

Semi-metallic hydroxide supported single-atom catalysts for sustainable water oxidation

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Abstract: Developing efficient electrocatalysts towards water oxidation is of critical importance for numerous environmental and energy technologies. Single-atom catalysts have been widely recognized as a promising route, they are however often anchored on semiconducting substrates such as hydroxides, which inherently limits the electrical conductivity and overall catalytic efficiency. In this work, single-atom Ru catalysts are immobilized on a semi-metallic layered double hydroxide substrate (Ru-SM LDH) to achieve highly efficient water oxidation. The as-designed Ru-SM LDH catalyst displayed an overpotential of 270 mV to deliver an oxygen evolution reaction (OER) current density of 100 mA cm⁻², which was 60 mV lower than that of commercial RuO₂. *In-situ* electrochemical impedance spectroscopy revealed that Ru-SM LDH exhibited a reduced solution resistance and diminished charge transfer resistance during catalysis, leading to enhanced OER performance. An electrolyzer assembled with Ru-SM LDH anode and commercial Ni mesh cathode achieved a water electrolysis current density of 400 mA cm⁻² at 1.65 V and demonstrated exceptional stability for 1000 h, surpassing that of Ni||Ni electrolyzer (1.92 V, 350 h). These findings establish the design of single-atom catalysts supported on semi-metallic hydroxide substrates as a robust pathway toward efficient water electrolysis catalysts.

Key words: Single-atom catalyst; Semi-metallic hydroxide; electronic conductivity; oxygen evolution reaction.

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1. Introduction

The global environmental deterioration and energy crisis have focused increasing research interest on the oxygen evolution reaction (OER), which is a pivotal half-reaction in various environmental processes such as wastewater oxidation treatment, and energy technologies such as water electrolysis hydrogen production^[1-9]. Catalyst materials are required to lower the overpotential and enable the reaction to proceed in a more sustainable manner^[10-16]. To date, the state-of-the-art OER catalysts are noble metal-based oxides, namely ruthenium-or iridium-based oxides^[17]. However, these materials suffer from limited natural reserves and prohibitive costs, which undermine the sustainability of OER-based environmental and energy technologies^[18-22]. Consequently, reducing the consumption of noble metals in catalysts while enhancing their activity and stability has emerged as a central development priority in OER research.

Single-atom catalysts, which feature 100 % atomic utilization efficiency and precisely tunable coordination environments, have emerged as ideal candidates for next-generation

OER electrocatalysts^[23-26]. However, owing to their high dispersion, these atomic sites necessitate immobilization on robust supports to prevent agglomeration while fully exploiting the intrinsic advantages of single-atom catalysis^[27]. Thus far, the supports for single-atom catalysts have predominantly been carbonaceous materials, oxides, or hydroxides^[28-30]. Carbon-based supports, however, are typically hydrophobic—a property that is unfavorable for efficient water oxidation^[31-34]. Conversely, while oxides and hydroxides are hydrophilic, they are intrinsically semiconducting, their inherently limited electrical conductivity inevitably compromises the kinetics of electrocatalytic processes, thereby impairing OER efficiency^[35-39]. In this context, our group has previously developed a semi-metallic hydroxide that integrates the hydrophilicity characteristic of conventional hydroxides with the high electrical conductivity of semi-metals^[40], rendering it suitable as catalyst supports. Consequently, the immobilization of single-atom catalysts on such semi-metallic hydroxide supports, coupled with the optimization of their OER performance, constitutes a highly promising yet challenging design paradigm.

In this study, the dispersion of highly active single-atom Ru sites on a semi-metallic layered double hydroxide (LDH) support (hereafter denoted as “Ru-SM LDH”) was proposed to enhance the electronic conductivity of the material, thereby affording lower electronic resistance compared to conventional semiconductor-supported Ru single-atom catalysts (hereafter denoted as “Ru-SC LDH”) and enabling highly efficient OER catalysis. Aberration-corrected scanning transmission elec-

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tron microscopy (AC-STEM) confirmed the atomically dispersed state of Ru in the as-prepared Ru-SM LDH material. The as-achieved Ru-SM LDH catalyst exhibited a low OER overpotential and Tafel slope (270 mV at 100 mA cm⁻² and 26.06 mV dec⁻¹), outperforming conventional Ru-SC LDH (300 mV at 100 mA cm⁻² and 35.52 mV dec⁻¹) and commercial RuO₂ (330 mV at 100 mA cm⁻² and 93.42 mV dec⁻¹). *In-situ* electrochemical analysis demonstrated that the Ru-SM LDH possessed higher intrinsic OER catalytic activity, arising from its lowered solution resistance and reduced charge transfer resistance. An electrolyzer assembled with the Ru-SM LDH anode and a commercial Ni mesh (NM) cathode achieved an industrial-scale current density of 400 mA cm⁻² at a voltage of only 1.65 V under 80°C and 30 wt% KOH conditions, which was 0.27 V lower than that of NM||NM electrolyzer. This work suggested that semi-metallic hydroxides served as ideal supports for single-atom catalysts, and the single-atom/semi-metallic LDH paradigm offers a fresh design pathway for next-generation high-performance OER electrocatalysts.

2. Experimental

2.1. Synthesis of Ru-SC LDH and Ru-SM LDH

The Ru-SM LDH materials were prepared by a co-precipitation method followed by an aqueous reduction process. Typically, 30.9 mmol Ni(NO₃)₂·6H₂O, 9.6 mmol Fe(NO₃)₃·9H₂O, 1.1 mmol Co(NO₃)₂·6H₂O, 1.1 mmol Mn(NO₃)₂·6H₂O, and 0.1 mmol RuCl₃ to prepare solution A. 20.3 mmol NaOH and 20.3 mmol Na₂CO₃ were dissolved in 40 mL deionized water to form solution B. The solution A and B were jointly injected to 40 mL deionized water at 60°C with a rate of 40 mL h⁻¹. The mixed solution was then reacted at 10°C for 6 h to produce a brown precipitate (Ru-SC LDH), which afterwards underwent an aqueous reduction process, namely, 20 mL of 5 mg mL⁻¹ Ru-SC LDH solution was added to 40 mL 3 M NaBH₄ solution and stirred for 1 h. The Ru-SM LDH products were then obtained, washed with distilled water, and dried in a vacuum oven before further use.

2.2. Materials characterization

The morphologies and element distribution information of the materials were analyzed with a Hitachi SU8010 field emission scanning electron microscopy (SEM) equipped with an Oxford ULTRAMAX 40 X-ray energy dispersive spectrometer (EDS), a Tecnai G² F20 field emission transmission electron microscopy (TEM), and a FEI Titan G² aberration-corrected scanning transmission electron microscopy. The crystal structure was characterized by a Bruker AXSD8-FOCUS X-ray diffractometer (Cu-Kα radiation, λ=1.5418 Å). X-ray photoelectron spectroscopy (XPS) analysis was carried out on an AXIS-ULTRA DLD XPS scanning microprobe. The electrical property was characterized by a Quantum Design physical property measurement system. The Ru concentration in the electrolytes was analyzed on an Agilent 7900 inductively coupled plasma mass spectrometer (ICP-MS).

2.3. Electrochemical measurements

Electrochemical performance of the materials was investigated on a CHI 660E electrochemical workstation with a three-electrode system using graphite counter electrode, Hg/HgO reference electrode, and 1 M KOH electrolyte. The working electrode was made by coating 20 μL catalyst ink on Ni mesh sub-

strate, the catalyst ink was prepared by mixing 2 mg carbon black, 5 mg catalyst powder, 10 μL Nafion, and 1 mL ethanol. The linear sweep voltammetry (LSV) was carried out at a low scan rate of 1 mV s⁻¹. The electrochemical impedance spectroscopy (EIS) was performed from 10 mHz to 100 kHz with an amplitude of 5 mV at open-circuit potential or OER working potentials.

3. Results

The morphology of the as-prepared Ru-SM LDH material was shown in Figure 1a and S1, which revealed a typical two-dimensional nanosheet architecture with a thickness of ~28 nm. AC-STEM images in Figure 1b and S2 demonstrated that Ru components are atomically dispersed on the hydroxide surface, confirming the presence of isolated Ru single-atom sites. EDS results showed that the metal components in Ru-SM LDH were uniformly distributed throughout the particles, with an atomic ratio of Ru: Ni: Fe: Co: Mn of 3.0: 66.7: 20.9: 3.0: 6.5 (Figure S3-S4). X-ray diffraction (XRD) in Figure 1c exhibited the characteristic LDH structure for Ru-SM LDH, further indicating that Ru is uniformly distributed within the LDH matrix without aggregation or formation of impurity phases. XPS analysis in Figure S5 and S6 revealed that NaBH₄ reduction induced the formation of abundant oxygen vacancy defects in Ru-SM LDH material and a decrease in the valence state of Ru sites, which was beneficial for enhancing the electrical conductivity and optimizing the catalytic activity of the material. Temperature-dependent electrical conductivity measurements of Ru-SM LDH over the range of 2–400 K was presented in Figure 1d. In the region of 2–130 K, the resistivity of Ru-SM LDH gradually decreased with increasing temperature, whereas in the region of 130–400 K, it increased progressively. Since the resistivity of metallic materials increases with rising temperature, that of semiconductors decreases sharply, while semi-metals exhibit a weak temperature dependence with a possible reversal in the low-temperature regime^[41,42], this turning point in the resistivity at 130 K indicated the semi-metallic character of Ru-SM LDH. At the room temperature of 298 K, Ru-SM LDH material exhibited a remarkably low resistivity of 5.1 Ω cm, underscoring its high electrical conductivity. Noted that the improvement in electrical conductivity of Ru-SM LDH would facilitate electron transfer during the electrocatalytic processes, thereby enhancing the OER kinetics and the catalytic performance of the catalyst.

Ru-SC LDH was also synthesized and tested. Its morphology and crystal structure were analogous to those of Ru-SM LDH, featuring two-dimensional nanosheets with a thickness of ~30 nm (Figure S7-S8) and a typical LDH framework (Figure S9). The Ru, Ni, Fe, Co, and Mn elements were also uniformly distributed in Ru-SC LDH material, with an atomic ratio of 3.2: 62.8: 24.6: 3.9: 5.5 (Figure S10-S11), which was very close to that of Ru-SM LDH. However, its resistivity decreased monotonically with increasing temperature over the entire 2–400 K range (Figure S12), demonstrating a characteristic of semiconducting behavior. At the room temperature, the resistivity of Ru-SC LDH material was as high as 818.5 Ω cm, which in turn highlighted the distinct advantage of high electrical conductivity afforded by anchoring single-atom Ru on a semi-metallic hydroxide support.

The electrocatalytic OER performance of Ru-SM LDH mate-

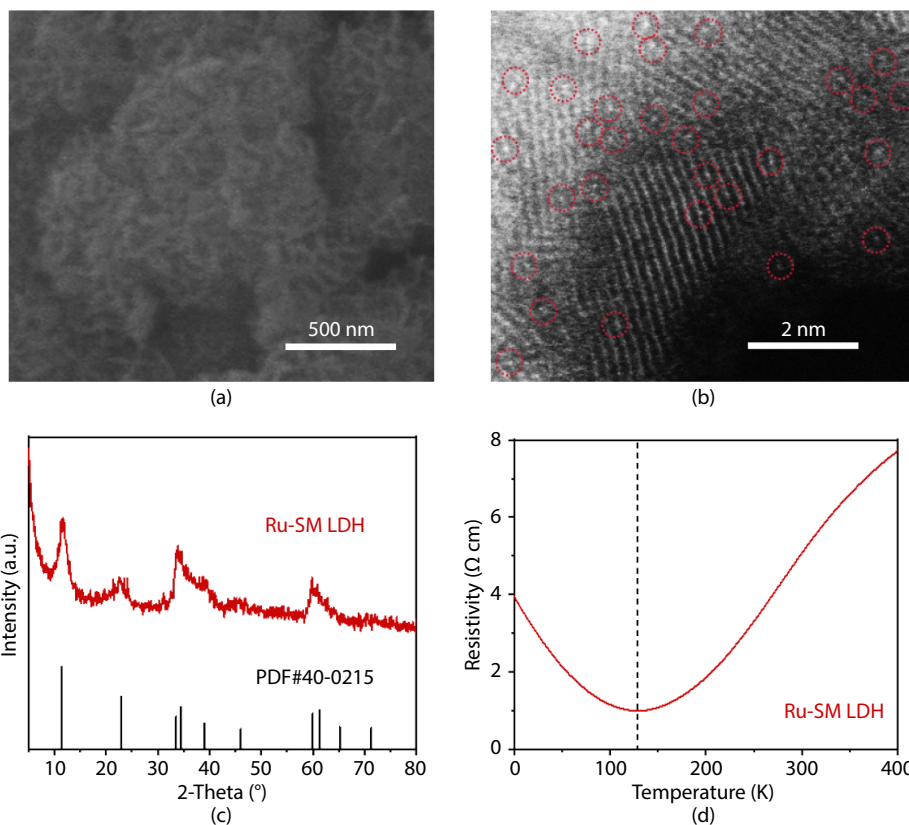


Fig. 1. (a) SEM image, (b) AC-STEM image, and (c) XRD analysis of as-prepared Ru-SM LDH catalyst, indicating that atomic Ru sites were anchored on LDH substrate. (d) temperature-dependent electrical resistivity of the Ru-SM LDH material, suggesting a semi-metallic nature of the material.

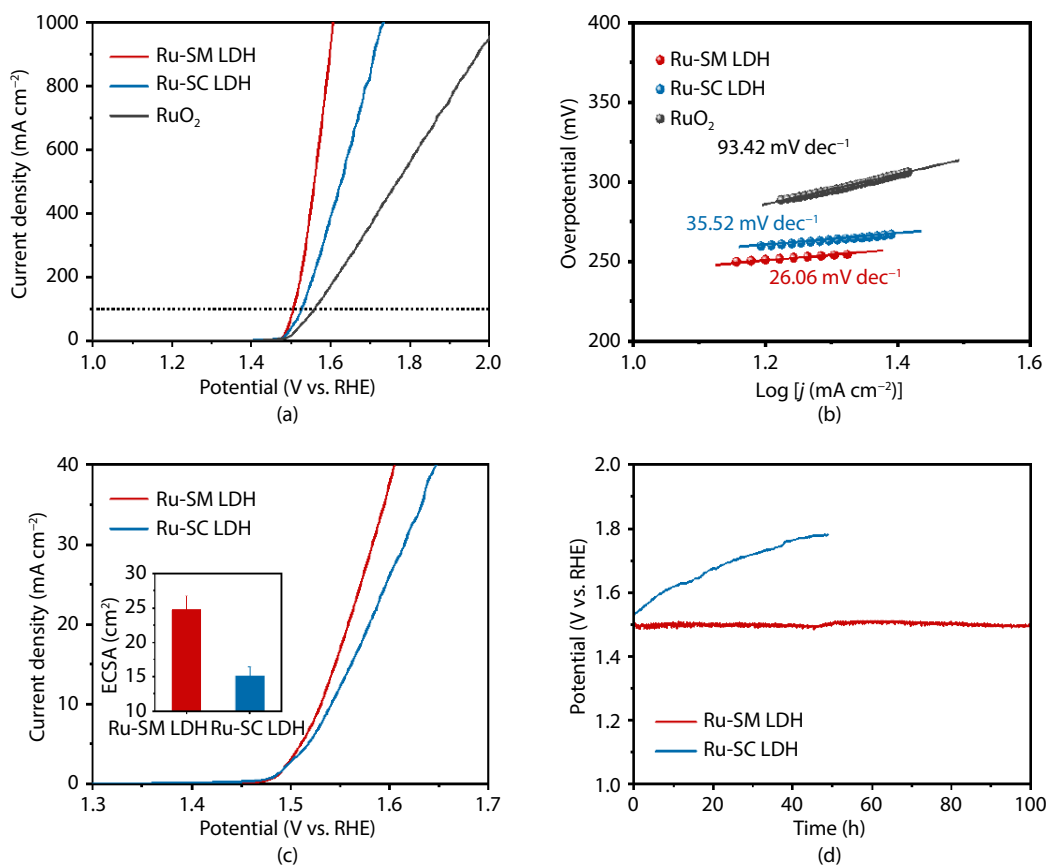


Fig. 2. (a) OER LSV curves and (b) Tafel slopes of the Ru-SM LDH, Ru-SC LDH, and commercial RuO₂ catalysts in 1 M KOH electrolyte. (c) ECSA-normalized OER LSV curves of the Ru-SM LDH and Ru-SC LDH catalysts in 1 M KOH electrolyte, suggesting the high intrinsic activity of Ru-SM LDH. The inset shows the ECSA of these two catalysts. (d) Chronopotentiometry curves of the Ru-SM LDH and Ru-SC LDH catalysts at 100 mA cm⁻² in 1 M KOH electrolyte, demonstrating the high electrochemical stability of the Ru-SM LDH catalyst.

rial was presented in Figure 2, all these electrochemical data were iR-corrected. Within the tested potential range (1.0–2.0 V) in a standard three-electrode configuration with 1 M KOH electrolyte, the Ru-SM LDH catalyst exhibited higher current densities than both Ru-SC LDH and commercial RuO₂ catalysts (Figure 2a), demonstrating its superior OER activity. Specifically, the Ru-SM LDH achieved an OER current density of 100 mA cm⁻² at 1.50 V, representing overpotentials 30 mV and 60 mV lower than those of Ru-SC LDH and RuO₂, respectively. Moreover, the Ru-SM LDH displayed the smallest Tafel slope (26.06 mV dec⁻¹, Figure 2b) among the three catalysts (35.52 mV dec⁻¹ for Ru-SC LDH and 93.42 mV dec⁻¹ for RuO₂), further corroborating the enhanced OER performance of Ru-SM LDH. To elucidate the origin of the improved activity of Ru-SM LDH, the electrochemically active surface areas (ECSA) of Ru-SM LDH and Ru-SC LDH were evaluated *via* cyclic voltammetry measurements in the non-Faradaic region (Figures S13–S14 and 2c). The ECSA of Ru-SM LDH was determined to be 24.75 cm², which is 165% that of Ru-SC LDH (~15.0 cm²). When the OER currents were normalized by ECSA, Ru-SM LDH still delivered higher current densities than Ru-SC LDH over the potential range of 1.3–1.7 V, indicating its improved intrinsic catalytic activity. These results suggested that the semi-metallic hydroxide support not only effectively dispersed Ru sites to enlarge the ECSA but also facilitated electron transfer, thereby boosting the intrinsic catalytic activity. Furthermore, the Ru-SM LDH exhibited robust electrocatalytic stability. As shown in Figure 2d, Ru-SM LDH sustained stable OER catalysis at 100 mA cm⁻² for 100 h with a negligible potential increase (~1.50 V). In stark

contrast, the operating potential of Ru-SC LDH rapidly rised from 1.53 V to 1.77 V within 40 h under identical conditions, underscoring the superior electrochemical stability of Ru-SM LDH.

To further investigate the semi-metallic hydroxide supported single-atom Ru catalyst during OER, *in-situ* EIS was employed, and the results were presented in Figures 3a–3b. Compared with the Ru-SC LDH, the Ru-SM LDH electrocatalyst exhibited lower solution resistance and charge transfer resistance, underscoring the critical role of high electronic conductivity in enhancing electrocatalytic performance. Quantitative analysis of the solution resistance (R_s) and charge transfer resistance (R_{CT}) during OER was conducted based on an equivalent circuit (Figure S15). As shown in Figure 3c, Ru-SC LDH displayed a R_s of 2.3 Ω under open-circuit potential (OCP). Upon applying OER overpotentials of 100–200 mV, its R_s gradually decreased to 2.0 Ω , likely due to the transformation of hydroxide into conductive oxyhydroxide active phase. However, at an OER overpotential of 300 mV, the R_s increased to 2.2 Ω , suggesting that the strongly oxidizing OER environment may oxidize the catalytically active species, leading to the diminished conductivity of catalysts. In contrast, the Ru-SM LDH exhibited a R_s of only 1.4 Ω under OCP, consistent with its superior electronic conductivity in Figure 1d. Over the OER overpotential range of 100–300 mV, its R_s decreased slightly and stabilized at approximately 1.3 Ω , indicating that it maintained excellent conductivity throughout the OER process. Regarding charge transfer resistance (Figure 3d), the Ru-SC LDH showed a R_{CT} of 9.1 Ω under OCP, which progressively declined to 1.4 Ω as the overpo-

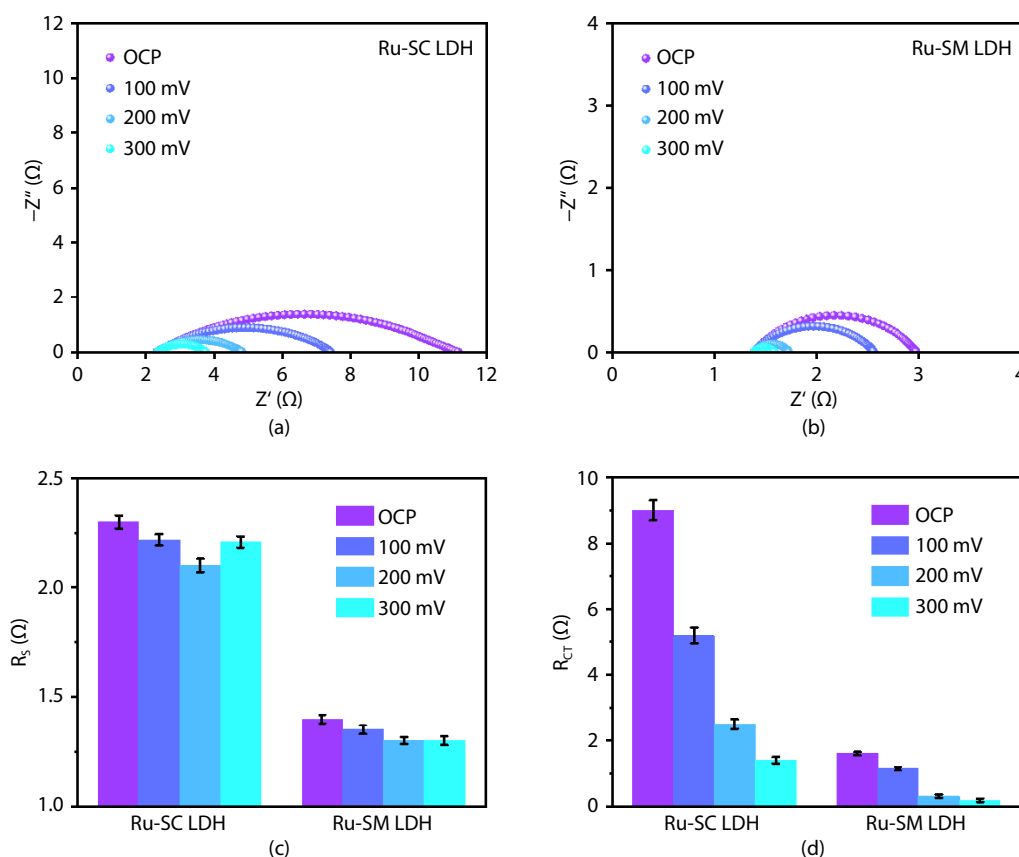


Fig. 3. *In-situ* EIS results of (a) Ru-SC LDH and (b) Ru-SM LDH catalysts under different OER overpotentials. (c) Solution resistance and (d) charge transfer resistance of the Ru-SC LDH and Ru-SM LDH catalysts under different OER overpotentials, indicating the improved electrical conductivity and accelerated OER kinetics of Ru-SM LDH.

tential increased from 100 to 300 mV, reflecting facilitated charge transfer kinetics during OER. By comparison, the Ru-SM LDH delivered an R_{CT} of only $1.6\ \Omega$ under OCP, and this value further diminished to as low as $0.2\ \Omega$ with increasing overpotentials, evidencing substantially lower charge transfer resistance enabled by its outstanding conductivity, which was a favorable attribute for electrocatalysis. Collectively, the electrochemical impedance analysis demonstrated that the enhanced conductivity of Ru-SM LDH effectively reduced both solution and charge transfer resistances during OER, thereby contributing to its superior electrocatalytic performance.

The exceptional electrochemical performance of Ru-SM LDH prompted us to assemble an alkaline electrolyzer to evaluate its practical applicability under industrial conditions (80°C and 30 wt% KOH). As shown in Figure 4a, when paired with a commercial NM cathode, the Ru-SM LDH anode delivered substantially lower voltages than the commercial NM anode at various current densities. Specifically, the Ni||Ru-SM LDH electrolyzer achieved voltages of 1.65 V and 1.92 V at an industrial current density of $400\ \text{mA cm}^{-2}$ and an ultrahigh current density of $1000\ \text{mA cm}^{-2}$, respectively, which were 0.27 V and 0.38 V lower than those of the Ni||Ni electrolyzer (Figure 4b). Besides, the Ni||Ru-SM LDH electrolyzer also showed 0.16 V and 0.24 V lower voltages than the Ni||Ru-SC LDH electrolyzer under the same test conditions (Figure S16), further suggesting the practical advantages of as-achieved Ru-SM LDH cata-

lyst. Moreover, the hydrogen production energy consumption of the Ni||Ru-SM LDH electrolyzer at $400\ \text{mA cm}^{-2}$ and $1000\ \text{mA cm}^{-2}$ was $3.94\ \text{kWh Nm}^{-3}$ and $4.59\ \text{kWh Nm}^{-3}$, respectively, which were $0.17\ \text{kWh Nm}^{-3}$ and $0.43\ \text{kWh Nm}^{-3}$ lower than those of Ni||Ni counterpart (Figure 4c). These results further substantiated the high efficiency of Ru-SM LDH catalyst under industrial water electrolysis conditions. Moreover, the Ni||Ru-SM LDH electrolyzer sustained stable hydrogen production at an industrial current density of $400\ \text{mA cm}^{-2}$ for 1000 h, with a voltage degradation rate of $0.06\ \text{mV h}^{-1}$ (Figure 4d). In stark contrast, the Ni||Ni electrolyzer operated for only 350 h under identical conditions, accompanied by a substantially higher voltage degradation rate of $2.20\ \text{mV h}^{-1}$, which was 36.7 times that of the Ni||Ru-SM LDH electrolyzer. Post-electrolysis SEM characterization of the Ru-SM LDH catalyst after 1000 h of continuous operation revealed that the material retained its two-dimensional nanosheet morphology, with a thickness of 29 nm, which was virtually unchanged from its pristine state. The Ru components in Ru-SM LDH remained atomically dispersed after 1000 h of electrolysis (Figure S17). Besides, no signal of Ru ($< 0.01\ \text{ppm}$) was detected in the electrolytes in the Ni||Ru-SM LDH electrolyzer either before or after 1000 h of electrolysis based on ICP-MS analysis (Figure S18). These evidences attested to the exceptional stability of Ru-SM LDH catalyst under industrial water electrolysis conditions.

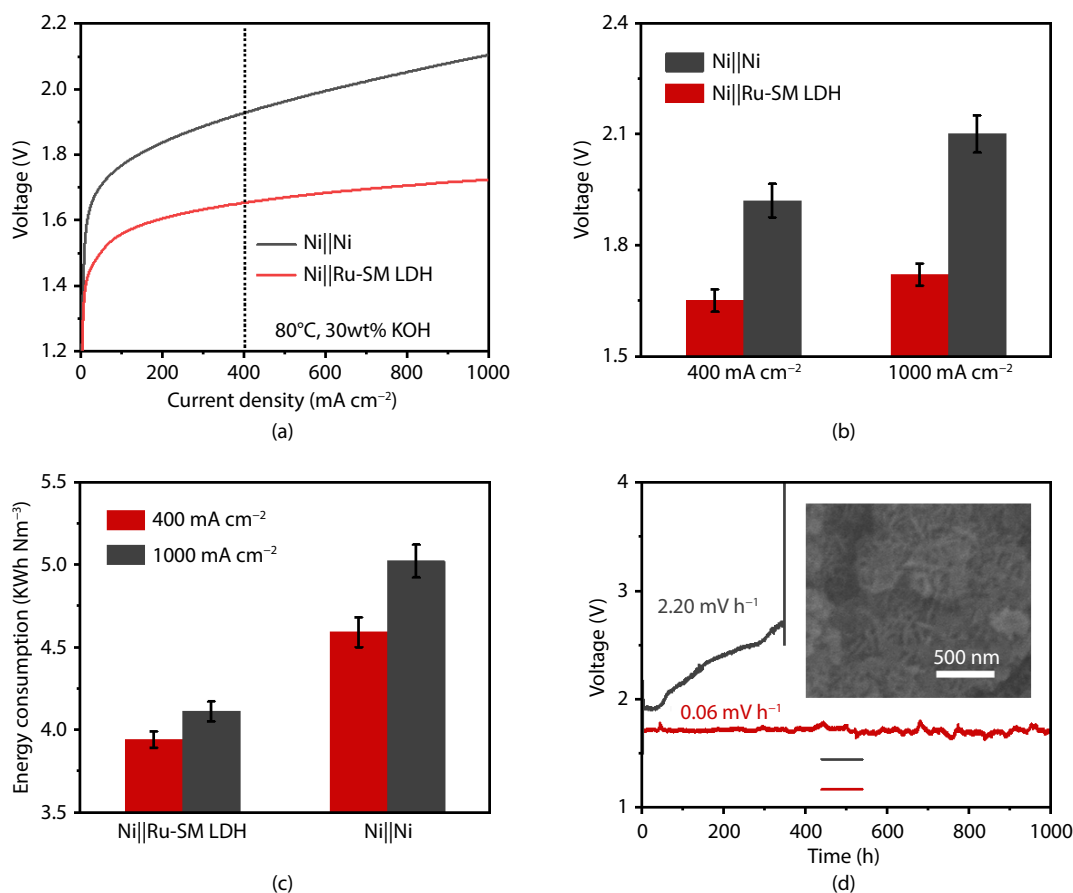


Fig. 4. (a) Polarization curves of the NM||NM and NM||Ru-SM LDH electrolyzers at 80°C , suggesting the enhanced electrocatalytic performance of Ru-SM LDH anode. (b) Voltages and (c) energy consumptions of NM||NM and NM||Ru-SM LDH electrolyzers to achieve 400 and $1000\ \text{mA cm}^{-2}$, the error bars were obtained by repeating the experiments for 5 times. (d) Chronopotentiometry curves of the NM||NM and NM||Ru-SM LDH electrolyzers at a constant current density of $400\ \text{mA cm}^{-2}$, the inset shows a SEM image of the Ru-SM LDH catalyst after 1000 h of electrolysis, demonstrating the high stability of Ru-SM LDH.

4. Conclusion

In summary, a semi-metallic hydroxide was employed as a support for atomically dispersed Ru sites to design efficient OER electrocatalysts. The as-prepared Ru-SM LDH catalyst not only featured atomic Ru distribution conducive to OER activity, but also exhibited typical semi-metallic characteristics and high electrical conductivity with a room-temperature resistivity as low as 5.1 Ω cm. Consequently, the Ru-SM LDH catalyst demonstrated lower solution resistance and charge transfer resistance during OER, alongside higher intrinsic catalytic activity, compared to the Ru-SC LDH counterpart. Moreover, an electrolyzer incorporating the Ru-SM LDH anode and a commercial NM cathode achieved a high current density of 400 mA cm⁻² at only 1.65 V under industrial alkaline water electrolysis conditions (80°C, 30 wt% KOH), and operated stably for over 1000 hours, underscoring its substantial application potential. This study not only validated the as-designed Ru-SM LDH as an exceptional and efficient OER electrocatalyst, but also opens new avenues for the future design of electrocatalysts targeting clean energy and environmental technologies.

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Conflicts of Interest

Zunhua Ke and Zhiwen Cao are currently employed by Daye Dahongshan Mining Co., Ltd.. The authors declare that there are no conflicts of interest in this work.

Data Availability

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

Electronic Supplementary Material (ESM)

The online version contains supplementary material available at <https://doi.org/10.26599/ECS.2026.9600029>.

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