) reflections of face-centered cubic Ni (JCPDS No. 04-0850).

Following Co deposition and treatment, these primary Ni peaks are fully retained with only minor variations in intensity and peak broadening. This demonstrates that the underlying Ni foam matrix remains structurally intact during Co deposition and laser treatment.⁴ Furthermore, the absence of new peaks following Co deposition and laser treatment, such as those associated with carbide species, indicates that the laser treatment

did not induce a bulk structural transformation of the substrate. Additionally, diffraction peaks for Co-based phases were not observed, which is attributable to either the low crystallinity of the deposited layer or a low mass-loading that falls below the detection limit of the XRD system⁵. Despite the lack of a measurable XRD signature, the successful deposition of Co on the Ni foam was confirmed through SEM and EDX.

3.2. Morphology and Elemental Composition. SEM was conducted to examine the surface features of the electrodes. As shown in Figure 3 (a), the bare Ni foam exhibits a three-dimensional, open-cell structure characterized by a wide, regular network of large pores²⁸. This porous architecture facilitates easy electrolyte penetration and mass transport. At higher magnification (Figure 3(b)), the Ni foam appears smooth and free of particulate impurities⁶. EDX spectroscopy of the bare Ni foam confirms the purity of the substrate, revealing predominantly strong Ni peaks (Figure 3 (c)). Although a minor oxygen signal is observed, possibly corresponding to the surface oxidation of Ni upon atmospheric exposure, no other elemental impurities are detected. Following Co deposition and laser treatment, the interconnected porous structure of the Ni foam remained intact (Figure 3 (d)). However, the microstructural surface morphology is notably altered (Figure 3 (e)): the previously smooth foam walls are now uniformly covered with small spherical particles and irregularly shaped clusters. This increase in surface roughness provides visual confirmation of the successful coating of Co on the Ni framework. The EDX spectrum of the treated foam exhibits both Ni and Co peaks, confirming the deposition of Co on the Ni framework (Figure 3 (f)). The relative intensities of the Co peaks further confirm the presence of Co on the Ni foam

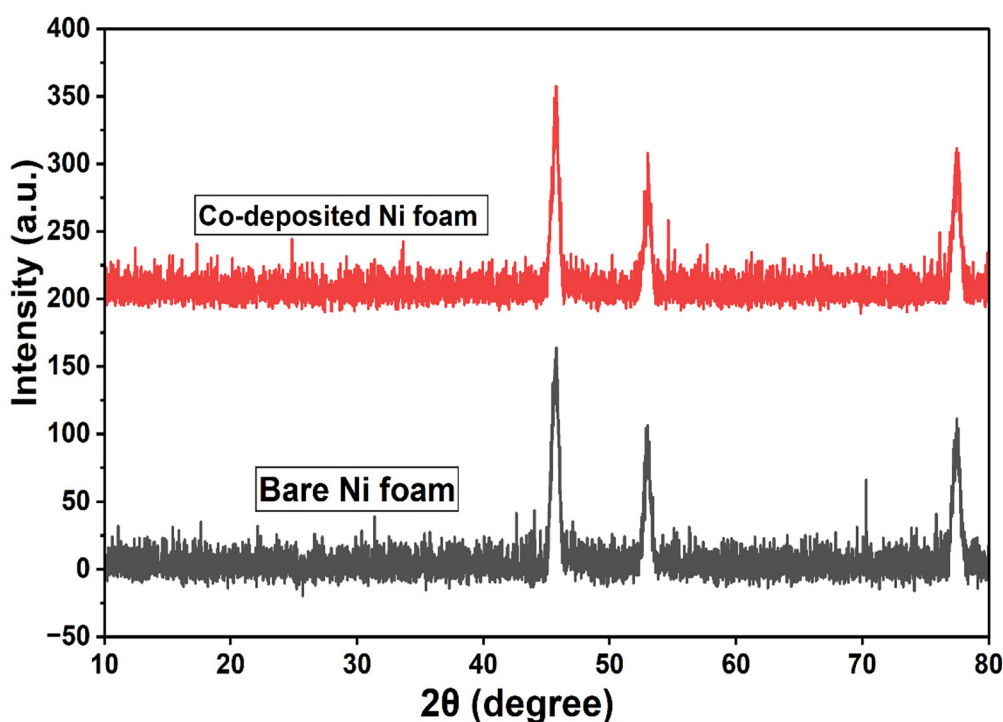


Figure 2. XRD patterns of the bare Ni foam and laser-treated Co-deposited Ni foam

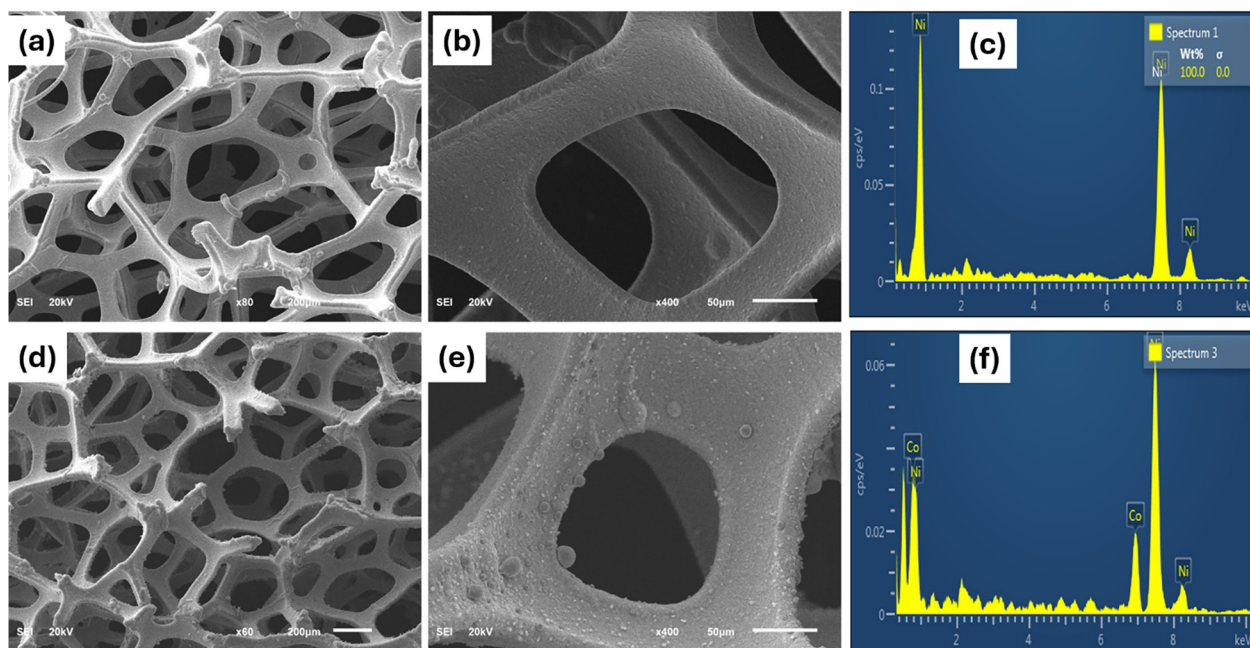


Figure 3. SEM images and EDX spectra of the (a–c) bare Ni foam and (d–f) Co-deposited Ni foam

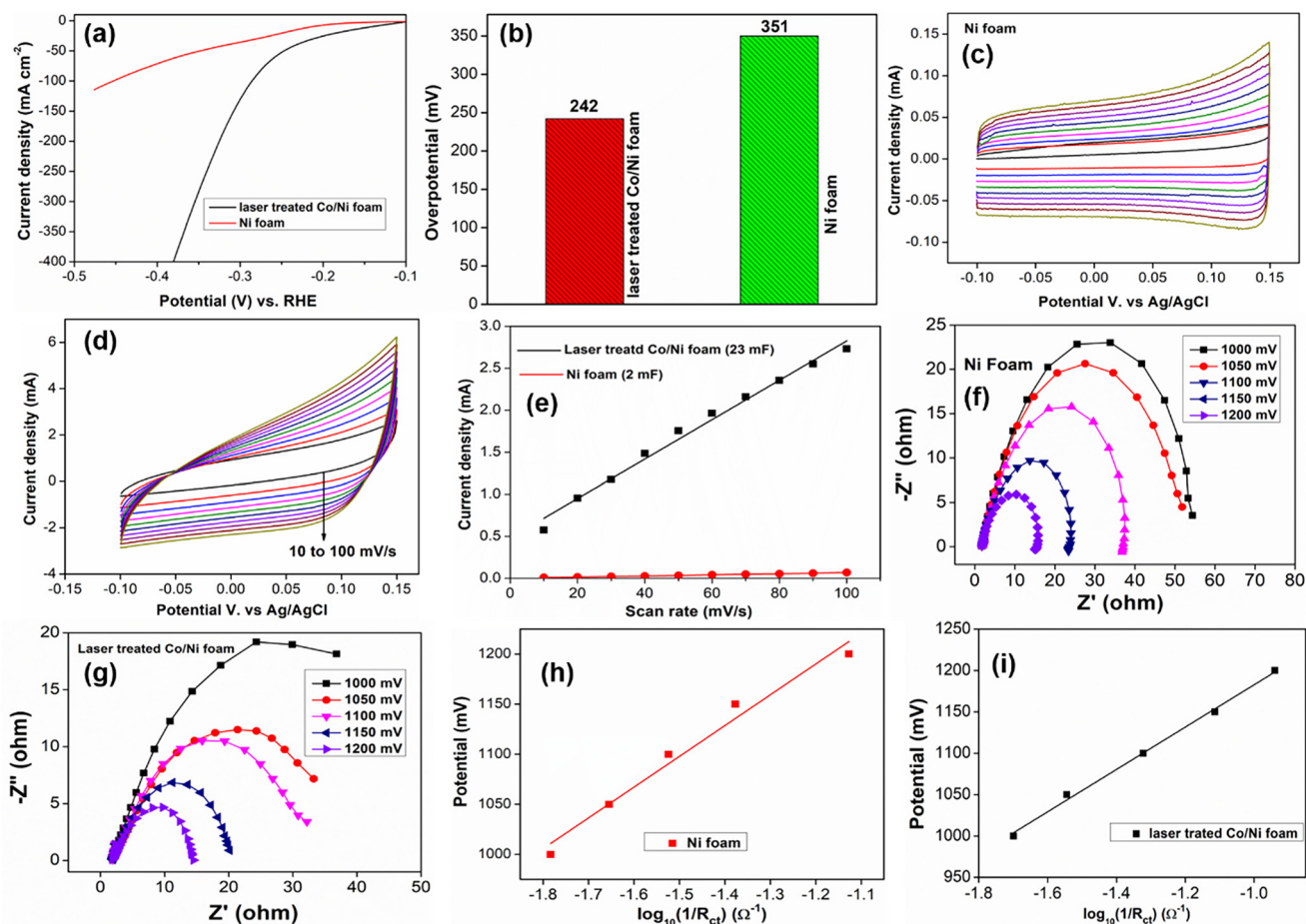


Figure 4. Electrochemical characterization of the bare Ni foam and laser-treated Co-deposited Ni foam: (a) LSV curves, (b) overpotential comparison, (c–d) CV curves obtained at different scan rates, (e) double-layer capacitance plots, (f–g) EIS Nyquist plots, and (h–i) Tafel analysis

surface. Although quantitative inductively coupled plasma spectroscopy was not performed, the qualitative EDX results confirm the presence of Co on the Ni foam substrate.

3.3. Electrochemical Performance. The electrocatalytic performance of the prepared electrodes toward the HER was systematically evaluated using LSV, CV, EIS, Tafel analysis, and double-layer capacitance (C_{dl}) measurements, with the corresponding results presented in Figure 4. As shown in Figure 4a, the laser-treated Co-modified Ni foam exhibits substantially improved HER activity compared with pristine Ni foam. Specifically, the Co-modified electrode requires an overpotential of only 242 mV to deliver a current density of 50 mA cm^{-2} , representing a reduction of 109 mV relative to bare Ni foam (351 mV). This pronounced decrease in overpotential demonstrates that laser-induced cobalt modification significantly enhances the intrinsic electrocatalytic activity of the Ni foam, facilitating more efficient hydrogen evolution under alkaline conditions.

To understand this enhanced performance, the ECSA of both electrodes was evaluated through CV conducted at various scan rates in the nonfaradaic regions (Figures 4(c) and (d)). The Co-deposited Ni foam exhibits a greater current response than the bare substrate. As a result, the calculated C_{dl} for the Co-treated electrode is 23 mF cm^{-2} , compared to only 2 mF cm^{-2} for the bare Ni foam (Figure 4e). This more than ten-fold increase in the ECSA confirms that the laser-induced structural transformation successfully generated a highly textured, catalytically accessible surface architecture.

Furthermore, EIS was performed to probe the charge-transfer kinetics at the electrode–electrolyte interface. The corresponding Nyquist plots (Figures 4(f) and (g)) were fitted using a standard equivalent circuit comprising solution resistance (R_s), charge-transfer resistance (R_{ct}), and C_{dl} . The Co-modified electrode exhibited a smaller semicircle diameter, corresponding to a fitted R_{ct} of 50.1Ω at 1.0 V potential, which is lower than that exhibited by the bare Ni foam (61.3Ω). This reduction indicates that the Co-based deposition facilitates more rapid interfacial electron transfer during the HER.

Finally, Tafel analysis was employed to evaluate the reaction kinetics. Figures 4(h) and (i) show that the Co-deposited Ni foam demonstrates a smaller Tafel slope than the bare Ni foam, indicating a more favorable reaction pathway that requires less energy to overcome the kinetic barriers of the HER.

3.4. Long-term Stability and Surface Activation. The long-term durability of the laser-treated Co-deposited Ni foam was evaluated through continuous chronoamperometry under consistent experimental conditions (Figure 5(a)). Over the 24 h operating period, the current density decreases by $\sim 3\%$, indicating good electrochemical stability of the catalyst. Notably, an initial increase in the current density was observed during the early stages of testing. This transient enhancement indicates in situ electrochemical activation, a phenomenon frequently observed in transition-metal-based electrocatalysts. This activation is typically driven by two factors: improved electrolyte infiltration deeper into the porous Ni foam, which exposes additional active sites; and the electrochemical surface reconstruction (or partial oxidation) of the Co sites, which enhances charge-transfer kinetics.⁹ Following the initial activation stage, the current density stabilizes over an extended period, indicating good durability under continuous electrolysis.

To further assess changes in catalytic activity over time, LSV was performed immediately after the stability test. The polarization curves (Figure 5(b)) indicate that the post-test electrode required a slightly lower overpotential than the fresh electrode. It might appear contradictory that the chronoamperometric current slightly decreased while the post-test LSV performance improved. However, this difference arises because chronoamperometry is conducted at a fixed potential over long durations, making the measurement sensitive to factors such as mass-transport limitations and the physical accumulation of bubbles at the electrode surface, which temporarily block active sites. In contrast, LSV reflects the intrinsic electrocatalytic capability of the material itself. Therefore, the reduced post-test overpotential confirms that the catalyst becomes more active over time despite minor current fluctuations during prolonged operation. Although pre-activation using CV was omitted prior to testing, the extended

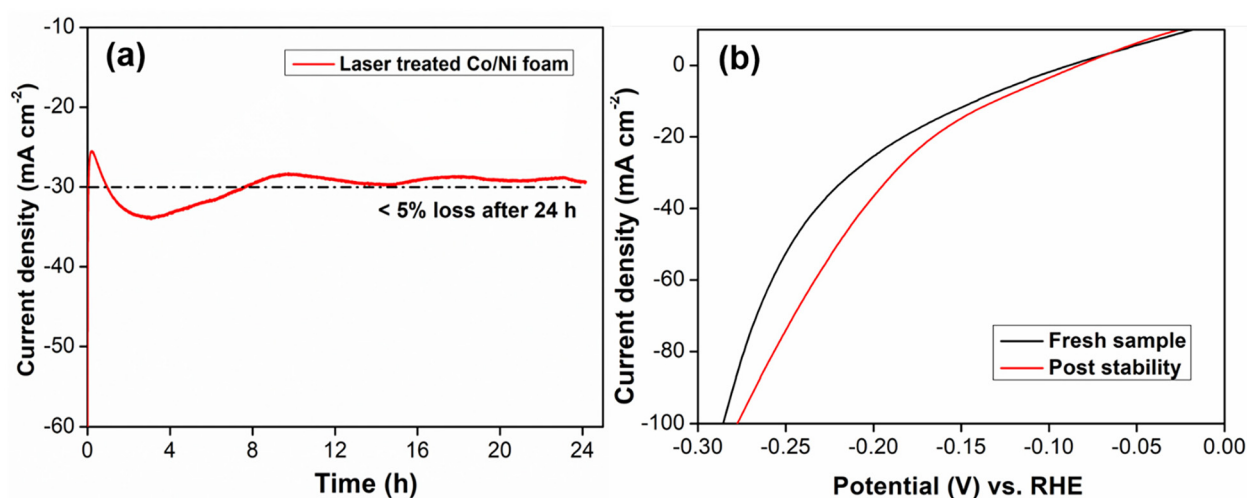


Figure 5. Stability assessment of the laser-treated Co-deposited Ni foam, including: (a) chronoamperometry profile over 24 h, and (b) LSV curves before and after the stability test

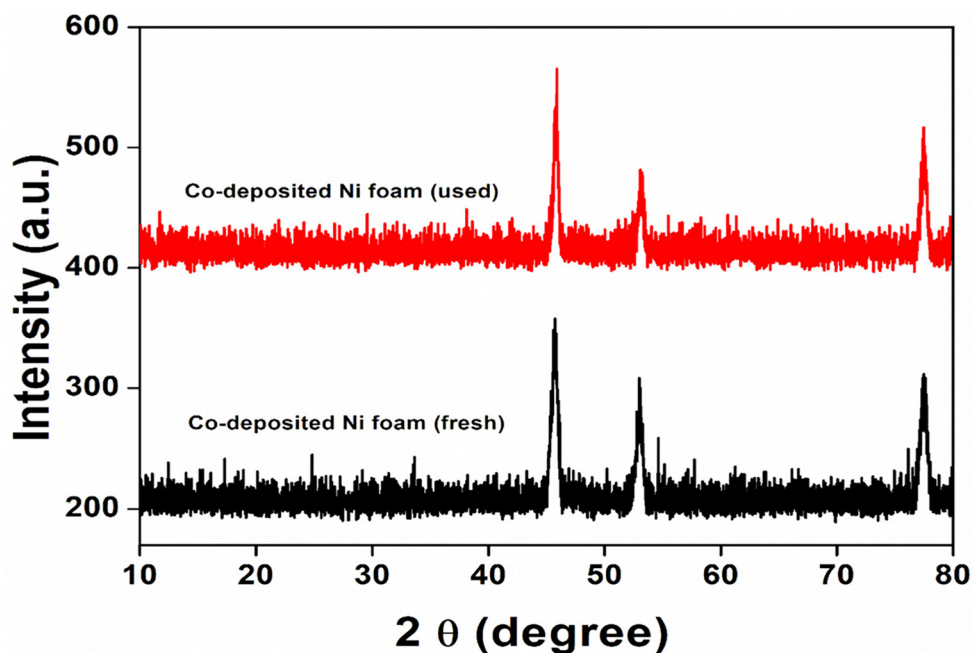


Figure 6. XRD patterns of the laser-treated Co-deposited Ni foam before and after electrochemical stability tests

chronoamperometric operation served the same purpose, allowing the electrode to reach its optimal active state.

3.5. Post-Electrolysis Phase Stability. To assess the catalytic durability after prolonged use, XRD patterns of the laser-treated Co-deposited Ni foam were recorded before and after the

stability test (Figure 6). Both patterns exhibit similar peak positions, indicating that the bulk crystal structure of the Ni foam is preserved after 24 h of continuous electrolysis. Minor variations, specifically slight peak broadening and a minor reduction in intensity, are observed in the post-test sample. Because deposited Co remained amorphous or below the XRD detection threshold,

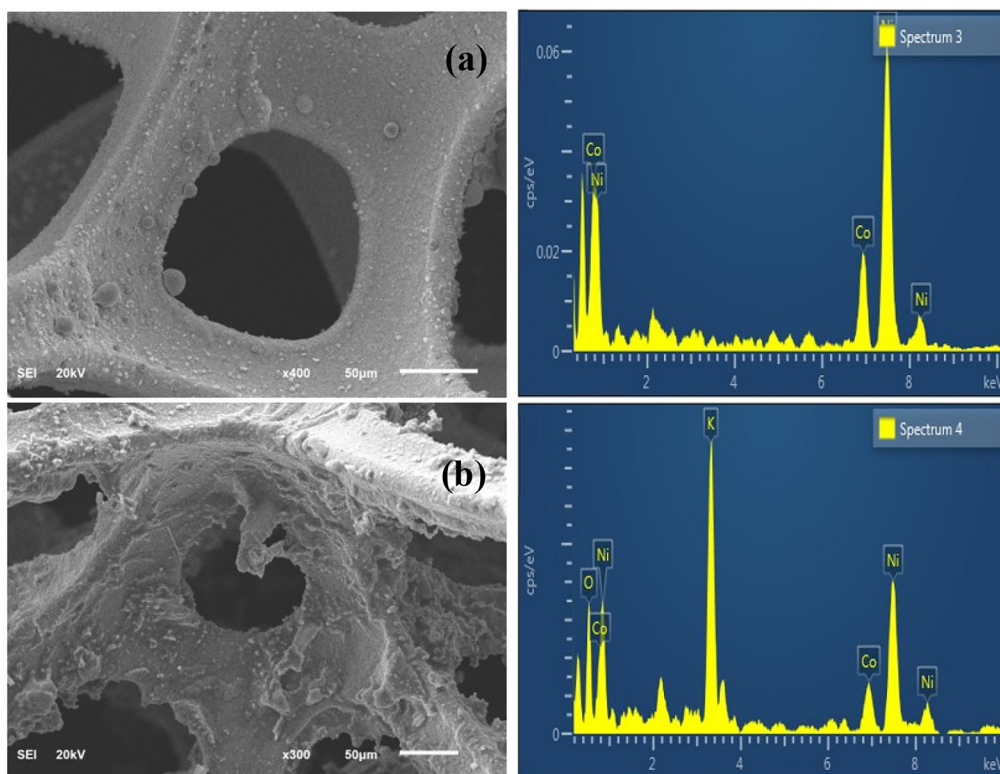


Figure 7. SEM image and EDX spectrum of the Co-based Ni foam (a) before and (b) after electrochemical testing

these crystallographic changes are associated with the Ni substrate itself. The minor peak broadening is likely attributable to increased surface roughness or partial oxidation induced by the testing conditions. Ultimately, the retention of the characteristic Ni diffraction peaks suggests that the electrode structure remains largely stable during prolonged operation.

3.6. Post-Electrolysis Morphology and Elemental Composition. To investigate the microstructural and elemental changes induced by prolonged hydrogen evolution, the surface morphology of the laser-treated Co-deposited Ni foam was examined via SEM and EDX before and after the 24 h stability test (Figure 7). The SEM images of the pre-test sample indicate that the surface of the Ni foam was covered with irregularly shaped Co clusters and particles. However, following exposure to the alkaline electrolyte during the test, the SEM images indicate noticeable changes to the surface characteristics of the electrode.

EDX spectroscopy revealed a reduction in the relative Co signal intensity compared to the fresh sample. Although this reduction indicates loss of a small amount of Co during operation, the continued presence of Co and Ni peaks in the EDX spectrum confirms that Co was largely retained on the substrate. Additionally, a new K signal appears in the post-test sample, likely originating from traces of the KOH electrolyte. Although the material went through some restructuring and minor Co loss, the activity of the electrode was largely maintained.

4. CONCLUSIONS

This study demonstrated that CO₂ laser processing is an effective and scalable process for the rapid fabrication of high-performance HER electrodes. A combined solvothermal deposition and laser-treatment approach enabled the formation of Co-based active species uniformly distributed on a Ni foam substrate. The resulting laser-treated Co-deposited Ni foam exhibited significantly enhanced hydrogen evolution activity in relation to the bare Ni foam, driven by an approximately tenfold increase in the ECSA. Furthermore, the Co-modified electrode demonstrated excellent electrochemical durability, maintaining stable performance over uninterrupted electrolysis for 24 h at 30 mA cm⁻². Importantly, this approach overcomes the limitations associated with conventional time- and energy-intensive fabrication methods, offering a rapid, cost-effective, and versatile route for electrode design. The demonstrated strategy provides a clear pathway for the production of efficient HER electrocatalysts and is expected to be readily adaptable to a wide range of transition-metal catalytic systems.

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