

构建用于高效析氢反应的核壳结构 CoNiMoO₄@Co₂P自支撑电催化剂

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摘 要

开发高效、稳定的非贵金属电催化剂对于碱性析氢反应(HER)至关重要, 但其动力学过程常受限于缓慢的水解离步骤。本文设计并成功合成了一种新型的核壳结构电催化剂(CoNiMoO₄@Co₂P), 该材料以结构稳定CoNiMoO₄纳米棒为核, 以具有优化氢吸附能力和亲水性的Co₂P纳米片为壳。这种独特的结构旨在通过核与壳的协同作用, 同时加速水的解离和氢气的脱附过程。实验结果表明, 所制备的CoNiMoO₄@Co₂P催化剂在1.0 M KOH电解液中表现出卓越的HER性能, 仅需9 mV的过电位即可达到10 mA·cm⁻²的电流密度。本研究为设计高性能碱性HER电催化剂提供了一种有效的核壳工程策略。

关键词

析氢反应, 电催化剂, 核壳结构, 协同效应, 碱性介质

Fabrication of a Freestanding CoNiMoO₄@Co₂P Core-Shell Electrocatalyst for Efficient Hydrogen Evolution

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Abstract

Developing efficient and stable non-precious metal electrocatalysts for the alkaline hydrogen evolution reaction (HER) is critical, yet the reaction kinetics are often hindered by the sluggish water dissociation step. To address this, we designed and synthesized a novel core-shell electrocatalyst,

CoNiMoO₄@Co₂P. This material features structurally robust CoNiMoO₄ nanorods as the core, coated with a shell of Co₂P nanosheets known for their optimized hydrogen adsorption energy and hydrophilicity. This unique architecture leverages synergistic core-shell interactions to simultaneously accelerate both water dissociation and hydrogen desorption. Electrochemical testing demonstrates that the CoNiMoO₄@Co₂P catalyst exhibits outstanding HER performance in 1.0 M KOH, requiring an overpotential of only 9 mV to achieve a current density of 10 mA·cm⁻². This work provides an effective core-shell engineering strategy for designing high-performance alkaline HER electrocatalysts.

Keywords

Hydrogen Evolution Reaction, Electrocatalyst, Core-Shell Structure, Synergistic Effect, Alkaline Medium

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1. 引言

当前全球能源转型呈现从高碳到低碳,从低碳向零碳的发展趋势。氢(H₂)作为一种能量密度高的零碳能源载体,被认为是一种很有前途的可持续能源[1]-[4]。电解水裂解制氢技术已受到广泛关注。这种绿氢技术是一种高效的制氢方法,其成本效益也更具优势[5]-[9]。贵金属基催化剂被认为是该技术的基准材料。然而,有限的储量和高昂的成本大大阻碍了它们的实际应用[10]-[14]。最近的研究集中在开发具有成本效益和效率的非贵金属替代品以及低贵金属负载量的催化剂[15]-[19]。然而,如何合理设计用于析氢反应(HER)的高效电催化剂仍是一项挑战。

过渡金属化合物的储量丰富成本低廉,是贵金属催化剂的替代品[20]-[22]。过渡金属氧化物具有较强的亲水性,有助于反应中快速吸附水[23]。过渡金属磷化物(TMP)中,金属中心和磷分别作为氢氧根和质子的受体,有利于快速的水解离,增强了 HER 的催化活性[24] [25]。但是单一金属化合物的活性有限,因此复合不同类型的材料可以持续完成水解离产氢气。异质结构工程是一种调节催化剂电子性质的有效途径,可以改善催化剂的活性[26]。肖等人,通过三步法制备了 FeNiP/MoO_x/NiMoO₄,其中 Fe₂P-Ni₅P₄和 Fe₂P/MoO_x 分别负责快速吸氢-放氢和吸水[27]。可见,磷化物的复合有效增强了碱性环境下 HER 的催化活性,证明构建具有不同功能的组件是一个可行的策略。掺杂异种元素也是一种改善电催化剂吸附能力的策略。根据密度泛函理论(DFT)计算表明, NiMoO₄ 具有优异的电子结构和稳定的结构框架,但是它的吸附强度 ΔG_{H*}不理想,为了调节它的吸附能力, Barik, S 等人加入 Co 元素,调节了 Ni 的电子性质进一步提升析氢的性能[28]。

核壳结构作为一种由异质材料构成的独特体系,能够有效地构建高效的电化学催化界面[29]。基于核与壳层之间的协同效应,这类结构展现出多重优势:如保护结构,增强离子和小分子对核的渗透,保护核不受外部影响,并提高催化性能[30]。因此,将核和壳设计为具有最佳催化活性和强吸水能力的不同组件是一种很有前途的策略。本文通过合成了一种新型的核壳结构电催化剂(CoNiMoO₄@Co₂P)加速水的解离和氢气的脱附过程,提升了反应效率。CoNiMoO₄@Co₂P 催化剂在 1.0 M KOH 电解液中表现出卓越的 HER 催化性能,仅需 9 mV 的过电位即可达到 10 mA·cm⁻² 的电流密度,且在持久性电化学测试中保持了良好的催化性能。

2. 实验部分

2.1. 在泡沫镍上合成 $\text{CoNiMoO}_4 \cdot x\text{H}_2\text{O}$ 纳米棒

首先将泡沫镍用盐酸处理，使表面清洁，然后用去离子水和乙醇超声使其完全漂洗。然后，将 $\text{NiSO}_4 \cdot 6\text{H}_2\text{O}$ 、 $\text{CoSO}_4 \cdot 7\text{H}_2\text{O}$ 和 $\text{Na}_2\text{MoO}_4 \cdot 2\text{H}_2\text{O}$ 倒入去离子水中搅拌，直到溶液变成透明液体。接下来，将溶液和一块泡沫镍放入不锈钢高压反应釜中，在 150°C 下加热 6 小时，使 $\text{CoNiMoO}_4 \cdot x\text{H}_2\text{O}$ 纳米棒在泡沫镍的表面均匀生长。漂洗后，并将其置于真空干燥炉中完全干燥。

2.2. 在 $\text{CoNiMoO}_4 \cdot x\text{H}_2\text{O}$ 纳米棒上电沉积 Co LDH

将 $\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ 倒入离子水中，搅拌得到紫色的透明溶液。在所得到的溶液中，以 $\text{CoNiMoO}_4 \cdot x\text{H}_2\text{O}$ 为工作电极，在 $\text{CoNiMoO}_4 \cdot x\text{H}_2\text{O}$ 纳米棒通过恒电位电沉积合成了 Co LDH。作为对比，通过上述合成方法，再以泡沫镍为工作电极，在泡沫镍电沉积合成了 Co LDH。

2.3. 以 $\text{CoNiMoO}_4 \cdot x\text{H}_2\text{O}@\text{Co LDH}$ 为前驱体，低温磷化合成了 $\text{CoNiMoO}_4@\text{Co}_2\text{P}$ 材料

在氮气流下，在管式炉的两侧分别放置面积 $\text{CoNiMoO}_4 \cdot x\text{H}_2\text{O}@\text{Co LDH}$ 前驱体和 NaH_2PO_2 粉末。然后将管式加热炉升温至 400°C ，保温 3 h，自然冷却后，制备了 $\text{CoNiMoO}_4@\text{Co}_2\text{P}$ 材料。作为对比，通过上述合成方法对制备的 Co LDH 进行了磷化，得到了典型的 Co_2P 材料。

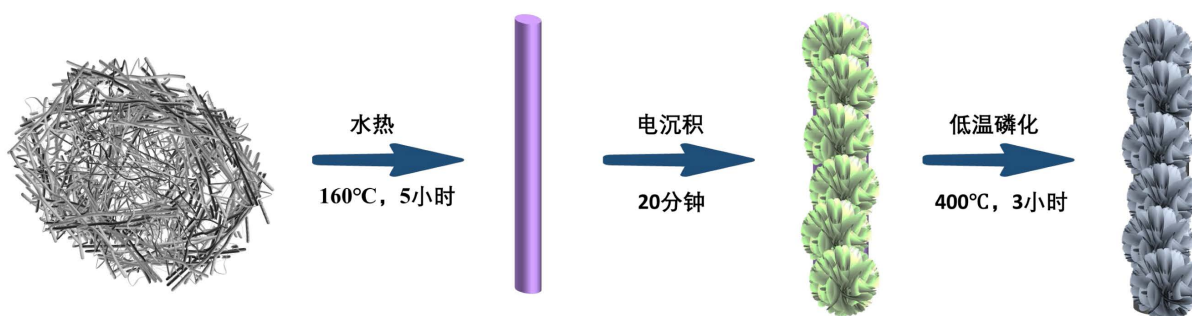


Figure 1. The synthesis process for the $\text{CoNiMoO}_4@\text{Co}_2\text{P}$ material

图 1. $\text{CoNiMoO}_4@\text{Co}_2\text{P}$ 材料的合成过程

3. 结果与讨论

图 1 展示了 $\text{CoNiMoO}_4@\text{Co}_2\text{P}$ 材料的合成过程。图 2 利用扫描电子显微镜观察样品表面形貌特征，图 2(a)和图 2(b)可以观察到， $\text{NiMoO}_4 \cdot x\text{H}_2\text{O}$ 是均匀致密地在泡沫镍衬底上的纳米棒。图 2(c)和图 2(d)显示了 $\text{CoNiMoO}_4 \cdot x\text{H}_2\text{O}$ 纳米棒的形貌，Co 掺杂后的纳米棒变细，可能是离子半径较小的 Co 离子部分取代 Ni 离子时，会引入晶格收缩和局部应变引起的。根据图 2(e)和图 2(f)显示，对比样 Co_2P 的形貌是均匀的纳米片阵列。图 2(g)和图 2(h)显示了合成的 $\text{CoNiMoO}_4@\text{Co}_2\text{P}$ 材料，可以发现 $\text{CoNiMoO}_4@\text{Co}_2\text{P}$ 的形貌为在 $\text{CoNiMoO}_4 \cdot x\text{H}_2\text{O}$ 纳米棒上生长了纳米片状的 Co_2P 材料， $\text{CoNiMoO}_4@\text{Co}_2\text{P}$ 材料结合了两种结构具有更大的比表面积，以自支撑的 $\text{CoNiMoO}_4 \cdot x\text{H}_2\text{O}$ 纳米棒的骨架结构，更有利于反应中电荷转移。

用 X 射线衍射仪对实验产物在泡沫镍上的相结构进行了分析。如图 3 所示，三条 X 射线衍射线的 44.6° 、 51.8° 和 76.4° 处的强衍射峰属于泡沫镍。 CoNiMoO_4 衍射峰的峰值为 28.7° 、 40.8° 、 48° 和 54.3° ，与 NiMoO_4 的标准卡(PDF#13-0128)衍射峰比，位置都向右偏移，这是因为 Co^{2+} 的离子半径(约 $0.745 \text{ \AA}$ ，高自旋)小于 Ni^{2+} 的离子半径(约 $0.830 \text{ \AA}$ ，高自旋)。最后，X 射线衍射图(XRD)图谱上的衍射峰会向更高的 2θ

角度方向发生移动(即“右移”)[31]。对比样 Co_2P 为主体非晶材料,但退火时导致非晶材料 Co_2P 在局部产生了小范围的晶体结构,新生晶粒尺寸极小,衍射峰会因尺寸宽化效应而变得强度微弱,易被强非晶背景信号掩盖,导致 XRD 图谱无明显射线衍射峰[32]。 $\text{CoNiMoO}_4@ \text{Co}_2\text{P}$ 的衍射峰与 CoNiMoO_4 的衍射峰基本一致,但由于 CoNiMoO_4 复合了 Co_2P 非晶材料,使 $\text{CoNiMoO}_4@ \text{Co}_2\text{P}$ 衍射峰强度与 CoNiMoO_4 相比降低了。

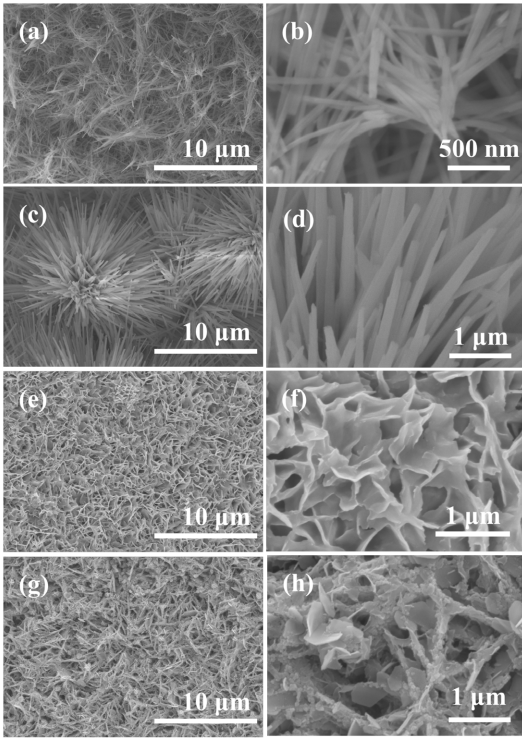


Figure 2. SEM images of (a), (b) $\text{NiMoO}_4 \cdot x\text{H}_2\text{O}$ nanorods; (c), (d) $\text{CoNiMoO}_4 \cdot x\text{H}_2\text{O}$ nanorods; (e), (f) Co_2P ; and (g), (h) $\text{CoNiMoO}_4@ \text{Co}_2\text{P}$

图 2. (a), (b) $\text{NiMoO}_4 \cdot x\text{H}_2\text{O}$ 纳米棒; (c), (d) $\text{CoNiMoO}_4 \cdot x\text{H}_2\text{O}$ 纳米棒; (e), (f) Co_2P 材料; (g), (h) $\text{CoNiMoO}_4@ \text{Co}_2\text{P}$ 材料

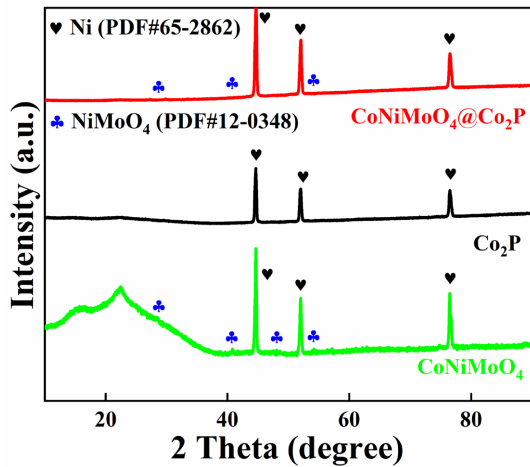


Figure 3. XRD patterns of the $\text{CoNiMoO}_4@ \text{Co}_2\text{P}$ material, Co_2P nanorods, and CoNiMoO_4 nanorods

图 3. $\text{CoNiMoO}_4@ \text{Co}_2\text{P}$ 材料, Co_2P 纳米棒和 CoNiMoO_4 纳米棒的 XRD 图

利用透射电子显微镜进一步表征了 $\text{CoNiMoO}_4@\text{Co}_2\text{P}$ 材料结构, 高分辨率电子显微镜图像如图 4(a)、图 4(b)所示, 生长在纳米棒上的取向各异的纳米片形成了均匀、交织的立体网络结构。图 4(c)、图 4(d)其中 0.29 nm 的晶格间距对应于 Co_2P 的(110)晶面。0.35 nm 的晶格间距对应于 NiMoO_4 的(110)晶面, 从这里也可以看出 Co 的掺杂并没有改变 NiMoO_4 的晶格结构。此外, 图 4(f)、图 4(j)显示, Ni、Co、Mo、P 和 O 元素分布在纳米棒上, Co、Mo 和 P 元素分布在纳米片中, 这些结果证明成功地制备了 $\text{CoNiMoO}_4@\text{Co}_2\text{P}$ 材料。

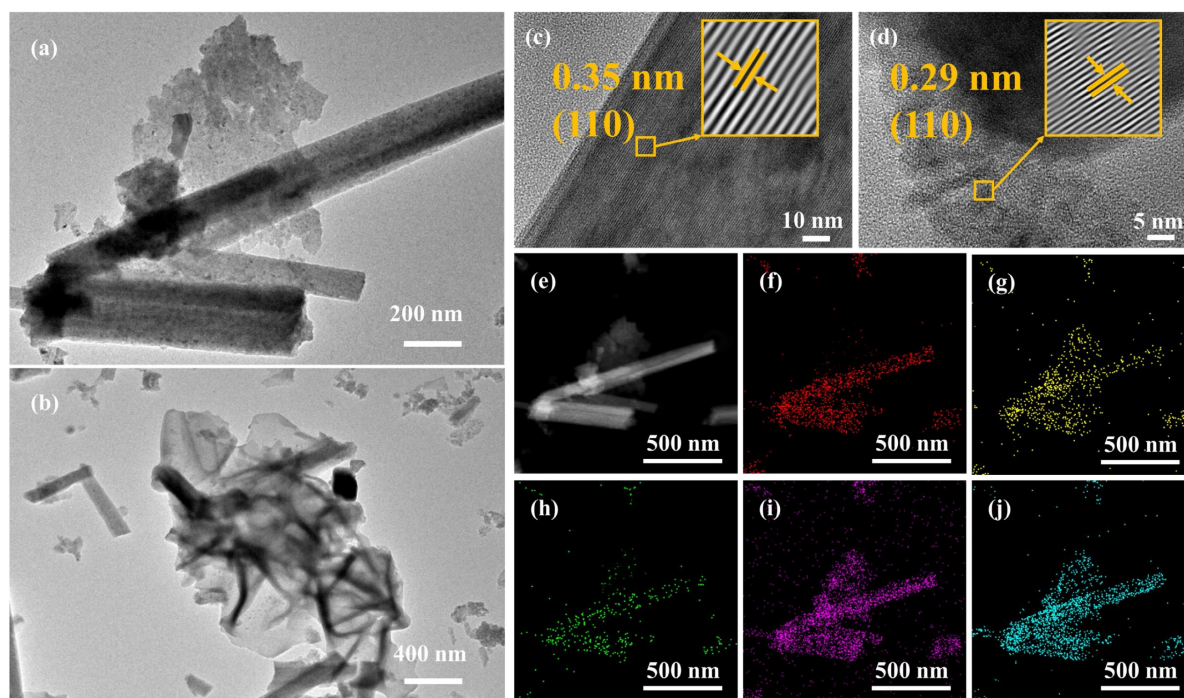
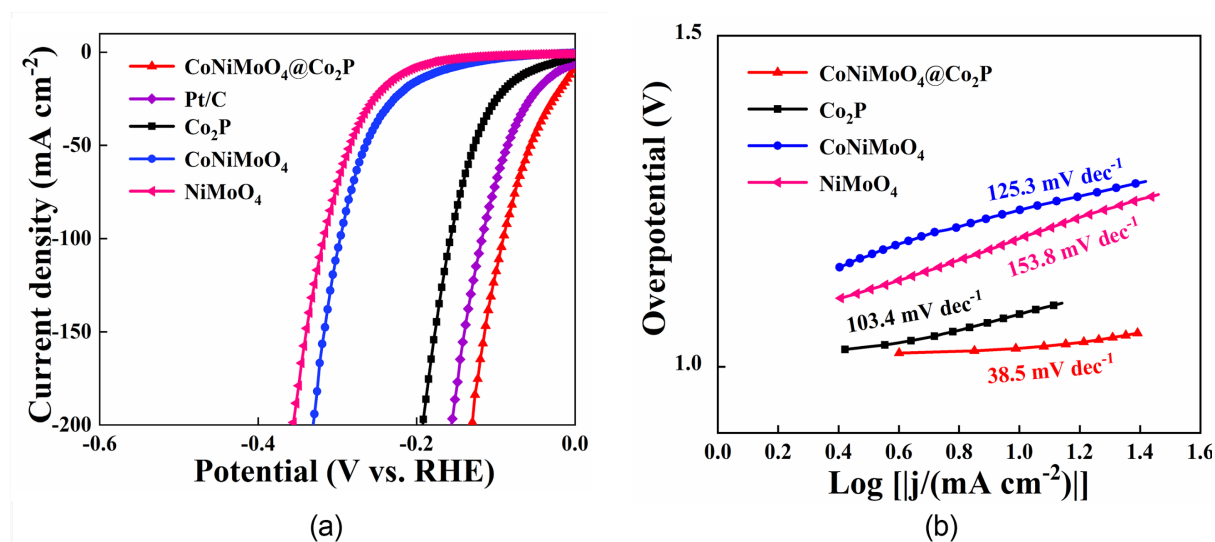


Figure 4. (a)~(d) TEM and HRTEM images of the $\text{CoNiMoO}_4@\text{Co}_2\text{P}$ material. (f)~(j) Corresponding elemental maps for Ni, Co, Mo, P, and O

图 4. $\text{CoNiMoO}_4@\text{Co}_2\text{P}$ 材料(a)~(d)的 TEM 和 HRTEM 图像。(f)~(j)为 $\text{CoNiMoO}_4@\text{Co}_2\text{P}$ 材料的 Ni、Co、Mo、P 和 O 的相应元素映射



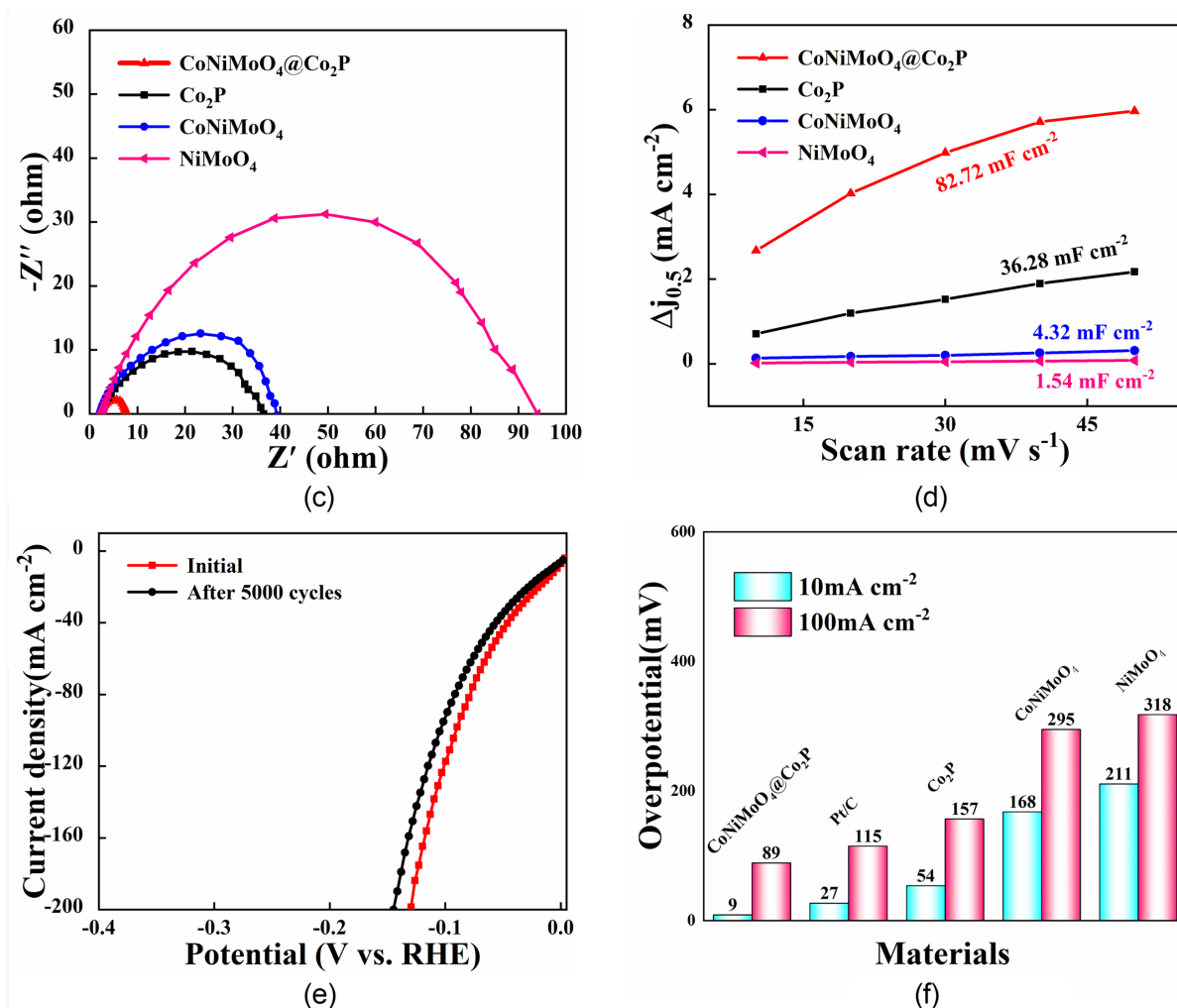


Figure 5. (a) HER polarization curves and (b) corresponding Tafel plots for CoNiMoO₄@Co₂P, Co₂P, CoNiMoO₄·xH₂O, and NiMoO₄·xH₂O in 1.0 M KOH. (c) Electrochemical Impedance Spectroscopy. (d) Electrochemical double-layer capacitance (C_{dl}) for the series of catalysts. (e) Chronopotentiometry curve for the CoNiMoO₄@Co₂P catalyst, demonstrating its long-term stability. (f) Bar chart comparing the overpotentials required to reach current densities of 10 and 100 mA·cm⁻² for all catalysts.

图 5. CoNiMoO₄@Co₂P 材料; Co₂P 材料; CoNiMoO₄·xH₂O 纳米棒和 NiMoO₄·xH₂O 纳米棒在 1.0 M KOH 中的 HER 极化曲线(a)。上述催化剂的相应塔菲尔图(b)。交流阻抗谱图(c)。上述催化剂的电化学双电层电容(d)。对 CoNiMoO₄@Co₂P 进行了稳定性测试(e)。上述催化剂的过电位电流密度分别为 10 mA·cm⁻² 和 100 mA·cm⁻²

在 1.0 M KOH 电解液中, 用典型的三电极系统测试了这些样品的 HER 性能。如图 5(a)显示了扫描速率为 5 mV·s⁻¹ 的 90% 补偿后的极化曲线。可以观察到, 当驱动 10 mA·cm⁻² 的电流密度时, CoNiMoO₄@Co₂P 材料只需要 9 mV 过电位, 比 Co₂P 材料(54 mV)、CoNiMoO₄·xH₂O 纳米棒(168 mV)和 NiMoO₄·xH₂O 纳米棒(211 mV)低得多, 因此 CoNiMoO₄@Co₂P 析氢反应活性高。进一步分析数据, 如图 5(f), 当电流密度为 100 mA·cm⁻² 时, CoNiMoO₄@Co₂P 材料的过电位(89 mV)优于 Co₂P 材料(157 mV)、CoNiMoO₄·xH₂O 纳米棒(295 mV)和 NiMoO₄·xH₂O 纳米棒(318 mV)。催化剂的塔菲尔斜率可以反映出析氢过程的反应动力学。如图 5(b)所示, CoNiMoO₄@Co₂P 的塔菲尔斜率为 38.5 mV·dec⁻¹, 优于 Co₂P (103.4 mV·dec⁻¹)、CoNiMoO₄·xH₂O (125.3 mV·dec⁻¹)和 NiMoO₄·xH₂O (153.8 mV·dec⁻¹)。结果表明, CoNiMoO₄@Co₂P 对 HER 具有相对快速的动力学反应过程。转移电荷电阻(R_{ct})也是决定 HER 活性的一个重要因素。如图 5(c)所示, 在这些催化剂中, CoNiMoO₄@Co₂P 材料的电化学阻抗谱显示 R_{ct} 是最小的, 这也代表着

CoNiMoO₄@Co₂P 材料在电极和电解液之间具有最好的电子传输速率,更有力地证明了 CoNiMoO₄@Co₂P 材料具有提高 HER 性能的作用。如图 5(d), CoNiMoO₄@Co₂P 材料的 C_{dl} 值为 82.72 mF·cm⁻², 明显优于 Co₂P 材料(36.28 mF·cm⁻²)、CoNiMoO₄·xH₂O 纳米棒(4.32 mF·cm⁻²)和 NiMoO₄·xH₂O 纳米棒(1.54 mF·cm⁻²)。这表明, CoNiMoO₄@Co₂P 材料具有较大的活化比表面积,在电化学过程中可以暴露出丰富的活性中心。随后我们对 CoNiMoO₄@Co₂P 材料的稳定性进行了表征。如图 5(e)所示, CoNiMoO₄@Co₂P 经历 5000 次循环后的 LSV 曲线几乎与初始的曲线一致,没有明显性能衰减。这表明 CoNiMoO₄@Co₂P 材料具有保持高性能的稳定性。

4. 结论

本研究成功通过水热、电沉积和磷化三步法,设计并制备了一种具有异质结构壳层的核壳电催化剂 CoNiMoO₄@Co₂P。应用异质结构巧妙地结合了 CoNiMoO₄ 核和 Co₂P 壳,结构增大了反应活性面积,有助于提升反应效率。得益于其独特的结构和组分协同效应,该催化剂在碱性介质中表现出低过电位、快速的反应动力学和出色的稳定性。这项作为设计高效、稳定的非贵金属析氢电催化剂提供了新的思路 and 有效的策略。

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