

Amorphous $\text{Ce}_{0.5}\text{Fe}_{0.5}\text{O}_{1.75}$ /NiO electrocatalyst synthesized by hydrothermal method and wet impregnation for enhanced oxygen evolution reaction

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Research Article

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Abstract

Herein, we fabricate a novel self-supported electrode with amorphous $\text{Ce}_{0.5}\text{Fe}_{0.5}\text{O}_{1.75}$ nanosheets directly grown on nickel foam (denoted as $\text{Ce}_{0.5}\text{Fe}_{0.5}\text{O}_{1.75}\text{@NF}$). The optimized $\text{Ce}_{0.5}\text{Fe}_{0.5}\text{O}_{1.75}\text{@NF}$ delivers a low overpotential of 309 mV at 100 mA cm^{-2} for the oxygen evolution reaction (OER) in alkaline freshwater, outperforming many reported amorphous oxide electrocatalysts. Moreover, outstanding long-term durability over 200 h is achieved when operated at 100 mA cm^{-2} . The electrocatalysts were characterized by various physical and electrochemical analyses. Experimental characterizations demonstrate that Ce incorporation triggers heterostructure construction and d–f orbital coupling to tune the electronic configuration of Fe sites. Such a characteristic realizes controllable lattice oxygen oxidation without structural degradation, which further modulates the adsorption strength of OER intermediates, multiplies exposed active sites, and accelerates directional electron transport. Meanwhile, the amorphous $\text{Ce}_{0.5}\text{Fe}_{0.5}\text{O}_{1.75}/\text{NiO}$ heterostructure induces spontaneous charge redistribution, which promotes interfacial charge transfer and optimizes intermediate binding to achieve near-thermoneutral adsorption. Benefiting from these merits, significantly boosted OER catalytic activity is obtained. Accordingly, the as-prepared $\text{Ce}_{0.5}\text{Fe}_{0.5}\text{O}_{1.75}\text{@NF}$ holds great promise for large-scale industrial hydrogen production.

Introduction

The progressive decline in fossil fuel supplies continues to focus significant academic attention on sustainable energy conversion and storage.¹ The accelerating depletion of fossil fuel resources has catalyzed a paradigm shift toward sustainable energy technologies. Among the various alternatives, hydrogen stands out as a carbon-neutral energy vector, owing to its high volumetric and gravimetric energy densities superior to those of conventional fuels, as well as its favorable characteristics for long-term storage and transportation.² The various hydrogen production technologies, anion exchange membrane water electrolysis (AEMWE) stands out due to its high efficiency, cost-effectiveness, and compatibility with renewable energy sources.^{1, 3, 4} Catalyst activity on the electrode support serves as the primary determinant of electrolyzer performance for hydrogen production.⁵ The sluggish kinetics of the OER represents a major bottleneck in water electrolysis for hydrogen production.^{6–8}

Owing to their narrow bandgap, strong covalent character, low cost, and ease of synthesis, transition-metal-based materials represent a promising class of electrocatalysts that have been the subject of intensive research.^{9,10} Rational electronic regulation is regarded as an efficient means to promote the electrocatalytic capabilities of transition-metal-based catalytic materials. The d-band center (ϵ_d) of transition metals is a well-established energy descriptor for evaluating such modified catalytic behavior.^{11–14} The d-band center (ϵ_d) relative to the Fermi level (E_F) governs the occupancy of antibonding states and thus the binding strength of electrocatalytic intermediates.^{15, 16} Orbital coupling—hybridizing the 3d orbitals of transition metals with heteroatomic orbitals—offers a versatile means to

shift ed.^{17, 18} In this context, the f orbitals of rare-earth (RE) elements stand out as superior electronic modulators compared with conventional d orbitals.¹⁹ The strong d–f orbital coupling can construct d–f electronic ladders, which facilitate wavefunction overlap and thereby establish a charge-transfer highway. This highway effectively lowers the energy barrier and balances the adsorption of intermediates during electrocatalytic reactions.^{20, 21}

CeO₂ is distinguished among rare-earth oxides by its exceptional stability, favorable oxygen affinity, and 4f¹5d¹6s² configuration that enables reversible Ce³⁺/Ce⁴⁺ switching—attributes that furnish abundant electron-transfer sites and position it as an ideal platform for interface engineering.^{22–26}

Figure 2 Fig. 2 Composition and structure characterization of Ce_{0.5}Fe_{0.5}O_{1.75}@NF. (a) Schematic for the preparation of Ce_{0.5}Fe_{0.5}O_{1.75}@NF. (b) XRD patterns of Ce_{0.5}Fe_{0.5}O_{1.75}@NF. (c, d) TEM images. (e) EDX mapping of the Ce_{0.5}Fe_{0.5}O_{1.75}@NF.

Electrocatalytic activity at the electrode support dictates the overall performance of water electrolysis for hydrogen generation.²⁷ To date, there has been no report on amorphous bimetallic cerium–iron oxide (Ce_{0.5}Fe_{0.5}O_{1.75}) as an electrocatalyst for the OER. Most existing Ce–Fe based OER catalysts focus on Fe-doped fluorite CeO₂, crystalline perovskite Ce_{0.5}Fe_{0.5}O_{1.75}, or Ce-modified iron (oxy) hydroxides, which exhibit distinct metal–oxygen stoichiometry and much lower oxygen vacancy concentration compared with Ce_{0.5}Fe_{0.5}O_{1.75}. This highly oxygen-deficient amorphous Ce_{0.5}Fe_{0.5}O_{1.75} delivers unique electronic coupling between Ce 4f and Fe 3d orbitals, enabling an optimized lattice oxygen-mediated OER pathway for AEMWE anodes. The Ce_{0.5}Fe_{0.5}O_{1.75}/NiO heterointerface triggers spontaneous charge transfer across the interface, which enhances charge transport, optimizes intermediate adsorption, and accelerates the overall reaction kinetics.

RESULTS AND DISCUSSION

Synthesis of amorphous Ce_{0.5}Fe_{0.5}O_{1.75} catalysts

As schematically illustrated in Fig. 2a, CeO₂@NF electrocatalysts were synthesized in situ on nickel foam by using a hydrothermal process. The obtained precursor was then calcined at 350°C for 3 h in flowing air to prepare the CeO₂@NF support, with Ce (NO₃)₃·6H₂O (99.5%, Macklin) employed as the cerium source.

Fe (NO₃)₃·9H₂O (Macklin) was introduced onto CeO₂ via incipient wetness impregnation with nominal Fe loadings of 1, 2, 3, 4, and 5 mmol, and the resulting materials were dried overnight in an oven. Subsequent quenching at room temperature yields amorphous Ce_{0.5}Fe_{0.5}O_{1.75}.

Characterization of amorphous CeFeO catalysts

Figure 3 XPS characterizations of Ni, Fe, and Ce composite. (a) Ni 2p, (b) Fe 2p, (c) Ce 3d, and (d) survey XPS spectra confirm the presence of C, O, Fe, Ni, and Ce elements. sat = satellite peaks.

X-ray diffraction (XRD) measurements were performed to characterize the crystalline structure and chemical composition of the $\text{Ce}_{0.5}\text{Fe}_{0.5}\text{O}_{1.75}$ @NF composite, and the corresponding diffraction peaks were analyzed to verify its material composition. XRD analysis (Fig. 2b) confirms the amorphous nature of $\text{Ce}_{0.5}\text{Fe}_{0.5}\text{O}_{1.75}$, which is characterized by a broad amorphous diffraction hump in the range of 20–40°. In addition, sharp diffraction peaks corresponding to the nickel foam substrate (PDF#01-017-4655) are clearly observed in the pattern.

Scanning electron microscopy (SEM) (Fig. 1a–d) was employed to characterize the microstructure and morphology of the as-prepared electrode material. The low-magnification SEM image (Fig. 1a, magnification: 114×, scale bar: 100 μm) reveals the three-dimensional porous skeleton of the nickel foam, where the light-white regions are ascribed to the generated NiO, and the dark regions on the surface correspond to the target product, amorphous $\text{Ce}_{0.5}\text{Fe}_{0.5}\text{O}_{1.75}$. Upon further magnification (Fig. 1b, magnification: 10.00 k×, scale bar: 1 μm), a uniform and dense thin layer of nickel oxide (NiO) is observed to cover the surface of the nickel foam skeleton. At higher magnifications (Fig. 1c and Fig. 1d, magnification: 30.00 k×, scale bars: 300 nm), the primary active material $\text{Ce}_{0.5}\text{Fe}_{0.5}\text{O}_{1.75}$ exhibits a distinctive fish-scale-like morphology. These nanoscale scales are interleaved, densely packed, and grow perpendicularly or obliquely on the NiO-modified nickel foam surface. Notably, a well-defined heterostructure is formed between the active material and the NiO/nickel foam substrate, which is beneficial for efficient charge separation and transport at the interface, thereby enhancing the alkaline electrocatalytic oxygen evolution performance. This uniform and dense fish scale like nanosheet structure effectively reduces the solid–gas contact area fraction, decreases adhesion, and consequently significantly suppresses bubble coalescence during OER.²⁷ Transmission electron microscopy (TEM) images confirmed the ultrathin nanostructure of $\text{Ce}_{0.5}\text{Fe}_{0.5}\text{O}_{1.75}$ and its close association with the nickel foam substrate, which improves the mechanical stability of the catalyst (Fig. 2c-d).EDX-mapping measurements were performed to analyse the specific elemental composition of the materials(Fig. 2e),acquire the quantitative atomic fraction of individual elements (Table S1). High-resolution transmission electron microscopy (HRTEM) images further reveal the presence of NiO within the material. Nickel oxide is generated during the calcination process in air, forming a dense oxide layer on the surface with a lattice spacing of 0.147 nm (Fig. 2c), which corresponds to the (220) plane of NiO(Figure S4).

XPS was adopted to determine both the surface elemental composition and the corresponding valence states. It should be noted that the Ni 2p_{1/2} and Ce 3d_{5/2} photoelectron peaks exhibit significant overlap in binding energy when using Al Kα radiation. Therefore, in this study, the analysis of Ni chemical states was primarily conducted using the well-resolved Ni 2p_{3/2} peak to avoid potential misinterpretation.²⁸ The Ce 3d spectra were analyzed based on the Ce 3d_{3/2} component.Complete XPS spectra for Ni 2p and Ce 3d can be found in Figures S6-S7. The Ni 2p spectrum revealed peaks at 855.2 and 872.8 eV, attributed

to Ni^{2+} , with satellite peaks at 860.8 and 881.4 eV (Fig. 3a).²⁹ For the Fe 2p orbitals (Fig. 3b), $\text{Ce}_{0.5}\text{Fe}_{0.5}\text{O}_{1.75}$ exhibits peaks for Fe^{3+} at 711.3 and 724.1 eV, along with satellite peaks at 718.1 and 732.4 eV. Under the high anodic potential, the catalyst undergoes significant structural reconstruction and valence-state evolution. (Fig. 3c) The extraction of lattice oxygen generates oxygen vacancies, which promote the reduction of Ce^{4+} , ultimately leading to a red shift of the Ce 3d peaks. Meanwhile, Ni and Fe are progressively oxidized to form highly active oxyhydroxide phases; the increase in metal ion valence states reduces the outer electron-cloud density, resulting in a blue shift of the Ni 2p and Fe 2p characteristic peaks. The intrinsic driving force behind these concerted spectral shifts is ascribed to the bidirectional electron transfer among the heterogeneous Fe–Ce–Ni components.

Electrocatalytic OER Performance

Electrochemical characterizations were first performed in alkaline water media. The Ce/Fe molar ratio was optimized. Among the impregnation concentrations of ferric nitrate nonahydrate ranging from 1 to 5 mmol, it was revealed that $\text{Ce}_{0.5}\text{Fe}_{0.5}\text{O}_{1.75}\text{@NF}$ derived from the material impregnated with 3 mmol ferric nitrate nonahydrate delivered the optimal OER activity in alkaline freshwater (Fig. 4a). Thus, the $\text{Ce}_{0.5}\text{Fe}_{0.5}\text{O}_{1.75}\text{@NF}$ electrode obtained under the above conditions was adopted for further characterization.

As illustrated in Fig. 4b, in 1 mol L^{-1} KOH alkaline electrolyte, amorphous $\text{Ce}_{0.5}\text{Fe}_{0.5}\text{O}_{1.75}\text{@NF}$ exhibits outstanding electrocatalytic performance toward the OER. To achieve 100 mA cm^{-2} , this catalyst merely demands an overpotential of 309 mV, which is substantially lower than those of CeO_2NF (349 mV), NF (463 mV), and benchmark commercial RuO_2 (376 mV). Long-term durability measurements performed in 1 mol L^{-1} KOH reveal that the catalyst maintains stable electrocatalytic operation for 200 h, accompanied by a current degradation rate of less than 5%.

The corresponding Tafel slopes for OER are further calculated via the Tafel equation. As presented in Fig. 4d, the amorphous $\text{Ce}_{0.5}\text{Fe}_{0.5}\text{O}_{1.75}\text{@NF}$ electrode exhibits the minimum Tafel slope of 43 mV dec^{-1} among all tested samples, which is far lower than those of bare NF (102 mV dec^{-1}), commercial RuO_2 (94 mV dec^{-1}), and $\text{CeO}_2\text{@NF}$ (70 mV dec^{-1}). This result confirms the fastest OER reaction kinetics of $\text{Ce}_{0.5}\text{Fe}_{0.5}\text{O}_{1.75}\text{@NF}$ in alkaline electrolyte, whereas $\text{CeO}_2\text{@NF}$ displays inferior catalytic activity compared with the target catalyst.

To rationalise the superior OER performance of $\text{Ce}_{0.5}\text{Fe}_{0.5}\text{O}_{1.75}\text{@NF}$, cyclic voltammetry (CV) measurements were used to calculate the double-layer capacitance (Cdl). (Figure S1- S3) the electrochemically active surface area (ECSA) for each electrode was derived from the Cdl normalised by an average specific capacitance (Cs) of $40 \mu\text{F cm}^{-2}$, measured in the non-faradaic region at the electrode–electrolyte interface.³⁰ The Cdl values, extracted from the linear fits of current density versus scan rate plots (Fig. 4e), are summarised below.

$\text{Ce}_{0.5}\text{Fe}_{0.5}\text{O}_{1.75}\text{@NF}$ delivers a prominent Cdl of 1032 mF cm^{-2} , which far surpasses those of $\text{CeO}_2\text{@NF}$ (711 mF cm^{-2}) and bare NF substrate (706 mF cm^{-2}). Since the ECSA is positively proportional to Cdl,³¹ this result unambiguously verifies that the $\text{Ce}_{0.5}\text{Fe}_{0.5}\text{O}_{1.75}$ composite supported on nickel foam possesses substantially more accessible electrocatalytic active sites compared with monometallic $\text{CeO}_2\text{@NF}$ and the pristine nickel foam substrate.

From the fitted Nyquist plots (Fig. 4f) with the inset displaying the adopted equivalent-circuit model (Figure S5), the $\text{Ce}_{0.5}\text{Fe}_{0.5}\text{O}_{1.75}\text{@NF}$ electrode delivered a charge-transfer resistance of only 2.8Ω , far smaller than $\text{CeO}_2\text{@NF}$ (5.2Ω), while bare NF exhibited a remarkably larger charge-transfer impedance of 6.5Ω , confirming the drastically accelerated interfacial charge-transfer kinetics of the bimetallic Ce–Fe oxide catalyst. All three electrodes displayed nearly identical solution resistance ($R_s \approx 0.3 \Omega$) at the high-frequency intercept of the real axis, ruling out the interference of electrolyte resistance and further verifying that the distinct electrocatalytic performance gaps stem solely from the optimized charge-transport capability of $\text{Ce}_{0.5}\text{Fe}_{0.5}\text{O}_{1.75}$.

Mechanism Exploration

To gain deeper insight into the catalytic origin of $\text{Ce}_{0.5}\text{Fe}_{0.5}\text{O}_{1.75}$ based on the available characterization techniques. Based on the above XPS results, the electrocatalytic process of the Fe–Ce–Ni oxide catalyst follows a typical lattice oxygen-mediated mechanism (LOM), rather than the conventional adsorption evolution mechanism (AEM). Under high anodic potential, the lattice oxygen in the catalyst bulk is directly involved in the electrochemical reaction and undergoes continuous extraction and reconstruction. The removal of lattice oxygen creates abundant oxygen vacancies, which modulate the local electronic structure and promote the reduction of Ce, thereby inducing the red shift of Ce 3d peaks.

Figure 4 OER electrocatalytic performances of various electrodes in 1 M KOH alkaline electrolyte. (a) LSV polarization curves of catalysts prepared with different $\text{Fe}(\text{NO}_3)_3$ feeding amounts. (b) OER polarization curves of bare NF, $\text{CeO}_2\text{@NF}$, $\text{Ce}_{0.5}\text{Fe}_{0.5}\text{O}_{1.75}\text{@NF}$, and commercial RuO_2 benchmark. (c) Bar chart comparison of overpotentials at varied current densities ($10\text{--}200 \text{ mA cm}^{-2}$) for NF, $\text{CeO}_2\text{@NF}$, $\text{Ce}_{0.5}\text{Fe}_{0.5}\text{O}_{1.75}\text{@NF}$, and RuO_2 . (d) Tafel plots were acquired based on the LSV curves shown in panel (b). (e) Linear fitting curves of capacitive current versus scan rate to calculate Cdl values. (f) Nyquist EIS plots collected at fixed OER overpotential for NF, $\text{CeO}_2\text{@NF}$, and $\text{Ce}_{0.5}\text{Fe}_{0.5}\text{O}_{1.75}\text{@NF}$ (g) Long-term chronopotentiometry stability measurement of $\text{Ce}_{0.5}\text{Fe}_{0.5}\text{O}_{1.75}\text{@NF}$ at 100 mA cm^{-2} for 200 h.

Furthermore, the LOM-driven bidirectional electron transfer within the Fe–Ce–Ni heterostructure dominates the valence evolution of metal active sites. The interfacial charge redistribution facilitates the progressive oxidation of Ni and Fe species into highly active oxyhydroxide phases. The elevated valence states of metal ions reduce the outer electron cloud density and strengthen metal–oxygen bonding, leading to the blue shift of Ni2p and Fe2p characteristic peaks. Such synergistic structural and electronic

modulation originating from lattice oxygen participation fundamentally optimizes the reaction kinetics and endows the catalyst with superior electrocatalytic performance.

The incorporation of Ce gives rise to d–f orbital hybridization with Fe, which effectively lowers the intrinsically high adsorption energy of Fe for oxygen-containing intermediates, thus being beneficial to the oxygen evolution process. Moreover, the Ce species, after such modification, act as additional active sites; their favorable interaction with OH intermediates also contributes to accelerating the overall oxygen evolution reaction.^{32–34}

Conclusion

In summary, amorphous $\text{Ce}_{0.5}\text{Fe}_{0.5}\text{O}_{1.75}$ @NF electrodes were fabricated via in situ hydrothermal growth of CeO_2 on NF, followed by physical impregnation of ferric nitrate and subsequent rapid thermal quenching. The incorporated Ce species enable strong d–f orbital coupling with Fe centers, which modulates the electronic configuration around active doping sites. This electronic regulation optimizes the lattice oxygen-mediated OER pathway for the anodic reaction of AEMWE, while substantially alleviating the intrinsically strong adsorption of oxygen-containing intermediates on Fe sites to facilitate oxygen evolution. Meanwhile, the heterostructure constructed between amorphous $\text{Ce}_{0.5}\text{Fe}_{0.5}\text{O}_{1.75}$ and nickel oxide triggers spontaneous self-driven charge redistribution within the heterostructure, accelerating interfacial charge transfer and tuning the binding strength of key reaction intermediates toward thermoneutral adsorption. Benefiting from the above synergistic electronic and interfacial modulation mechanisms, the amorphous oxide electrode delivers drastically boosted OER catalytic activity. Impressively, at a current density of 100 mA cm^{-2} , the catalyst maintains a low OER overpotential of 309 mV, coupled with a Tafel slope of 43 mV dec^{-1} and a minimal charge-transfer resistance of 2.8Ω . Moreover, outstanding long-term durability with $\sim 96\%$ activity retention is maintained over a continuous 200 h chronopotentiometry test. This facile synthetic strategy, combining in situ growth of nanostructured substrates with wet impregnation of metal precursors, can be generalized to construct other high-performance OER electrocatalysts for practical water splitting applications.

Declarations

Author Contribution

Author contributions Conceptualization: Shu Chang, Cheng Hao; Methodology: Shu Chang; Formal analysis and investigation: Shu Chang; Writing - original draft preparation: Shu Chang; Writing - review and editing: Cheng Hao; Supervision: Cheng Hao.

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Conflicts of interest

There are no conflicts to declare

Data availability

All experimental data supporting the conclusions of this work are available within the supporting information.

Compliance with Ethical Standards

Disclosure of potential conflicts of interest

The authors declare that they have no known competing financial interests or non-financial interests that could have appeared to influence the work reported in this paper.

Research involving Human Participants and/or Animals

No human participants or animals were involved in this study.

Informed consent

Not applicable.

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Figures

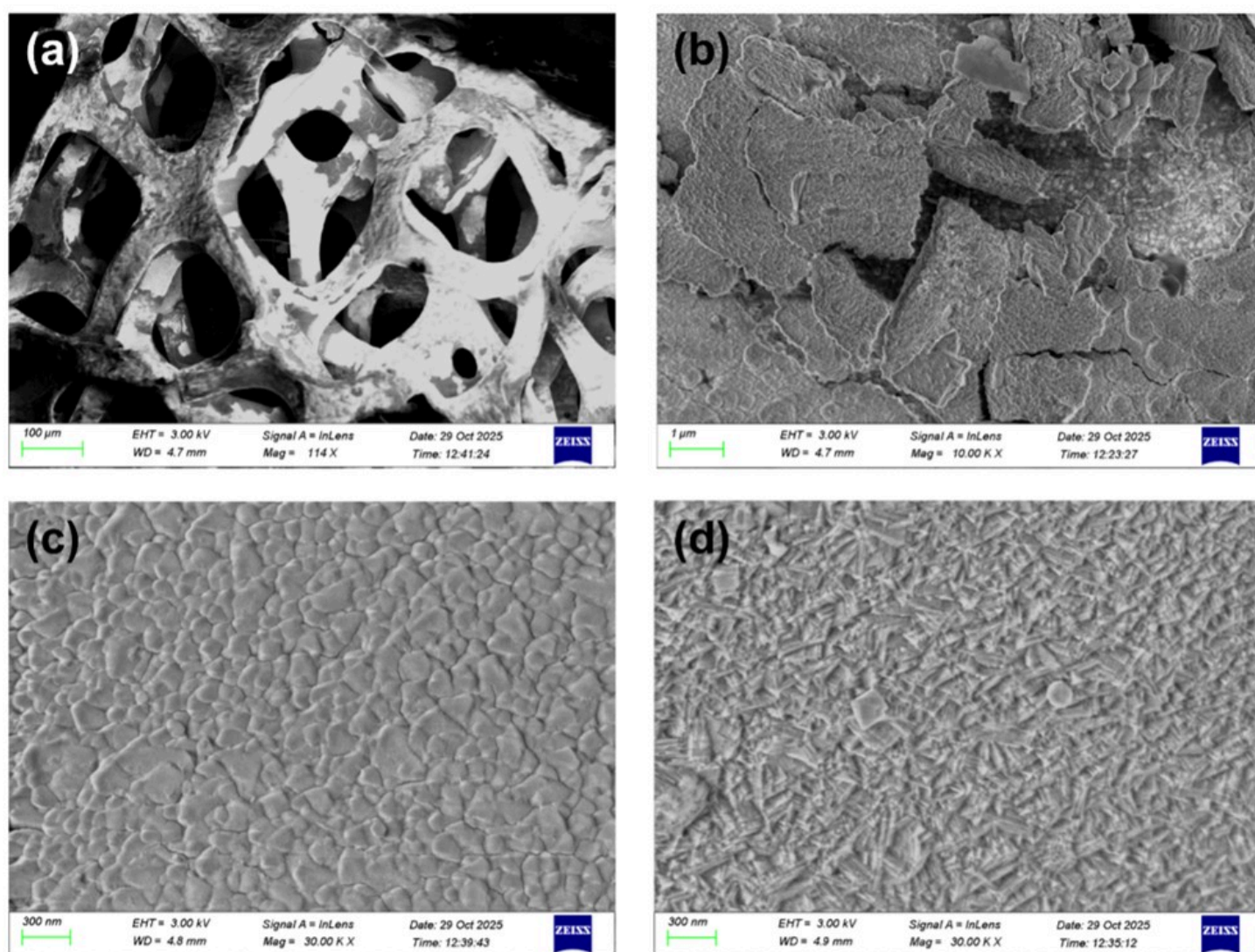


Figure 1

SEM micrograph of $\text{Ce}_{0.5}\text{Fe}_{0.5}\text{O}_{1.75}\text{@NF}$ (a-d) FE-SEM images

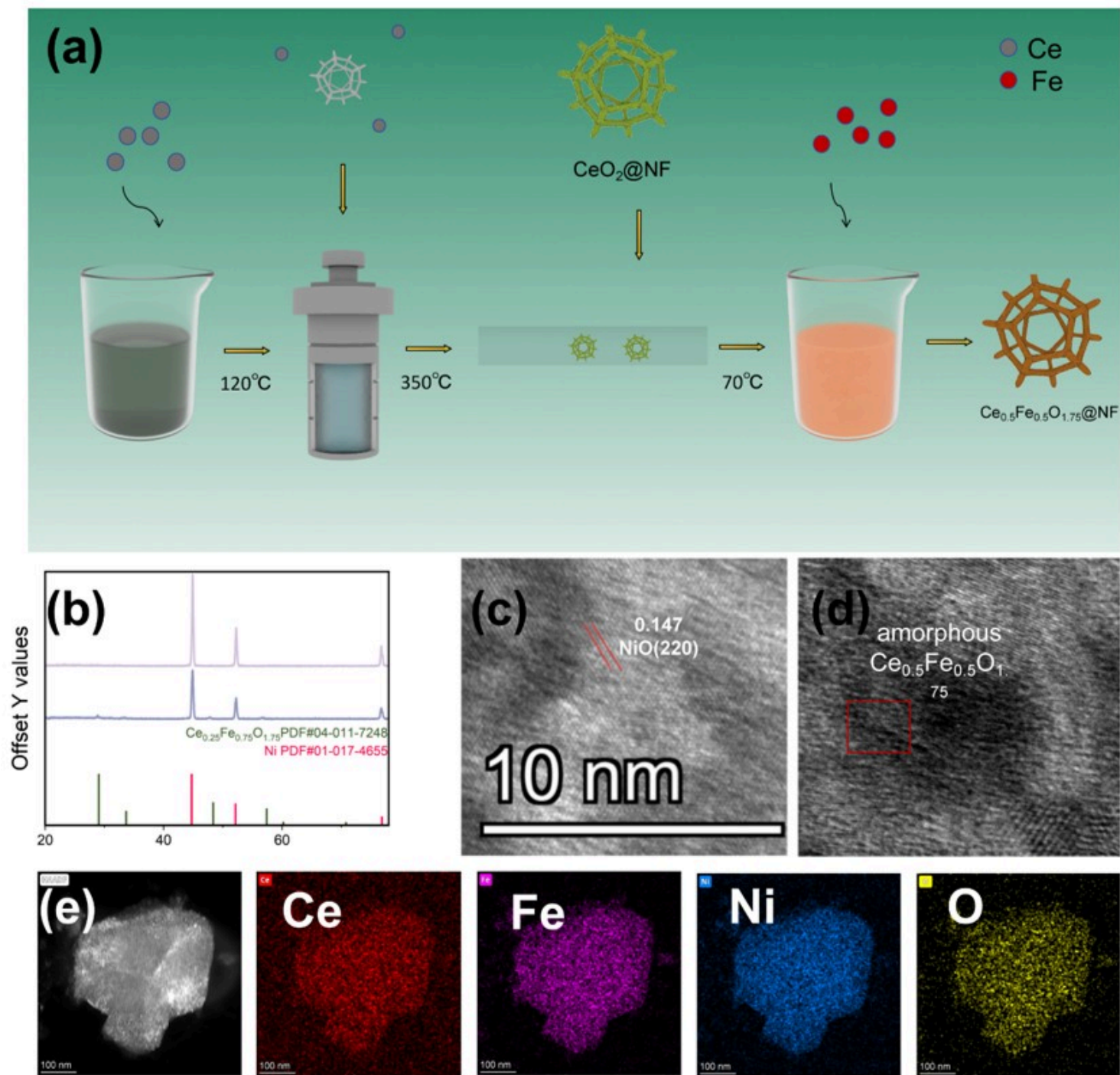


Figure 2

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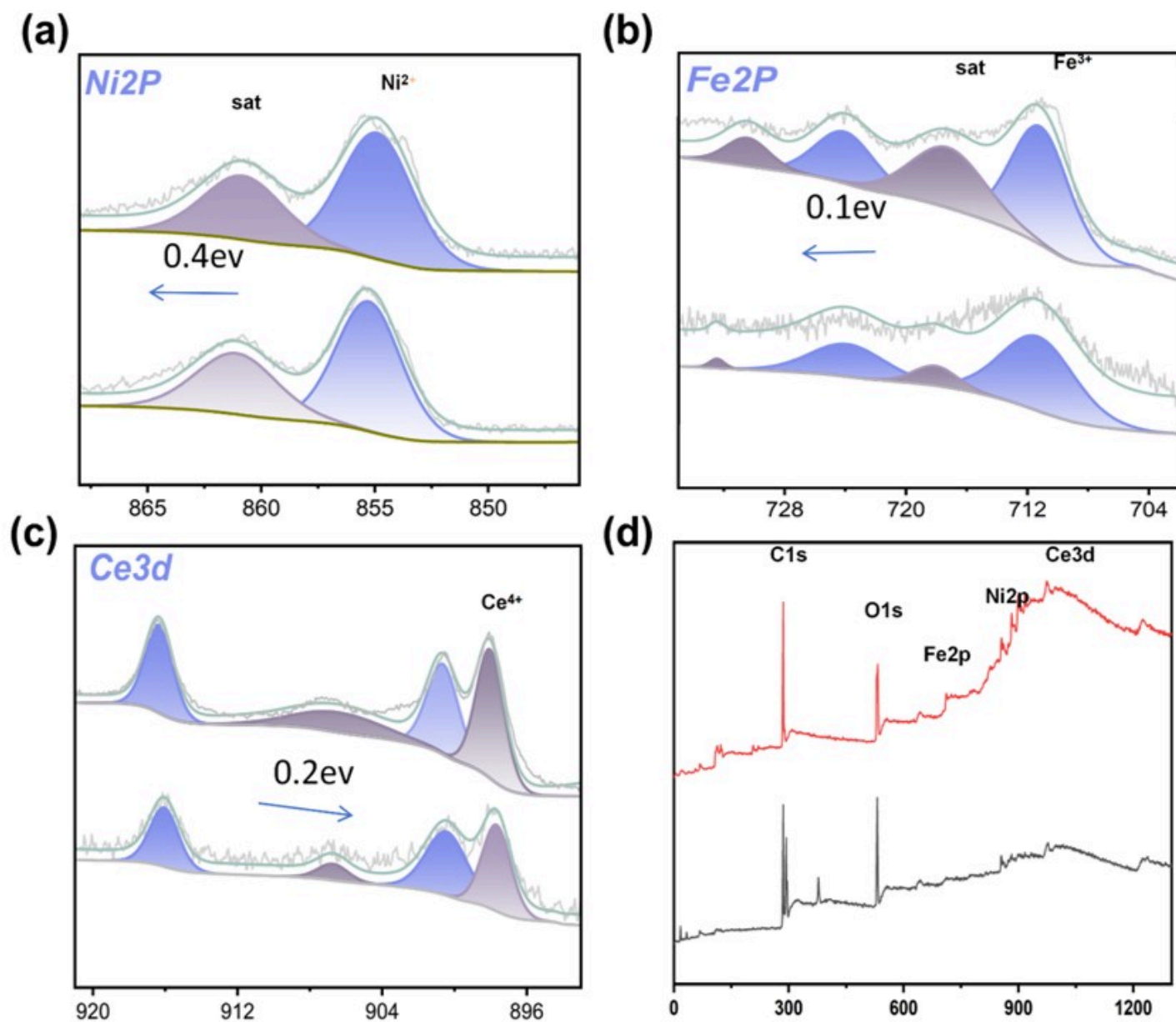


Figure 3

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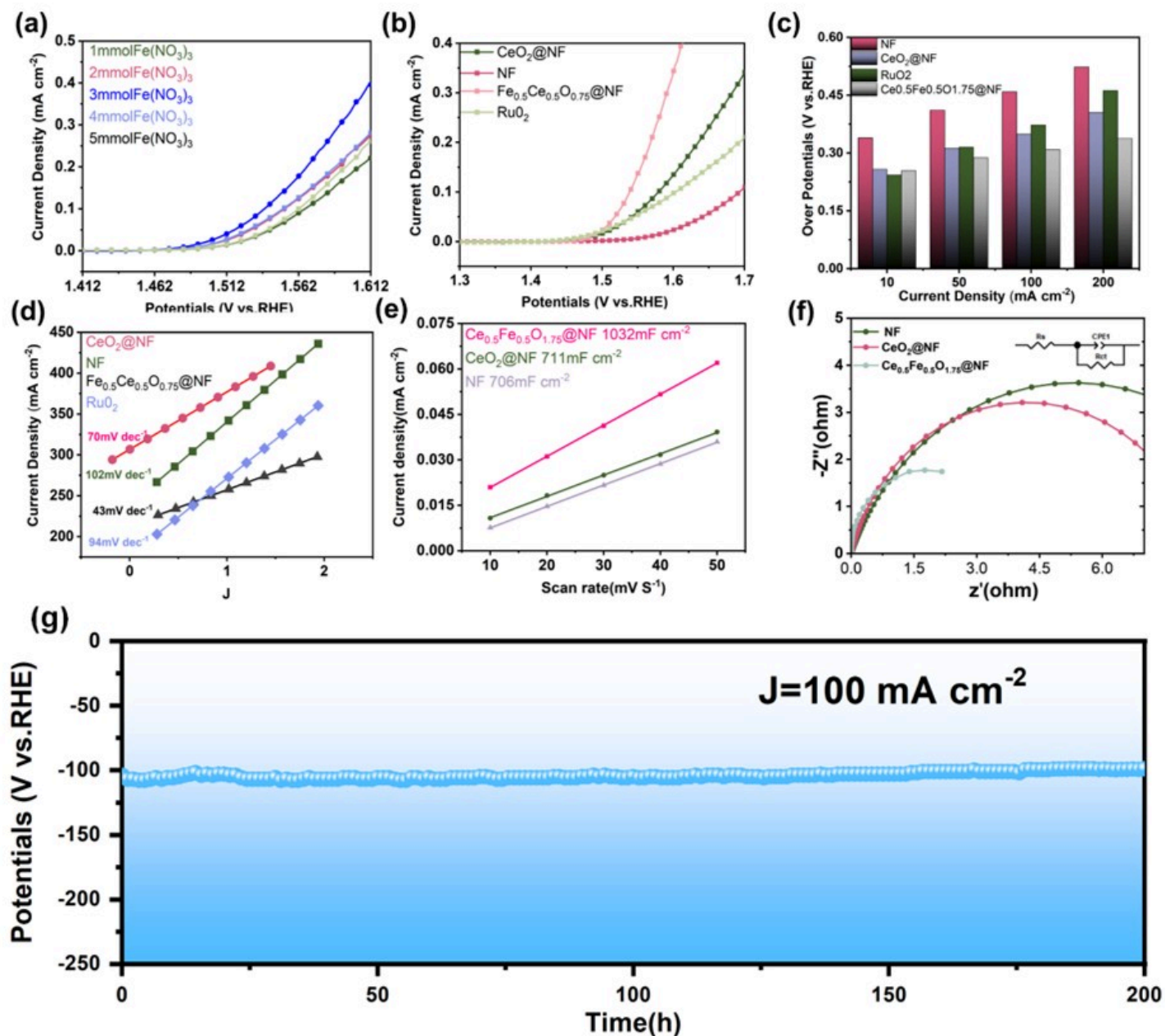


Figure 4

OER electrocatalytic performances of various electrodes in 1 M KOH alkaline electrolyte. (a) LSV polarization curves of catalysts prepared with different $\text{Fe}(\text{NO}_3)_3$ feeding amounts. (b) OER polarization curves of bare NF, $\text{CeO}_2\text{@NF}$, $\text{Ce}_{0.5}\text{Fe}_{0.5}\text{O}_{1.75}\text{@NF}$, and commercial RuO_2 benchmark. (c) Bar chart comparison of overpotentials at varied current densities (10–200 mA cm^{-2}) for NF, $\text{CeO}_2\text{@NF}$, $\text{Ce}_{0.5}\text{Fe}_{0.5}\text{O}_{1.75}\text{@NF}$, and RuO_2 . (d) Tafel plots were acquired based on the LSV curves shown in panel (b). (e) Linear fitting curves of capacitive current versus scan rate to calculate C_{dl} values. (f) Nyquist EIS plots collected at fixed OER overpotential for NF, $\text{CeO}_2\text{@NF}$, and $\text{Ce}_{0.5}\text{Fe}_{0.5}\text{O}_{1.75}\text{@NF}$. (g) Long-term chronopotentiometry stability measurement of $\text{Ce}_{0.5}\text{Fe}_{0.5}\text{O}_{1.75}\text{@NF}$ at 100 mA cm^{-2} for 200 h.

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