

Cassie-impregnated interface design enables efficient O₂ bubble mass transfer in NiCo₂O₄ array electrodes

Zheng Wang¹, Hanxiao Wang²✉, Runlai Jiang¹, Hui Wang¹✉, Jianwei Ren³, Xuyun Wang¹, and Jian Liu²

¹ College of Chemical Engineering, Qingdao University of Science and Technology, Qingdao 266042, China

² State Key Laboratory of Photoelectric Conversion and Utilization of Solar Energy, Qingdao New Energy Shandong Laboratory, Qingdao Institute of Bioenergy and Bioprocess Technology, Chinese Academy of Sciences, Qingdao 266101, China

³ Department of Chemical Engineering, University of Pretoria, cnr Lynnwood Road and Roper Street, Hatfield 0028, South Africa



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ABSTRACT: Three-dimensional array structures are recognized as hydrophilic and gas-repellent architectures for water electrolysis electrodes. In reported studies, the interfacially accelerated bubble detachment process is generally attributed to the role of the gas-liquid-solid three-phase contact line under the ideal Wenzel model, while in-depth investigations into bubble dynamics under the practically occurring Cassie-impregnating mode remain scarce. In this study, a stable Cassie-impregnating wetting state was constructed and optimized via regulation of the depth in NiCo₂O₄ nanowire arrays. As the array depth increases from 1.43 to 4.14 μm, the wettability and gas-repellent performance exhibit a “volcano-type” trend, with a maximum underwater bubble contact angle of 150° and a minimum bubble adhesion force of 9.51 μN. Dynamic bubble statistics reveal that at 100 mA·cm⁻², the optimized NiCo₂O₄ nanowire array delivers a high O₂ bubble generation rate of 107 cm²·s⁻¹ for bubbles with an average size of ~120 μm. The mass transport overpotential for the oxygen evolution reaction (OER) on the NiCo₂O₄ nanowire electrode is reduced to 157 mV at 300 mA·cm⁻². Moreover, the anion exchange membrane water electrolyzer delivers a cell voltage of only 1.81 V at a current density of 1000 mA·cm⁻², while maintaining stable operation for more than 400 h.

KEYWORDS: Cassie-impregnating, O₂ bubble, NiCo₂O₄ nanowire arrays, oxygen evolution reaction, anion exchange membrane water electrolysis

1 Introduction

Within the spectrum of energy technologies, anion exchange membrane water electrolysis (AEMWE) represents an attractive pathway for energy conversion and storage, which leverages hydrogen as a clean carrier while delivering efficient and sustainable performance [1–5]. The electrode is a critical component in AEMWE, and its electrochemical performance decisively influences the electrolysis efficiency of AEMWE systems [6–9]. Numerous studies have demonstrated that self-supporting electrodes composed of a three-dimensional (3D) nickel substrate integrated

with a nickel-based catalytic layer represent the most cost-effective water electrolysis electrodes at present, owing to their stable electrochemical properties, low cost, and easy availability [10, 11]. Moreover, researchers have continuously improved their composition and structure, to enhance the catalytic activity of electrodes and upgrade the performance of AEMWE systems [12–15]. However, in practical water electrolysis operation, high current densities lead to massive gas bubble evolution, which causes severe negative effects such as active site blockage and electrolyte mass transport obstruction at the electrode interface, thereby increasing the polarization of both electrodes. The mass transport overpotential (η_{trans}), governed in part by bubble release dynamics, represents nearly 35% of the overall voltage loss [16–18]. Therefore, besides improving catalyst activity, effective regulation of gas behavior during evolution is equally critical for developing highly efficient and stable water electrolysis electrodes and realizing high-performance AEMWE industrial applications.

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✉ Address correspondence to Hanxiao Wang, wanghx@qibebt.ac.cn; Hui Wang, wangh@qust.edu.cn

Interfaces with nanoarray structures are widely acknowledged as effective electrode architectures for bubble management during gas evolution, also known as superaerophobic interfaces [19–22]. The interpretation of this concept is as follows: Assuming that the interface material exhibits superaerophilic properties and the array is filled with gas, based on the Cassie–Baxter model, the high roughness of the nanoarray structure enables nascent nanoscale bubbles to be “pinned” and stabilized at the electrode interface, followed by rapid growth [23]. Meanwhile, the rough interface disrupts the gas–liquid–solid three-phase contact region, enhancing bubble contraction and enabling bubbles to detach from the interface at smaller sizes, thus accelerating bubble dynamics on the electrode (as illustrated on the left side of Fig. 1(a)) [24–26]. This model satisfactorily explains the rapid detachment of small-sized bubbles from array electrodes. Structure engineering serves as a core strategy to overcome bubble blockage and mass transport limitations in oxygen evolution reaction (OER) electrodes at high current densities. Significant progress has been made in multilevel morphology, three-dimensional channels, and interface functionalization designs. Constructing hierarchical nanocone–nanoneedle structures strengthens the local electric field via the high-curvature effect, accelerates bubble detachment, and simultaneously improves OER kinetics and the stability of AEMWE devices [27]. Laser-fabricated three-dimensional macro–micro synergistic electrodes integrate superwetting interfaces with high-efficiency mass transport channels, remarkably reducing concentration polarization and satisfying the requirements of industrial high-current operation [28]. Furthermore, pump-like

capillary array three-dimensional structures actively guide bubble ejection and suppress interfacial bubble accumulation from a hydrodynamic perspective [29]. Although these structural designs effectively optimize bubble dynamics and mass transport performance, most existing studies focus on morphology, channel, and surface modification regulations, and generally interpret interfacial mechanisms based on the ideal Wenzel wetting model. Nevertheless, in practical materials, for micro/nanocomposite structures with high roughness and relatively low surface energy, liquid remains at the solid–gas contact interface due to capillary forces, resulting in a coexisting state of solid–liquid contact and gas–liquid contact, namely the Cassie-impregnating mode (as shown on the right side of Fig. 1(a)). Furthermore, the fine structure of array motifs on the electrode, such as motif spacing and motif size, influences the Cassie-impregnating state and alters bubble dynamics at the interface. For instance, during the OER catalyzed by “ridge-like” nickel array electrodes supported on nickel foils, the spacing of the “ridge” motifs is proportional to the size of oxygen bubbles. A ridge spacing of 200 μm facilitates the growth and detachment of O_2 bubbles, and the electrolyte within the spacing exhibits higher fluidity, promoting mass transport and achieving the maximum gas evolution efficiency [30]. Similar results were reported in studies on water electrolysis hydrogen evolution electrodes with “micron-scale Ni pillar” array structures [31]. Additionally, a higher pillar array height increases H_2 molecular concentration within the enclosed narrow gaps, facilitating nucleation and benefiting H_2 bubble generation [31]. These results indicate that regulating the Cassie-impregnating state

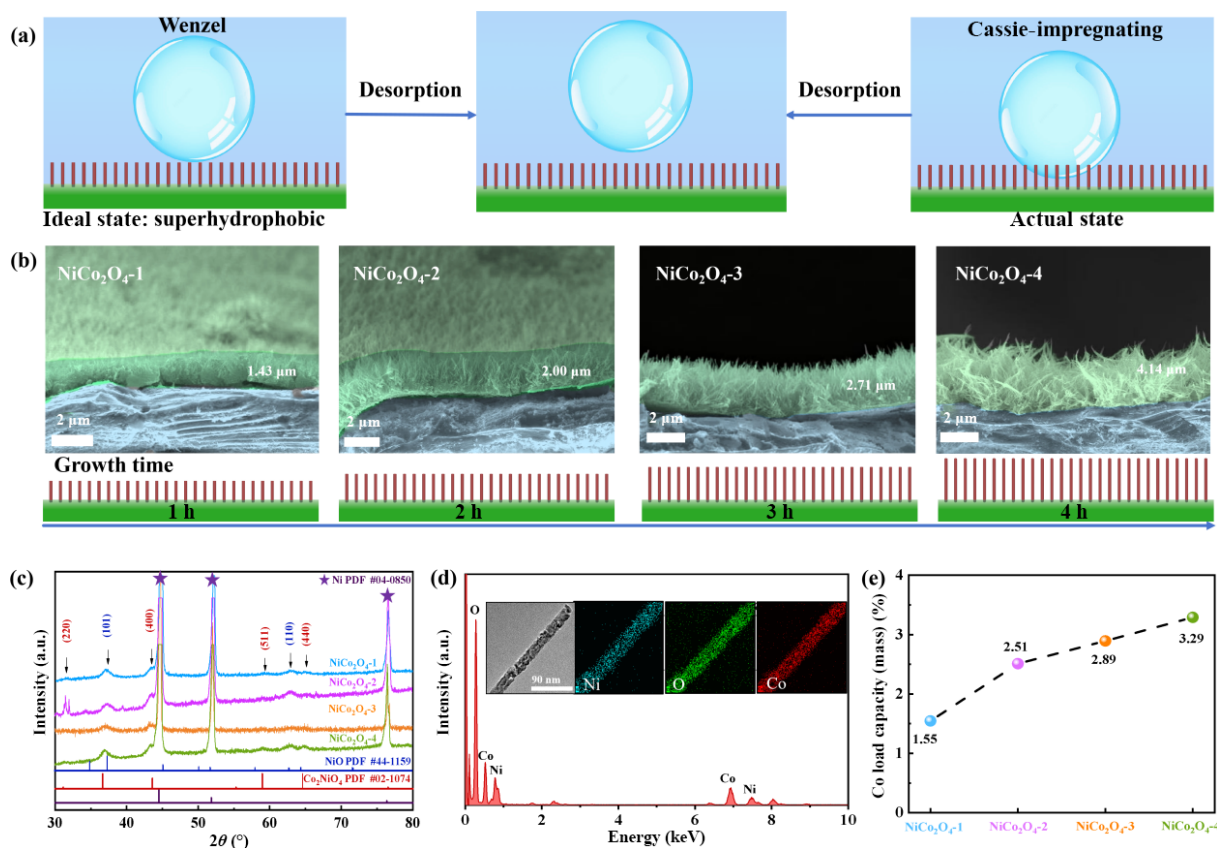


Figure 1 Simulated bubble behavior and structural/compositional characterization of electrodes. (a) Two states of bubble detachment from the material surface. (b) SEM images of NiCo_2O_4 samples and corresponding nanowire growth heights. (c) XRD patterns of NiCo_2O_4 samples. (d) EDS spectrum of NiCo_2O_4 -3, with inset showing dark-field STEM and corresponding elemental mapping images. (e) Co content in NiCo_2O_4 samples.

by tuning the fine structure of motifs in array electrodes can further improve bubble dynamics at the electrode interface, enhance interfacial mass transport, and boost AEMWE electrolysis efficiency.

Building on this understanding, this study regulates the Cassie-impregnating state at the interface of the array electrode by controlling the depth of the NiCo_2O_4 array structures. The wettability and gas-repellent performance show a “volcano-type” variation trend, with a maximum underwater bubble contact angle of 150° and a minimum bubble adhesion force of $9.51\ \mu\text{N}$. Quantitative evaluation of *in-situ* high-speed bubble imaging was conducted using a deep learning-based bubble recognition model reveals that at $100\ \text{mA}\cdot\text{cm}^{-2}$, the optimized Cassie-impregnating interface achieves an ultrahigh O_2 bubble generation rate of $107\ \text{cm}^3\cdot\text{s}^{-1}$ with an average bubble size of $\sim 120\ \mu\text{m}$, accompanied by an electrode potential fluctuation of only 6 mV. At a current density of $300\ \text{mA}\cdot\text{cm}^{-2}$, the η_{uns} associated with the OER on the NiCo_2O_4 nanowire electrode decreases to 157 mV. An AEMWE single cell assembled with symmetric electrodes delivers a low overall water splitting voltage of 1.81 V at $1000\ \text{mA}\cdot\text{cm}^{-2}$ and operates stably for over 400 h.

2 Experimental

Synthesis of NiCo_2O_4 nanowire arrays: A precursor solution was prepared by dissolving 8 mmol of $\text{Co}(\text{NO}_3)_2\cdot 6\text{H}_2\text{O}$, 4 mmol of $\text{Ni}(\text{NO}_3)_2\cdot 6\text{H}_2\text{O}$, and 12 mmol of urea in a mixed solvent containing 30 mL of deionized water and 30 mL of ethanol. After achieving a homogeneous mixture through stirring, the solution was transferred into a 100 mL Teflon-lined stainless-steel autoclave. Subsequently, a nickel foam substrate ($2\ \text{cm} \times 3\ \text{cm}$), previously pretreated via ultrasonication in 1 M HCl to remove surface oxides, was immersed in the solution. The hydrothermal process was conducted at $150\ ^\circ\text{C}$ for varying durations of 1, 2, 3, and 4 h. Following the reaction, the resulting Ni-Co precursor was subjected to calcination in air at $350\ ^\circ\text{C}$ for 2 h with a heating rate of $5\ ^\circ\text{C}\cdot\text{min}^{-1}$, yielding NiCo_2O_4 nanowire arrays. The obtained samples were labeled as NiCo_2O_4 -1, NiCo_2O_4 -2, NiCo_2O_4 -3, and NiCo_2O_4 -4, corresponding to hydrothermal treatment times of 1, 2, 3, and 4 h, respectively.

3 Results and discussion

3.1 Preparation of nanowire arrays and regulation of array depth

NiCo_2O_4 nanowire array structures supported on nickel foam were fabricated via a two-step hydrothermal and thermal treatment process. The growth height of NiCo_2O_4 nanowire motifs was tuned by varying the hydrothermal reaction time (1, 2, 3, and 4 h), realizing the regulation of NiCo_2O_4 nanowire array depth (as shown at the bottom of Fig. 1(b)). Cross-sectional scanning electron microscopy (SEM) images of the NiCo_2O_4 nanoarrays visualize this process. As displayed in Fig. 1(b), with prolonged growth time, the NiCo_2O_4 nanowire array depth increases from 1.43 to $4.14\ \mu\text{m}$. By comparing the growth time and height of nanowires, the nanoarray depth does not increase linearly, which arises from the non-linear decrease of precursor concentration in the reaction system and measurement errors. Top-view SEM images of a series of NiCo_2O_4 nanowire arrays (Fig. S1 in the Electronic Supplementary Material

(ESM)) confirm that the arrays consist of NiCo_2O_4 nanowires. Individual motifs do not exhibit perfect vertical alignment but feature a tapered tip morphology. Dozens of adjacent nanowires cluster at the top and interlace into bundles. Moreover, the clustering becomes more pronounced as the nanowire length increases, which is another reason for the non-linear increase of nanoarray depth with time. Furthermore, severe clustering prevents unlimited growth of nanowire array depth with time, as shown in Fig. S2 in the ESM. Concerning the diameters of individual nanowires, as labeled in Figs. S1(a)–S1(d) in the ESM, they remain relatively uniform across NiCo_2O_4 -1 to NiCo_2O_4 -4, with an average size of 30–40 nm. The X-ray diffraction (XRD) profiles of the four samples (Fig. 1(c)) exhibit distinct diffraction peaks corresponding to the spinel NiCo_2O_4 structure at $2\theta = 36.7^\circ$, 44.6° , 59.0° , and 64.7° , corresponding to the (311), (400), (511), and (440) planes, as well as characteristic peaks of face-centered cubic NiO at $2\theta = 37.0^\circ$ and 62.9° , indexed to the (101) and (110) planes, respectively. This indicates that all four samples are composed of NiCo_2O_4 and NiO crystalline phases with identical phase compositions. The significantly higher intensity of the 37.0° peak for NiCo_2O_4 -2, compared to the other samples, is because this peak is an overlap of the NiCo_2O_4 (311) and NiO(101) reflections. At a hydrothermal time of 2 h, the NiCo_2O_4 (311) crystal plane exhibits the strongest preferred orientation. Simultaneously, the instantaneous relative content of NiO, generated by the dissolution of Ni^{2+} from the nickel foam substrate, is at its highest. The diffraction signals of the two phases overlap, and additionally, the lattice stress in this sample is minimized, resulting in a sharper peak shape. Furthermore, no obvious characteristic diffraction peak of NiO was observed near 35° , which is because the intrinsic intensity of the NiO diffraction peak at 35° is extremely low and is completely overshadowed by the strong main peak of the spinel NiCo_2O_4 . However, the characteristic diffraction peak of the NiO(110) crystal plane can be clearly identified at 62.9° , directly confirming the presence of the NiO phase. Additionally, the X-ray photoelectron spectroscopy (XPS) characterization results (Fig. S3 in the ESM) show that Ni is predominantly present in the Ni^{2+} valence state, which perfectly matches the characteristic binding energy of NiO, further verifying the NiO phase from the perspective of elemental valence states. Transmission electron microscopy (TEM) was utilized to examine the detailed structural features of NiCo_2O_4 -3 as a representative sample. Individual NiCo_2O_4 motifs appear as nanowires with a diameter of $\sim 40\ \text{nm}$, consistent with SEM observations. Further magnification reveals that the nanowires are assembled from aggregated particles with irregular interparticle pores. Its energy dispersive X-ray spectroscopy (EDS) spectrum (Fig. 1(d)) confirms that the as-prepared sample consists of Ni, Co, and O elements. XPS analysis indicates that the Ni and Co atoms are present in combined chemical states (Fig. S3 in the ESM). Dark-field scanning TEM (STEM) elemental mapping (inset of Fig. 1(d)) reveals a homogeneous distribution of Ni, Co, and O elements across the nanowire structure, demonstrating excellent structural homogeneity of the material. Meanwhile, the Co loading was measured by inductively coupled plasma, and the results in Fig. 1(e) show that the catalytically active Co content first increases and then stabilizes with reaction time, reaching a maximum (3.29 wt.%) at NiCo_2O_4 -4 before plateauing, which is consistent with the increasing trend of array depth. By integrating XPS, EDS mapping, and inductively coupled plasma (ICP) characterizations, the sample phase is verified from multiple dimensions as NiCo_2O_4 and NiO.

3.2 Characteristics of the solid-liquid-gas three-phase interface of samples

During water electrolysis, the three-phase interface characteristics formed by the electrode-electrolyte-bubble system, including wettability and bubble adhesion force, are key factors affecting electrolysis efficiency [32, 33]. Accordingly, the three-phase interfacial properties of the NiCo_2O_4 series of samples were systematically characterized. Figures 2(a)–2(d) present optical images of underwater bubble contact angles for NiCo_2O_4 samples. As shown in Fig. 2(a), underwater bubbles spread on the surface of NiCo_2O_4 -1 with a contact angle of 122° , indicating certain affinity between bubbles and the interface. For NiCo_2O_4 -2 (Fig. 2(b)), the underwater bubble profile becomes more spherical, and the bubble contact angle rises to 140° . For NiCo_2O_4 -3 (Fig. 2(c)), underwater bubbles form an almost perfect sphere on the surface, with the bubble contact angle reaching a maximum of 150° , exhibiting superaerophobic behavior (bubble contact angle $> 150^\circ$). For NiCo_2O_4 -4 (Fig. 2(d)), the spherical profile of underwater bubbles slightly collapses, and the bubble contact angle drops to 133° , with weakened bubble repellence compared to NiCo_2O_4 -3 but still higher than NiCo_2O_4 -1, representing moderate aerophobicity. Overall, from NiCo_2O_4 -1 to NiCo_2O_4 -3, the aerophobicity of the samples continuously increases with array depth. After NiCo_2O_4 -3 achieves superaerophobicity, further increasing the array depth leads to a decline in aerophobicity for NiCo_2O_4 -4. These outcomes highlight the superior aerophobic performance of the 3D array interface is closely related to its fine structure and cannot be simply attributed

to a vague aerophobic interface based on the three-phase contact line. According to the Cassie-impregnating model (Figs. 2(e)–2(h)), with increasing motif height (h), the contact line between the bubbles and the nanowire tips becomes longer, accompanied by an increase in the curvature of the solid-liquid-gas interface. Such topological changes of the contact line enhance the vertical constraint on bubbles and weaken the horizontal spreading tendency, resulting in an increased bubble contact angle and higher surface repellence toward bubbles. When the array depth reaches approximately $2.7\ \mu\text{m}$, the bubble contact angle reaches its maximum, and the array interface achieves the optimal Cassie-impregnating state. With a further increase in array depth, the distortion of the three-phase contact line between nanowire motifs intensifies, weakening the vertical constraint on bubbles and enhancing horizontal spreading, which manifests as a reduced bubble contact angle. Therefore, topological changes of the three-phase contact line induced by variations in the depth of NiCo_2O_4 nanowire array motifs significantly modify the interfacial aerophobic characteristics. Combining the classical Cassie-Baxter three-phase wetting model with the array geometry, this work further derives the quantitative relationship among array depth, three-phase contact line length, and bubble contact angle, elucidating the regulatory mechanism of microstructure evolution on interfacial bubble wettability. The bubble contact angle at the underwater gas-solid-liquid composite interface satisfies the classical Cassie equation for underwater bubble wetting

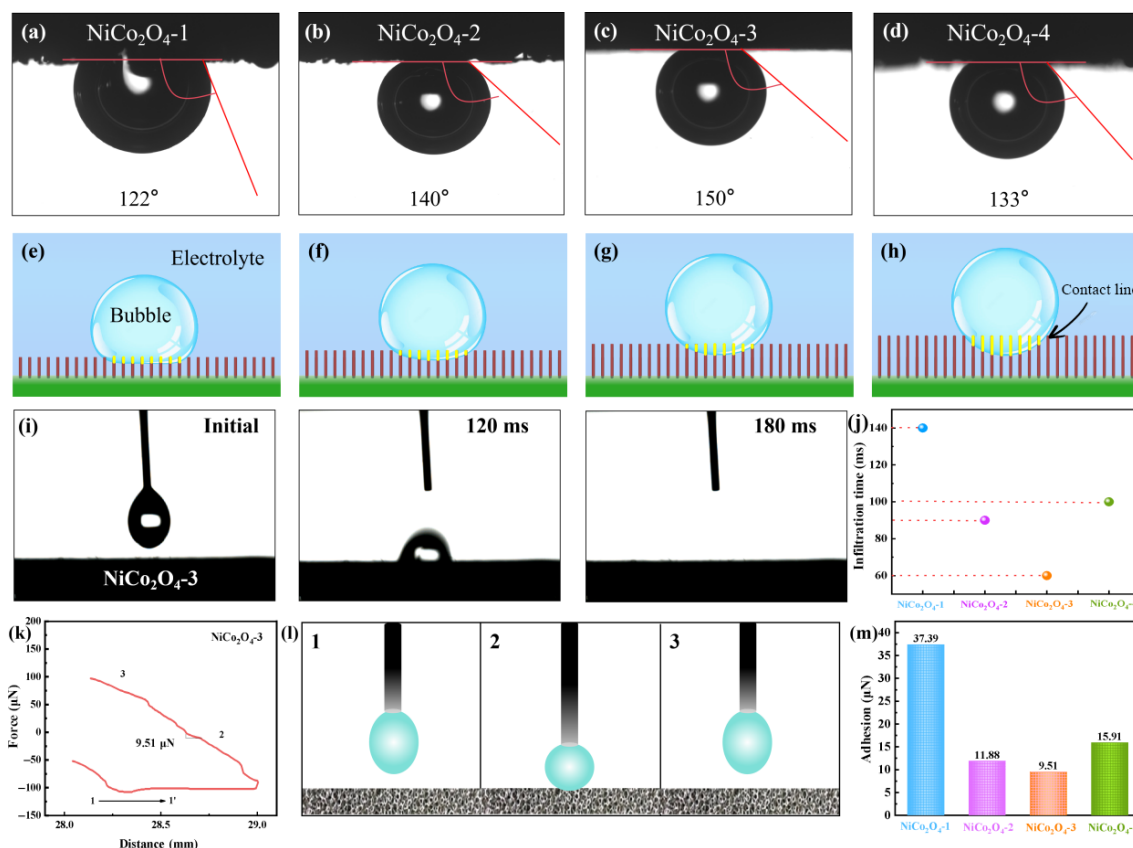


Figure 2 Surface wettability and bubble adhesion force of electrodes. ((a)–(h)) Optical images and corresponding schematic diagrams of underwater bubble contact angles for ((a) and (e)) NiCo_2O_4 -1, ((b) and (f)) NiCo_2O_4 -2, ((c) and (g)) NiCo_2O_4 -3, and ((d) and (h)) NiCo_2O_4 -4 (schematic proportion for clear interface display, not strict 1:1 scale). (i) Wettability test of NiCo_2O_4 -3. (j) Wetting time of different NiCo_2O_4 samples. (k) Measurement of underwater bubble adhesion force for NiCo_2O_4 -3. (l) Schematic diagram of different stages of bubbles in the test system. (m) Variation of adhesion force for NiCo_2O_4 samples.

$$\cos\theta = f_s \cos\theta_s - f_v \quad (1)$$

where θ is the macroscopic bubble contact angle, f_s is the solid contact area fraction of the nanowires, θ_s is the intrinsic bubble contact angle of the bare nanowire solid material, and f_v is the fraction of trapped gas voids at the interface, satisfying $f_s + f_v = 1$. The total three-phase contact line length per unit area is defined as L . Within a reasonable growth range, increasing the array depth h increases the number of upright nanowire boundaries, and the three-phase contact line length increases approximately linearly.

$$L = kh \quad (2)$$

where k is a proportionality constant related to the nanowire array density. When the array depth exceeds a critical threshold ($h > 2.7 \mu\text{m}$), excessive growth induces nanowire agglomeration and structural collapse. The effective boundary of individual nanowires becomes shielded and diminished in utilization. The three-phase contact line no longer continues to increase with depth but instead decreases.

$$L = kh - \Delta L_{\text{cluster}} \quad (3)$$

where $\Delta L_{\text{cluster}}$ represents the loss of three-phase contact line length caused by nanowire agglomeration and distortion. Combining the geometric relationship with the wetting equation yields

$$\cos\theta = (1 - f_v(L)) \cos\theta_s - f_v(L), \text{ with } L = f(h) \quad (4)$$

In summary, the nanowire array depth regulates the geometric length and connectivity of the three-phase contact line, thereby modifying the interfacial gas storage capacity and composite wetting state. An array depth of $2.7 \mu\text{m}$ represents the optimal structural critical point. Excessive growth-induced nanowire agglomeration disrupts the continuous contact line topology, reduces the gas-solid composite effect at the interface, and ultimately leads to a significant attenuation of the surface gas repellency.

Figure 2(i) and Fig. S4 in the ESM display the dynamic wetting process of water droplets on NiCo_2O_4 sample interfaces, and the time required for complete wetting of different samples is quantitatively compared in Fig. 2(j). As shown, when the array depth increases from 1.43 to $2.7 \mu\text{m}$ (from NiCo_2O_4 -1 to NiCo_2O_4 -3), the complete wetting time of water droplets gradually decreases. When the depth exceeds $2.7 \mu\text{m}$ (NiCo_2O_4 -4), the wetting time slightly increases due to nanowire agglomeration. NiCo_2O_4 -3 exhibits the shortest wetting time of only 60 ms , demonstrating outstanding superhydrophilicity, which is opposite to its superaerophobic feature. A shorter wetting time indicates that the electrolyte can rapidly spread and fully penetrate into the array interior, achieving efficient three-phase interface contact and mass transport.

Furthermore, the dynamic behavior of bubbles at the array interface was observed. As shown in Fig. 2(k), the adhesion force curve during the separation between NiCo_2O_4 -3 and bubbles is presented; adhesion force curves for other samples are shown in Fig. S5 in the ESM. As illustrated in the bubble interaction stage diagram (Fig. 2(l)): Process 1 corresponds to the electrode approaching the bubble, process 2 represents interfacial interaction, and process 3 denotes electrode-bubble separation. Quantitative comparison (Fig. 2(m)) reveals that NiCo_2O_4 -3 possesses the lowest bubble adhesion force ($\sim 9.51 \mu\text{N}$), significantly lower than that of

other samples (11.88 – $37.39 \mu\text{N}$). This mechanical quantitative result directly confirms that the Cassie-impregnating interface constructed with the optimal array depth can maximally weaken gas-solid interfacial interactions, lower the bubble detachment energy barrier, and realize efficient and rapid bubble detachment. This fundamentally optimizes bubble dynamics at the electrode interface and provides core support for improving water electrolysis mass transport efficiency.

3.3 Influence of array depth on O_2 bubble dynamics at the electrode interface

Changes in the gas-liquid-solid three-phase interface characteristics induced by NiCo_2O_4 array depth exert a significant influence on interfacial O_2 bubble dynamics. By combining a high-speed imaging system with deep learning techniques, the full-cycle behavior of bubbles from generation and detachment to gas production efficiency was systematically quantified, further confirming the effect of array depth on bubble dynamics. High-speed imaging in Fig. 3(a) enables direct observation of the NiCo_2O_4 -3 electrode surface and provides visual monitoring of bubble generation. The recorded bubble videos are available in Videos S1–S4 in the ESM, corresponding to the NiCo_2O_4 -1, NiCo_2O_4 -2, NiCo_2O_4 -3 and NiCo_2O_4 -4, respectively. As described in the ESM, a trained deep learning bubble model with detail provided in Fig. S7 in the ESM was employed to perform bubble recognition and quantitative statistics on *in-situ* gas evolution videos of the electrode, and the raw bubble data generated by the model without any mathematical calibration were used for subsequent statistical analysis. Figure 3(b) shows the time-resolved bubble density and cumulative bubble volume curves at the NiCo_2O_4 -3 electrode interface. The bubble density rises rapidly with time and stabilizes after 20 s . This observation reflects the rapid activation of active sites in the initial reaction stage and the achievement of dynamic equilibrium between bubble generation and detachment in the later stage. Bubble temporal density, defined as the number of bubbles generated per unit area over a given time interval, is quantitatively evaluated in Fig. 3(c): The actual O_2 bubble generation rate on the NiCo_2O_4 -3 electrode reaches as high as $106.67 \text{ cm}^{-2}\text{s}^{-1}$, much higher than that of NiCo_2O_4 -1 ($11.54 \text{ cm}^{-2}\text{s}^{-1}$, Video S1 in the ESM), NiCo_2O_4 -2 ($78.94 \text{ cm}^{-2}\text{s}^{-1}$, Video S2 in the ESM), and NiCo_2O_4 -4 ($57.42 \text{ cm}^{-2}\text{s}^{-1}$, Video S4 in the ESM). The steady-state bubble density after 20 s is 1400 cm^{-2} , remarkably higher than that of the other three samples, implying a higher bubble nucleation probability at the NiCo_2O_4 -3 electrode interface. It should be noted that although the electrode interfacial bubble generation rate is not completely equivalent to the bubble nucleation rate, it exhibit a positive correlation. These data indicate that the bubble nucleation probability on NiCo_2O_4 nanowire array electrodes follows a non-monotonic relationship (first increasing then decreasing) with array depth, with an inflection point corresponding to the critical array height.

Earlier investigations have indicated that the stochastic nature of the rate of bubble nucleation ($\text{J}\cdot\text{s}^{-1}$) at platinum electrode of nanometer size interfaces is affected by multiple factors including electrode interfacial microenvironment, electrolyte, current density, and temperature, whose coupling effect is expressed by Eq. (5) [34, 35]

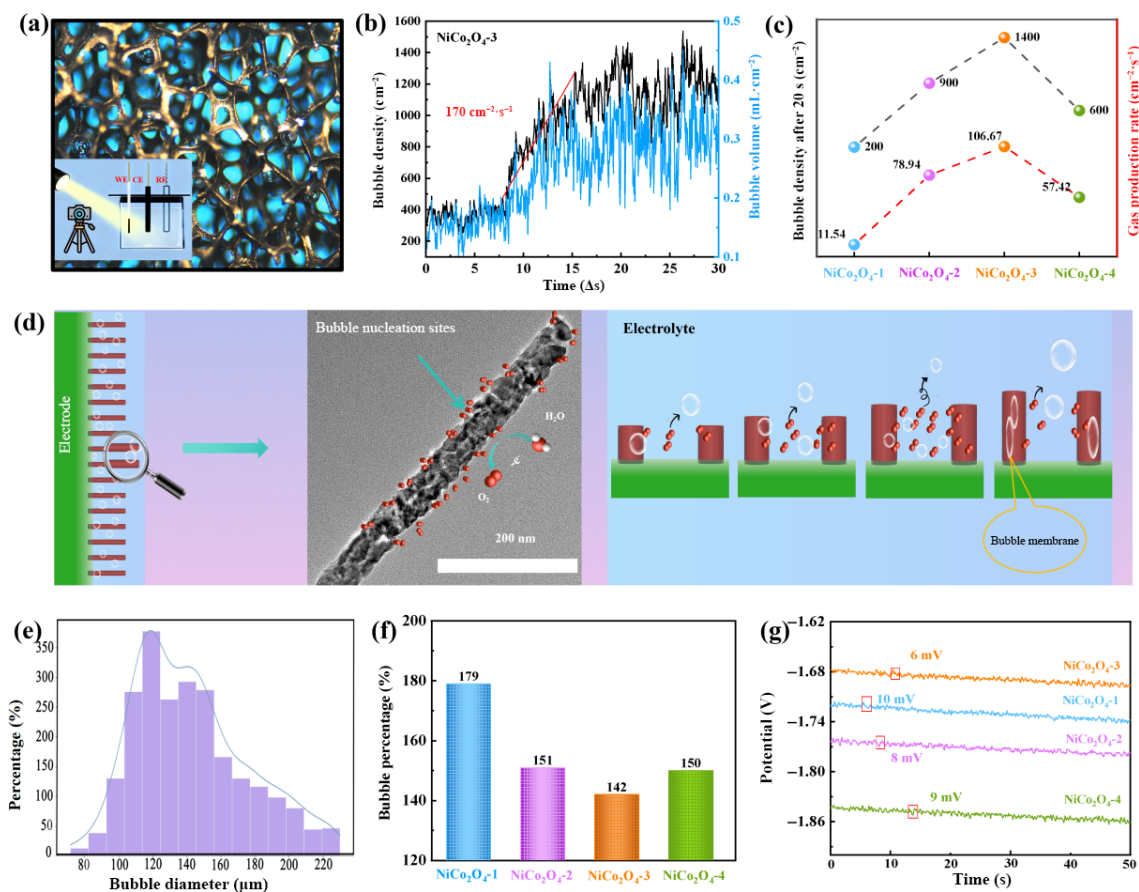


Figure 3 Electrode bubble dynamics and electrolysis stability. (a) Photographs of sample surfaces captured by operando high-speed imaging. (b) Time-resolved bubble density and cumulative bubble volume at the NiCo₂O₄-3 electrode interface. (c) Average bubble density and gas generation rate of NiCo₂O₄ samples from 20 to 30 s. (d) Schematic illustration of bubble nucleation and detachment on electrodes. (e) Bubble size distribution detached from the NiCo₂O₄-3 electrode. (f) Comparison of average bubble size distributions of NiCo₂O₄ samples from 20 to 30 s. (g) CP curves of NiCo₂O₄ samples at 1000 mA·cm⁻².

$$J = J_0 \exp \left(\frac{-16\pi\gamma^3(\theta)}{3kT \left(\frac{C_{O_2}^{\text{surf}}}{K_H} - P_{\text{ambient}} \right)^2} \right) \quad (5)$$

where k corresponds to the Boltzmann constant and T refers to the temperature. The pre-exponential term J_0 accounts for the stochastic and molecular-scale growth of nuclei and can be approximated as constant with respect to current density (i). γ refers to the gas-liquid interfacial tension, which is about 0.072 N·m⁻¹ for a water system at room temperature. $\phi(\theta)$ is the geometric factor for heterogeneous nucleation, $\phi(\theta) = \frac{(1 - \cos\theta)^2(2 - \cos\theta)}{4}$, and θ is the contact angle of the bubble nucleus. $C_{O_2}^{\text{surf}}$ is the dissolved oxygen concentration on the electrode surface, K_H is the Henry's law constant, used to relate the partial pressure of a gas to its concentration in solution, and $C = PK_H \cdot P_{\text{ambient}}$ is the environmental pressure, usually standard atmospheric pressure. In this work, the array electrodes are composed of a collection of individual nanowires, with the diameter of the nanowires around 50 nm. Based on the research of the White's group, we divide the single nanowire array into individual nanoscale electrodes. The probability of gas nucleation on the surface of a single unit conforms to Eq. (1) with respect to the gas molecule concentration. The relationship between gas molecule concentration and its electrode structure is

$$C_{O_2}^{\text{surf}} = \frac{i}{4nFD_{O_2}\alpha} \quad (6)$$

where n represents the number of electrons transferred per H₂ molecule, F is the Faraday constant, D_{O_2} denotes the diffusion coefficient of O₂ (4.5×10^{-5} cm²·s⁻¹), and α corresponds to the effective radius of the electrode. In this work, the array electrode is a collection of individual nanoelectrodes, and the diffusion of O₂ molecules is different from that of a single nanoelectrode, being affected by array structures such as the density and depth of array units.

Combining Eqs. (5) and (6), under the same electrolysis system, electrode composition/structure, and current density in this work, the variation in bubble nucleation rate is attributed to the effect of array depth on the local O₂ molecular concentration in solution, also known as the change in O₂ supersaturation.

As illustrated in Fig. 3(d), gaseous O₂ molecules generated by the gas evolution reaction accumulate and nucleate at array nucleation sites such as nanowire pits (middle part of Fig. 3(d)). When the array depth is shallow, electrolyte convection/diffusion is strong, gaseous molecules diffuse rapidly, local O₂ concentration is low, and the probability of gas molecule aggregation and nucleation is small. With increasing array depth, gaseous molecule diffusion is restricted, gaseous molecules accumulate, local O₂ concentration rises, supersaturation significantly increases, and nucleation probability greatly improves. When the array depth is excessively

high (rightmost part of Fig. 3(d)), excessive gas retention forms a gas film that blocks the array, newly generated gaseous molecules are trapped, the solid-liquid interface is drastically reduced, H_2O decomposition on array motifs is suppressed, the amount of generated O_2 gas molecules drops sharply, the interfacial dissolved O_2 concentration rapidly decreases, and nucleation probability declines accordingly. Collectively, data in Fig. 3(b) and Fig. S6 in the ESM suggest that array electrode depth influences interfacial gas molecule supersaturation, thereby affecting bubble nucleation probability with an inflection point, pointing out a direction for further optimizing bubble dynamics at array electrode interfaces. The array depth in the Cassie-impregnating interface also exerts a significant influence on the bubble detachment process. The bubble size distribution of NiCo_2O_4 -3 shows a unimodal concentrated feature (Fig. 3(e)), with the peak corresponding to a size of $\sim 120\ \mu\text{m}$, indicating massive synchronous detachment of bubbles at a specific size, which effectively avoids excessive bubble growth and long-term coverage of active sites, thereby reducing mass transport resistance and ensuring sustained and efficient reaction progress. Quantitative comparison (Fig. 3(f) and Fig. S6 in the ESM) further reveals that NiCo_2O_4 -3 has the smallest average bubble size and the highest bubble density. Bubble size is a key indicator reflecting the synergy between bubble growth and detachment [36], and small-sized and high-frequency bubble detachment significantly reduces bubble residence time on the electrode surface. As shown in Fig. 3(g), in the chronopotentiometry (CP) curve at $1000\ \text{mA}\cdot\text{cm}^{-2}$, the voltage fluctuation of NiCo_2O_4 -3 caused by bubble detachment is only 6 mV, remarkably lower than that of other samples (8–10 mV). This indicates more stable bubble generation and detachment, effectively reducing the loss in electrolysis efficiency caused by bubble accumulation.

3.4 Mass transport performance of NiCo_2O_4 nanowire array electrodes

Regulation of the Cassie-impregnating interface significantly promotes gas evolution efficiency at the array electrode interface. As shown in Fig. 4(a), at the same electrode potential, the four NiCo_2O_4 nanowire array electrodes with identical composition exhibit different response current densities for the OER, with the NiCo_2O_4 -3 electrode delivering the largest current density. At current densities of 50 and $200\ \text{mA}\cdot\text{cm}^{-2}$, the total overpotential (η_{total}) on the NiCo_2O_4 -3 electrode is 262 and 294 mV, respectively (Fig. 4(b)), showing a descending order of NiCo_2O_4 -1 > NiCo_2O_4 -2 > NiCo_2O_4 -4 > NiCo_2O_4 -3. This variation trend conforms to a “V-shaped” curve corresponding to array depth changes, indicating a strong correlation between them. To further resolve the contribution of bubble behavior to OER efficiency, the η_{trans} primarily governed by bubble-related effects, were calculated, and the corresponding results are presented in Fig. 4(c). The detailed calculation procedures are described in the ESM, with the corresponding data presented in Fig. S8 in the ESM [37]. As illustrated, η_{trans} increases with rising current density for all electrodes, which can be attributed to the greater accumulation of bubbles on the electrode surface. At low current densities, the η_{trans} values of the four electrodes show little difference, approximately 5 mV at $10\ \text{mA}\cdot\text{cm}^{-2}$. With increasing current density, the gap in η_{trans} values among the four electrodes gradually widens. For example, at $300\ \text{mA}\cdot\text{cm}^{-2}$, η_{trans} for NiCo_2O_4 -1 (241 mV) is about 0.39 times higher than that of NiCo_2O_4 -3 (173 mV), clearly

demonstrating the negative effect of bubble behavior. It is evident that as the current density increases, η_{trans} induced by bubble effects follows the descending order NiCo_2O_4 -1 > NiCo_2O_4 -2 > NiCo_2O_4 -4 > NiCo_2O_4 -3, consistent with the trend of η_{total} . The lowest η_{trans} on the NiCo_2O_4 -3 electrode originates from its superior interfacial bubble dynamics, such as high nucleation probability, small bubble size, and rapid detachment, which effectively reduce bubble blockage of mass transport channels, enhance convective mass transport efficiency, and thus significantly lower η_{trans} . These results prove that regulation of the Cassie-impregnating characteristics of array electrodes can promote the interfacial mass transport process.

Regulation of the Cassie-impregnating interface also promotes the intrinsic activity of the electrode. As shown in Fig. 4(d), the active overpotential (η_{act}) stemmed from the reduced intrinsic activity of the electrode increases with current density, which arises from the enhanced restriction of reaction rate by active site density at high currents. Within the full current density range, NiCo_2O_4 -3 consistently exhibits the lowest OER η_{act} . Generally, electrode catalytic activity is mainly determined by the intrinsic activity and density of its active sites. In this study, the four electrodes were prepared via identical experimental procedures with the same elemental composition and crystalline structure, implying consistent activity of Ni and Co sites. Therefore, the difference in η_{act} for the NiCo_2O_4 -3 electrode should arise from variations in active site density, as verified by its electrochemical active surface area (C_{dl}). As shown in Fig. 4(e), the NiCo_2O_4 -3 electrode exhibits a C_{dl} value of $1.37\ \text{mF}\cdot\text{cm}^{-2}$ (calculated based on Fig. S9 in the ESM), which is higher than those of the other three electrodes. This difference in active site density should reflect the improved utilization of active sites accelerated by the mass transport process. The charge transfer resistance (R_{ct}) derived from representative electrochemical impedance spectroscopy (EIS) Nyquist plots further supports the aforementioned conclusion. As shown in Fig. 4(f), the NiCo_2O_4 -3 electrode has the smallest semicircle diameter and the lowest R_{ct} ($5.9\ \Omega$), indicating the fastest charge transfer. The R_{ct} of the four NiCo_2O_4 samples follows the order: NiCo_2O_4 -1 ($9.2\ \Omega$) > NiCo_2O_4 -2 ($7.0\ \Omega$) > NiCo_2O_4 -4 ($6.2\ \Omega$) > NiCo_2O_4 -3. The above deconvolution analysis of overpotentials indicates that structural regulation of NiCo_2O_4 arrays based on the Cassie-impregnating model not only optimizes intrinsic catalytic activity (reduced η_{act}) and interfacial electron transport (reduced ohmic resistance R_{oh}) but also improves mass transport efficiency (reduced η_{trans}) by modifying O_2 bubble behavior. The combined synergistic effect of these three factors ultimately ensures efficient performance in both OER and hydrogen evolution reaction (HER), as evidenced in Figs. S10 and S11 in the ESM.

3.5 Practical application expansion

To verify the practical application value of the Cassie-impregnating interface regulation strategy, the as-prepared series of NiCo_2O_4 electrodes were assembled into AEMWE devices (Fig. 5(a)), and their overall water splitting performance was systematically tested. As depicted in Fig. 5(b), the AEMWE polarization profiles reveal that the NiCo_2O_4 -3 electrolyzer consistently exhibits a low overall water splitting voltage during the continuous increase of cell current density. The overall water splitting voltages at 1 and $3\ \text{A}\cdot\text{cm}^{-2}$ are 1.80 and 1.95 V, respectively (inset of Fig. 5(b)), lower than those of other electrolyzers, directly reflecting the optimal overall water splitting energy efficiency of NiCo_2O_4 -3. During wide-range current density switching from 50 to $2000\ \text{mA}\cdot\text{cm}^{-2}$ (Fig. 5(b)), the voltage

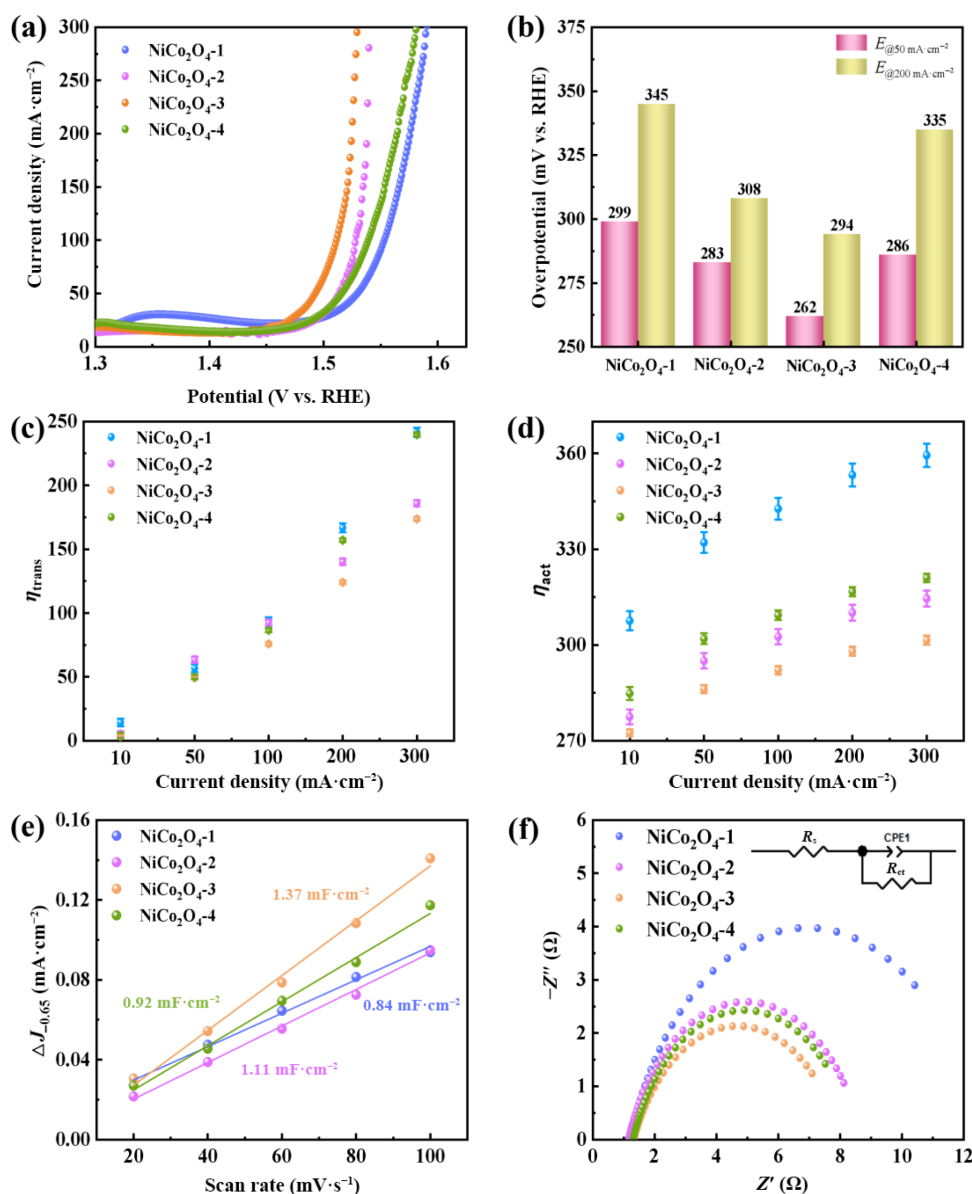


Figure 4 Electrochemical performance and overpotentials of electrodes. (a) 100% *iR*-corrected linear sweep voltammetry (LSV) curves for OER. (b) Overpotentials at 50 and 100 mA·cm⁻². (c) Variation trends of OER η_{trans} . (d) η_{act} with current density for NiCo₂O₄-1, NiCo₂O₄-2, NiCo₂O₄-3, and NiCo₂O₄-4 electrodes. (e) C_{dl} curves. (f) EIS curves for OER.

response of NiCo₂O₄-3 is rapid and stable, demonstrating excellent high-current tolerance. This feature precisely matches the core requirements of industrial electrolysis for high current density and fast load response [19], originating from structural regulation that optimizes bubble dynamic behavior and avoids impedance mutation caused by bubble accumulation at high currents, highlighting the operability of this strategy in practical scenarios. EIS plots at different current densities further support the above results.

Figure 5(c) displays step current galvanostatic tests for AEM water electrolyzers with current density stepped from 50 up to 2000 mA·cm⁻² and then reset to 50 mA·cm⁻². All devices show stable voltage plateaus with little drift. NiCo₂O₄-3 delivers the lowest cell voltage thanks to favorable bubble removal, while NiCo₂O₄-1 suffers higher voltage from bubble induced mass transport losses. Full voltage recovery after current step cycling verifies good reversibility, validating depth modulation for high current AEMWE

improvement. Figure 5(d) shows EIS spectra at 300 mA·cm⁻². The initial portion of the first semicircle is associated with the solution resistance (R_s) together with the ohmic resistance (R_b), which arises from a limited number of bubbles adhering to the electrode surface. The diameter of this semicircle reflects the R_{ct} at the electrode–electrolyte interface. In this AEMWE system, NiCo₂O₄-3 possesses the smallest R_{ct} of 11.56 Ω (Fig. 5(f)). (Note: the equivalent circuit diagram is placed in the corresponding figure in the main text). The second semicircle represents the effects of both large bubbles and newly generated smaller bubbles on the electrode surface. At elevated current densities, bubble accumulation markedly alters the contact between the electrode and electrolyte, thereby becoming a critical factor that limits the interfacial reaction kinetics. By comparison, the NiCo₂O₄-3 electrode is less affected by bubble effects than other electrodes ($R_b = 38.5$ Ω, Fig. 5(f)). Figure 5(e) displays EIS spectra at 10 mA·cm⁻², showing a single semicircle (only a small number of bubbles are generated at this

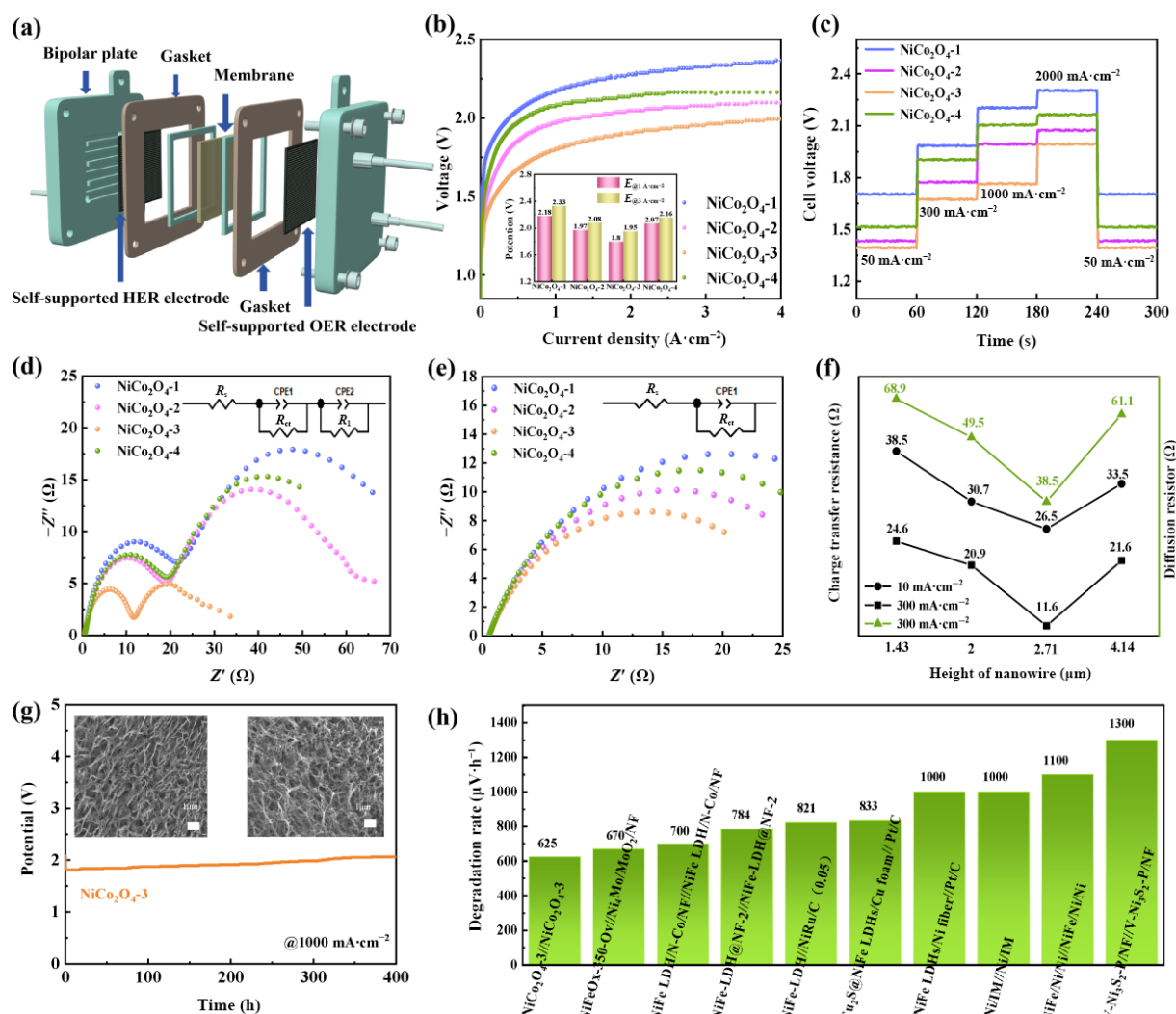


Figure 5 Overall water splitting performance and practical application verification. (a) Schematic diagram of the electrolyzer. (b) Polarization curves of AEMWE with different electrodes. (c) Voltage variation during current density switching. (d) EIS spectra at 300 mA·cm⁻². (e) EIS spectra at 10 mA·cm⁻². (f) R_{ct} and diffusion resistance values in EIS at different current densities corresponding to nanowire height variation. (g) Long-term stability of NiCo₂O₄-3||NiCo₂O₄-3 at 1000 mA·cm⁻². (h) Comparison of performance degradation rates for overall water splitting.

current density), whose diameter corresponds to the surface R_{ct} . In this AEMWE system, NiCo₂O₄-3 has the smallest R_{ct} of 26.5 Ω (Fig. 5(f)). By comparing resistance characteristics according to nanowire height (Fig. 5(f)), both the R_{ct} and diffusion resistance of NiCo₂O₄-3 reach the minimum values simultaneously. This implies that the Cassie-impregnating interface regulation strategy can also modulate interfacial charge distribution, improve the dispersion of bubble nucleation sites, and optimize the mass transport–reaction synergy efficiency.

Benefiting from the synergistic optimization of microstructure and interface properties, NiCo₂O₄-3 also exhibits outstanding long-term stability at high current densities. After continuous operation for 400 h at 1000 mA·cm⁻² (Fig. 5(g)), the potential shows no obvious decay (1.81–2.08 V), and SEM image in the inset reveals relatively intact electrode structure. The XRD patterns and XPS spectra of the material in Fig. S12 in the ESM show that its phase composition and surface chemical state are stable. This stability originates from rapid bubble detachment, which effectively reduces impact corrosion of the electrode surface by the electrolyte and ensures continuous exposure of active sites. In the comparison of

overall water splitting performance degradation rates (Fig. 5(h)), the degradation rate of NiCo₂O₄-3 is 625 $\mu\text{V}\cdot\text{h}^{-1}$, which is significantly lower than that of other materials (Table S1 in the ESM). Combined with its long-term stability test results, this proves its excellent durability and low performance attenuation risk in practical electrolysis devices, making it a highly competitive candidate catalyst for overall water splitting technology. It not only achieves comprehensive optimization of electrolysis performance but also provides an efficient and feasible technical route for the preparation of low-loss and long-life electrodes.

4 Conclusions

This work addresses the core challenges of bubble retention, mass transport obstruction, and high overpotential faced by AEMWE at high current densities and innovatively proposes a strategy to construct an ideal Cassie wetting state interface by precisely regulating the depth of NiCo₂O₄ nanowire arrays. This successfully realizes synergistic enhancement of O₂ bubble rapid detachment, interfacial mass transport efficiency, and electrocatalytic performance. NiCo₂O₄ nanoarrays with varying array depths from

1.43 to 4.14 μm were prepared by tuning the nanowire growth time. A deep learning approach was utilized to extract quantitative information on bubble generation from high-speed video recordings. Dynamic bubble statistics reveal that at 100 $\text{mA}\cdot\text{cm}^{-2}$, the optimized NiCo_2O_4 nanowire array interface achieves an ultrahigh O_2 bubble generation rate of $107\text{ cm}^2\cdot\text{s}^{-1}$ with a density maintained at 1000 cm^{-2} . This optimal bubble nucleation probability results from the balance between interfacial O_2 supersaturation and molecular mass transport efficiency. The minimum average size of detached bubbles is $\sim 120\text{ }\mu\text{m}$, originating from the variation of the upward force exerted on bubbles caused by height changes of the gas-liquid-solid three-phase contact line in the Cassie-Impregnating interface, which first increases and then decreases. At 300 $\text{mA}\cdot\text{cm}^{-2}$, η_{trans} for OER on the NiCo_2O_4 nanowire electrode is reduced to 157 mV, following the descending order $\text{NiCo}_2\text{O}_4\text{-1} > \text{NiCo}_2\text{O}_4\text{-2} > \text{NiCo}_2\text{O}_4\text{-4} > \text{NiCo}_2\text{O}_4\text{-3}$. An AEMWE single cell assembled with symmetric electrodes delivers a low overall water splitting voltage of 1.81 V at 1000 $\text{mA}\cdot\text{cm}^{-2}$ and operates stably for over 400 h. The strategy of constructing and optimizing a stable Cassie-impregnating wetting mode by tuning the array depth in nanowire arrays, accelerating bubble dynamics at the water electrolysis electrode interface, and promoting electrode mass transport performance provides a new research perspective for further improving electrode gas evolution efficiency.

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Data availability

All data needed to support the conclusions in the paper are presented in the manuscript and the Electronic Supplementary Material. Additional data related to this paper may be requested from the corresponding author upon request.

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Declaration of competing interest

All the contributing authors report no conflict of interests in this work.

Author contribution statement

Z. W.: Data collection, investigation, writing – original draft. H. X. W.: Data collection, investigation, writing – original draft. R. L. J.: Methodology, formal analysis. H. W.: Data curation, writing – original draft. J. W. R.: Formal analysis, editing. X. Y. W.: Formal analysis, methodology. J. L.: Data curation, editing, resources. All the authors have approved the final manuscript.

Use of AI statement

During the preparation of this work, the authors used the generative AI tool Doubao only for grammatical correction, English

language polishing, sentence optimization, and text repetition reduction. This AI tool was not used to generate any research design, experimental data, research analysis, discussion, or any original scientific content. All academic content, analysis, interpretations, and conclusions in this manuscript were independently completed, reviewed, and finalized by the authors. The authors take full responsibility for the final published content of this manuscript.

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