An effective hybrid electrocatalyst for the alkaline HER: Highly dispersed Pt sites immobilized by a functionalized NiRu-hydroxide

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**ABSTRACT**

Developing a highly active and robust catalyst for the hydrogen evolution reaction (HER) using the minimal amount of Pt is a continuing challenge. Herein, we demonstrate a functionalized NiRu-hydroxide (NiRu-OH) catalyst that acts as a capable candidate to immobilize highly dispersed single Pt atoms. We synthesize this catalyst by means of Ru leaching from a NiRu-layer double hydroxide (NiRu-LDH) under oxygen evolution reaction (OER) conditions. The hybrid catalyst shows extremely high HER activity, with an overpotential of 38 mV to drive a typical current density of 10 mA cm\textsuperscript{-2} in alkaline media. We ascribe the excellent catalytic activity to the synergistic effect of the highly dispersed single Pt atoms and NiRu-OH in a dual-site alkaline HER mechanism. This work forms the basis for the exploration of matrices capable of trapping single atoms for various applications.

1. Introduction

In this study we describe a new catalyst for the hydrogen evolution reaction (HER) that is effective because it has single sites of platinum atom functionalization in a NiRu-hydroxide matrix. Electrochemical splitting of water in an alkaline electrolyzer plays a crucial role in developing energy systems, especially in the “hydrogen-based economy” \cite{1, 2, 3}. Highly efficient and inexpensive electrocatalysts for oxygen evolution reactions (OER), such as Ni-based hydroxide used in electrocatalysts, perform well in only alkaline or neutral media. In contrast, state-of-the-art electrocatalysts for HER, such as Pt, exhibit relatively sluggish kinetics in alkaline media, being at least two orders of magnitude slower than those in acidic media \cite{4, 5}. One widely accepted hypothesis for the slow kinetics is that under alkaline conditions, the reaction pathway for the HER consists of the dissociation of water, production of hydrogen ad-atoms (H\textsubscript{ad}) intermediates, and upon the desorption of hydroxide (OH\textsuperscript{-}) to refresh the catalyst surface, the recombination of H\textsubscript{ad} can generate H\textsubscript{2} \cite{6, 7}. Pt is generally considered to have an outstanding catalytic activity for H\textsubscript{2} desorption but a weak activity toward the dissociation of the H--OH bond. This creates a large kinetic energy barrier for the overall HER \cite{5}. To overcome this, hybrid catalysts have been created with a combination of metal oxides or hydroxides with Pt, promoting the dissociation of water and producing H\textsubscript{ad} that is subsequently adsorbed on the nearby Pt surfaces to form H\textsubscript{2} \cite{8, 9}. These catalysts exhibit a synergistic boosting in the overall HER kinetics. However, the scarcity and high cost of Pt represent a primary bottleneck, hindering the large-scale implementation of electrolyzers for the HER. Successful catalysts for HER will reduce the Pt loading while maintaining high catalytic activity.

The strategy of isolating Pt atoms has been pursued recently because it uses less platinum and also exhibits high catalytic activity. Nevertheless, avoiding aggregation or detachment of Pt atoms during operation is an unmet challenge. To create highly active and stable single metal atom catalysts, one promising strategy is to functionalize a conductive matrix to trap single atoms through strong interactions. For example, O-vacancy defects on oxides, C-vacancy defects on graphene, N-vacancy defects on TiN and C\textsubscript{3}N\textsubscript{4}, as well as cation vacancies, such as Mo-vacancy defects on MXene (Mo\textsubscript{2}TiC\textsubscript{2}Tx) all immobilize single Pt atoms successfully in recent years \cite{10, 11, 12, 13, 14, 15, 16}. However, it is difficult for transition metal hydroxides, which have sufficient catalytic capability toward the cleavage of the H--OH bond during a HER in alkaline media, to form enough functional groups by traditional hydrothermal methods \cite{13, 16}. Recently, we found that the NiRu-layer double hydroxide (NiRu-LDH) is very unique. The sizes of the Ru and Ni hydroxides are considerably different, resulting in NiRu-LDH being unstable and easily producing defects \cite{17, 18}. Moreover, RuO\textsubscript{4} and Ru(OH)\textsubscript{4} are prone to...
be oxidized to soluble RuO₄ during OER catalysis [19,20]. The active oxygen-coordinated Ru moieties with high valence states delaminate from the matrix causing degradation of the OER activity and the oxidative release of lattice oxygen during OER are both likely to create anchor sites for immobilization of single Pt atoms [21,22].

Herein, we designed a novel hybrid electrocatalyst consisting of single Pt atoms immobilized on a partially amorphous NiRu-hydroxide (NiRu-OH) with abundant functional groups (termed Pt/NiRu-OH). Both the formation of the functionalized NiRu-OH and the immobilization of the Pt atoms occurred in an alkaline media three-electrode system where Pt acted as a counter electrode as the OER and HER were run in series. The as-synthesized Pt/NiRu-OH exhibited high catalytic activity toward the HER under alkaline conditions with impressively low overpotentials (η) at 10 mA cm⁻² and a high amount of intrinsic catalytic activity. This study demonstrates that NiRu-LDH can be converted into partial amorphous NiRu-OH during the OER, which produces abundant functional groups for immobilizing single Pt atoms. This hybrid catalyst with low Pt loading offers a promising pathway toward highly efficient HER catalysis in alkaline media.

2. Results and discussion

We fabricate the NiRu-LDH supported on Ni foam via a hydrothermal method (see details in the Supporting Information), and we characterize its morphology and structure by scanning electron microscopy (SEM) and transmission electron microscopy (TEM). The SEM images in Fig. 1a and b indicate that the NiRu-LDH layer is uniformly grown on the Ni foam. The magnified SEM image and the TEM images reveal the stacked nanoparticle structure of the NiRu-LDH layers. This morphology is beneficial to the solid-liquid contact and in turn to the electrocatalytic reactions. The lattice spacing in the TEM image (Fig. 1c) is measured to be 0.594 nm and assigned to the (003) plane of NiRu-LDH. Importantly, no defects can be observed in the high-resolution images, and clear diffraction rings are exhibited in the selected-area electron diffraction (SAED) pattern from the low-resolution image (Fig. 1e), revealing good crystallinity in the NiRu-LDH.

We first investigate the HER performance by linear sweep voltammetry (LSV) in a 1 M KOH solution at a scan rate of 1 mV s⁻¹. Unfortunately, an overpotential of up to 166 mV is needed to drive the typical current density (j) of 10 mA cm⁻², similar to previously reported Ni-based hydroxide (Fig. 1f) [23–25]. Subsequently, we conduct the OER on the NiRu-LDH at 50 mA cm⁻² for 10 h to leach the Ru species and erode the crystal structure (Fig. S3). After the OER, we then investigate the HER performance of NiRu-OH. Note that after the OER, we carry out ~20 cyclic voltammetry (CV) cycles until the HER curve became stable presumably from the metal oxhydroxide sites that are generated during the OER that oblique the HER active sites. Specifically, the NiRu-OH shows a similar overpotential at 10 mA cm⁻² to that of the original NiRu-LDH, and the value increases to 191 mV. The slightly increased overpotentials are from the leaching of Ru, which is crucial for the active site in the HER [7,26].

We utilize inductively coupled plasma-mass spectrometry (ICP-MS) to measure the samples and the corresponding electrolytes before and after the OER. The amount of Ru detected confirms its leaching during the OER (Fig. 1g). Next, we perform CV on the NiRu-OH with a Pt wire counter electrode and a scan rate of 20 mV s⁻¹. The HER current density dramatically increases during the first 100 cycles and stabilizes after approximately 500 cycles, and we observe significant positive shifts in the onset potential (Fig. 1h). However, in contrast, we find a slow growth trend when a similar process was run for the original NiRu-LDH, NiFe-LDH and oxidized NiFe-LDH (after a 10 h OER). All these samples require at least 2000 cycles to stabilize (Fig. 1i, S4). Furthermore, we also conduct a long-term HER catalytic reaction at −0.235 V with a Pt wire counter electrode for both NiRu-OH and the original NiRu-LDH. It is surprising that the current density of NiRu-OH increases rapidly and tends to stabilize after approximately 40 h as the HER proceeds (Fig. S5). Whereas the NiRu-LDH displays continuously slow growth with time. In addition, we performed CV on the NiRu-OH with a graphite rod counter electrode for 500 cycles under similar conditions. As shown in Fig. S6, the HER activity of NiRu-OH after CV cycles changed slightly. Accordingly, we posit that during the initial period of the HER, the functionalized vacancies, especially Ru vacancies, and a partially amorphous surface on NiRu-OH could effectively immobilize single Pt atoms (from the Pt wire) and prevent Pt atom aggregation. These highly dispersed Pt atoms then combine with water dissociation sites from the hydroxide to effectively boost the overall alkaline HER. Because of this interesting phenomenon and the promising performance we systematically investigate the materials to confirm the hypothesis for its activity.

We use X-ray diffraction (XRD) to detect changes in the crystal texture. Fig. 2g shows the diffractograms of three samples. We index two major peaks to the Ni foam in all samples. The initial NiRu-LDH peaks (red trace) are consistent with the Bragg reflections of the transition metal LDH structure, especially the diagnostic reflection located at 2theta of 14.8°, which is indicative of interlayer galleries [17,18]. Other reflections are all in agreement with the pattern for a NiRu-LDH powder (Fig. S7). However, after the OER, the reflections from the interlayer galleries essentially disappear, and other new reflections indicate weak crystalline features, suggesting that the layered structure of the hydroxide is destroyed and that NiRu-LDH is now more amorphous. Thus, we refer to the NiRu-LDH after the OER as NiRu-OH (the −OH is confirmed by X-ray photoelectron spectroscopy (XPS) as discussed below). Moreover, the Pt/NiRu-OH shows a similar pattern to the NiRu-OH and lack Pt peaks. The absence of Pt reflections is due to the low content of Pt and its lack of crystallinity. The control sample, consisting of the original NiRu-LDH without the OER that acted as the working electrode for Pt deposition, is also characterized by XRD, and as expected the LDH structure persisted (Fig. S7).

The surface morphology of the NiRu-OH and Pt/NiRu-OH is shown in Fig. 2a–d. Both NiRu-OH layers with similar nanoparticle-stacked structures are uniformly deposited on the Ni foam, although cracks on surfaces and edges are created because of the extended electrochemical corrosion and Ru leaching. The TEM images of the NiRu-OH layer in Fig. 2e clearly illustrate the vacancies (circled in yellow), irregular crystal lattice fringes and amorphous phase, revealing abundant defects in the NiRu-OH. The low-magnification TEM-SEAD pattern further suggests a partially amorphous structure (Fig. 2f). Apart from the defects in the Pt/NiRu-OH, we observe high-brightness spots with large sizes in the high-magnification high-angle annular dark field scanning transmission electron microscopy (HAADF-STEM), confirming the immobilization of the highly dispersed single Pt atoms in both the crystalline and amorphous regions in the NiRu-OH (Fig. 2h and i, S8). The corresponding energy dispersive X-ray spectroscopy (EDS) elemental mapping analysis showed in Fig. 2j further reveals the highly dispersed Pt atoms on Pt/NiRu-OH.

The peaks of Ni, Ru, O and C are clearly presented in the survey spectrum (Fig. S9), illustrating the purity of the as-synthesized materials. Fig. 3a shows the Ni 2p spectrum of the three catalysts. Generally, the peaks apart from the satellite peaks (labeled Sat.) are the overlap of the doublets of Ni²⁺ and Ni³⁺ in Ni-based hydroxide [27,28]. We deconvolute the Ni 2p/sub/2 peaks into Ni²⁺ peaks at 855.10 ± 0.05 eV and Ni³⁺ peaks at 856.25 ± 0.05 eV by fitting the experimental data in all three catalysts [7,26]. We determine the ratio of Ni³⁺ to Ni²⁺ for NiRu-LDH to be approximately 1.1 by taking a ratio of the areas of the deconvoluted peaks. In stark contrast, the values are 4 times greater for both NiRu-OH and Pt/NiRu-OH, revealing that the oxidation of Ni³⁺ during the OER is not reversed during the subsequent HER. It is also likely that the Ni³⁺ did not result in positive HER activity based on our results (Fig. 1f) even though it has been reported that Ni³⁺ is highly efficient toward the cleavage of H₂O [29]. The Ru 3d peaks and C 1s peaks overlap directly in Fig. 3b; therefore, Ru 3d/sub/2 is mainly used as a guide for peak fitting (we use the constraints that the binding energy
As expected, we can index all of the peaks for Ru 3d5/2 at 281.80 ± 0.05 eV to the typical binding energy of Ru3+ [30,31]. We assign a small peak at 280.20 eV in Pt/NiRu-OH to Ru°, which was converted from high valence state Ru species during the HER (this is consistent with our previous observation [7]). The spectra also show C–C, C–O, C–O=O and C=O bonds presented from 284.60 eV to 289.60 eV from impurities from the ambient. The O 1s peaks of all catalysts show high peaks at a binding energy of 531.10 eV, which is indicative of the −OH, providing direct evidence of the hydroxide structure in all three catalysts (Fig. 3c) [7,32]. Moreover, we also compare the Pt 4f peaks of Pt/NiRu-OH with those of commercial Pt/C catalysts (Fig. 3d). The spectrum shown here is the result after background correction since the binding energy of Ni 3p is close to Pt 4f. We attribute two peaks of Pt 4f in Pt/C located at 71.00 and 74.30 eV to the signal of Pt°, reflecting the active center in Pt/C being metallic Pt [33,34]. In contrast, a weak and broad peak located between ~ 71.70 and ~ 74.40 eV is observed in Pt 4f7/2 of Pt/NiRu-OH. The valence states of Pt species in Pt/NiRu-OH fall in a range between +2 and +4; thus, the Pt on Pt/NiRu-OH will bond with O or OH. We also use extended X-ray absorption fine structure (EXAFS) technology to analyze the local coordination environment of Pt species in Pt/NiRu-OH. Fig. 3f describes the Fourier transforms of the Pt L3-edge EXAFS oscillations of the k^2-weighted χ(k) signals (Fig. 3e), in which there is one prominent peak at ~ 1.6 Å from the Pt–O contribution but no peak at ~ 2.2 Å from the Pt–Pt contribution, strongly indicating the sole presence of single Pt atoms in the Pt/NiRu-OH. X-ray absorption near edge structure (XANES) measurements in Fig. 3g show the intensity of Pt/NiRu-OH is similar to that of PtO2. The signals of both Pt/NiRu-OH and PtO2 are much higher than that of Pt foil, further confirming the electronic state of the Pt species in Pt/NiRu-OH are positively charged.

The HER electrocatalytic activities of the Ni foam, NiRu-LDH, NiRu-OH, Pt/NiRu-OH and Pt/C are assessed in 1 M KOH aqueous solution. Fig. 4a displays the 90 % iR corrected polarization of various samples. Among them, Pt/C undoubtedly exhibits the best HER activity of the aforementioned samples with an overpotential of 31 mV at a current
density of 10 mA cm$^{-2}$. The Pt/NiRu-OH shows a similar HER activity with only $\sim$ 38 mV to 10 mA cm$^{-2}$, which outperforms the other control samples of bare Ni foam (274 mV), NiRu-LDH (165 mV) and NiRu-OH (191 mV) at the same current density. Such a value is even less than those reported thus far for other alkaline compounds, such as Co$_{0.85}$Se/NiFe-LDH hybrid ($\eta_{10} = 260$ mV) [35], NiFe-OH-F ($\eta_{10} = 206$ mV) [23], amorphous SrCo$_{0.85}$Fe$_{0.1}$P$_{0.05}$O$_{3-\delta}$ nanofilm ($\eta_{10} = 206$ mV) [36], Li$^+$-Ni(OH)$_2$-Pt ($\eta_{10} = 100$ mV) [8], Ni$_{0.89}$Co$_{0.11}$Se$_2$ mesoporous nanosheet networks ($\eta_{10} = 85$ mV) [37] and edge-rich layered WS$_2$-Pt ($\eta_{10} = 50$ mV) [34] (Table S2, Supporting Information). The excellent HER activity of Pt/NiRu-OH is also confirmed by its smaller Tafel slope (39 mV decade$^{-1}$), which is closed to that of benchmark Pt/C (30 mV decade$^{-1}$) and lower than those of bare Ni foam (168 mV decade$^{-1}$), NiRu-LDH (107 mV decade$^{-1}$) and NiRu-OH (106 mV decade$^{-1}$), verifying the fast reaction kinetics on Pt/NiRu-OH (Fig. 4b).

The Nyquist plot of various samples is investigated to provide insight into HER pathways. As displayed in Fig. 4c, all five samples exhibit almost the same ohmic resistance ($R_0$) at high frequencies ($\sim 1.7 \pm 0.1 \Omega$), revealing that the supported catalysts did not influence the total resistances of the electrode and electrolyte. We observe two semicircles, which are much smaller, on both Pt/NiRu-OH and Pt/C, suggesting a decreased charge transfer resistance ($R_{ct}$) under that overpotential during the HER. We determined the electrocatalytic durability of Pt/NiRu-OH by measuring 5000 CV scans and a long-term test under the potential of -0.075 V without IR compensation. As shown in Fig. 4d, the overpotential of Pt/NiRu-OH at 10 and 100 mA cm$^{-2}$ increases to only 8 mV and 21 mV, respectively, after 5000 CV cycles. Moreover, the Pt/NiRu-OH also exhibits a robust operation over a period of 30 h with negligible activity loss (insert in Fig. 4d). After 30 h operation, the structural information of the Pt/NiR-OH is examined using SEM. As displayed in Fig. S11, the morphology of the Pt/NiRu-OH have no obvious variations. These data demonstrate the excellent stability during HER of our Pt/NiRu-OH catalyst, suggesting that the Pt atoms are firmly immobilized on NiRu-OH under alkaline and reduction potential conditions.

The outstanding intrinsic activity of Pt/NiRu-OH toward HER is further evidenced by specific (evaluated by normalizing LSV curves with electrochemically active surface area, ESCA) and mass activity. The ESCA is assessed by double-layer capacitance ($C_{dl}$), which is linearly proportional to ESCA (see the calculation of $C_{dl}$ in Supporting Information). As shown in Fig. S2, the Pt/NiRu-OH demonstrates a larger ESCA than NiRu-OH, which is due to the surface effect of the single Pt atoms. Fig. 4e illustrates the normalizing LSV curves with obtained $C_{dl}$, explicitly revealing the excellent intrinsic activity on the active site of Pt/NiRu-OH. Furthermore, it should be noted that the Pt...
loading of the Pt/NiRu-OH is determined to be $\sim 0.016$ mg (1.0wt%), which is $\sim 94$ times lower than that of Pt/C (measured by ICP-MS, Table S1). Impressively, after normalizing the current density with Pt mass loading and eliminating the contributions of the NiRu-OH and Ni foam (see the calculation details in Supporting Information), the mass activities under an overpotential of 50 mV are determined to be 1.01 and 0.07 A mg$^{-1}$ for Pt atoms on Pt/NiRu-OH and Pt/C, respectively. Moreover, as the applied overpotential reaches 77 mV, the mass activities are calculated to be 2.08 and 0.18 A mg$^{-1}$ for Pt/NiRu-OH and Pt/C, respectively (Fig. 4f). These dramatic values strongly indicate the advantages of Pt/NiRu-OH not only in improving HER activity of hydroxide in alkaline media but also in efficiently decreasing Pt usage.

For comparison, 1 %, 5 % and 10 % Pt particles were coated onto NiRu-OH (PtNP/NiRu-OH) to investigate the HER performance. The coating method are described in Supporting Information. The Pt particles are obtained from commercial Pt/C powder and the size of Pt particles falls in a range between 5–20 nm based on the TEM results (Fig. S12). As shown in Fig. S13, along with the increase of Pt loading, the HER activity enhances with the $\eta$ decreasing from 151 mV to 50 mV gradually. The 1% PtNP/NiRu-OH, which was loaded identical Pt mass with Pt/NiRu-OH, exhibits tenuous HER activity, further confirming the highly dispersed Pt atoms on NiRu-OH are more effective.

Based on the data above, we propose a possible mechanism for the electrochemical destruction of NiRu-LDH. Under the OER, the OH-terminated NiRu basal plane provides the active centers for oxygen evolution. On one hand, a high applied oxidizing voltage is conducive to frequent adsorption/desorption of oxygenated species on Ru and Ni atoms. Moreover, as the applied overpotential reaches 77 mV, the mass activities are calculated to be 2.08 and 0.18 A mg$^{-1}$ for Pt/NiRu-OH and Pt/C, respectively (Fig. 4f). These dramatic values strongly indicate the advantages of Pt/NiRu-OH not only in improving HER activity of hydroxide in alkaline media but also in efficiently decreasing Pt usage.

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3. Conclusion

In summary, our work demonstrates a novel strategy in which Ru leaching under OER conditions can convert crystalline NiRu-LDH into functionalized NiRu-OH. The resulting NiRu-OH acts as a capable candidate to immobilize highly dispersed single Pt atoms and enhance the catalytic activity. The hybrid catalyst, with NiRu-OH sites designed to accelerate water dissociation and single Pt atom sites to enhance hydrogen recombination, provides highly efficient HER catalysis in alkaline media. Moreover, Pt/NiRu-OH with low Pt loading exhibits much higher mass activity than commercial Pt/C and superior specific activity to other Ni-based hydroxides. The simple and scalable strategy described in this work opens an alternative path for the synthesis of other functionalized metal hydroxides and the development of highly active catalysts for application in water-splitting, fuel cells and metal-air batteries.

CRediT authorship contribution statement

Dan Li: Methodology, Formal analysis, Writing - original draft. Xufang Chen: Data curation, Methodology, Writing - review & editing. Yaozha Lv: Writing - review & editing. Guoyang Zhang: Methodology. Yu Huang: Methodology. Wei Liu: Methodology. Yang Li: Data curation, Writing - review & editing. Rongsheng Chen: Formal analysis, Writing - review & editing. Colin Nuckolls: Writing - review & editing. Hongwei Ni: Validation, Supervision, Writing - review & editing.

Declaration of Competing Interest

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

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Appendix A. Supplementary data

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References


