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Fe_3O_4/Ni复合材料用于增强析氧催化反应

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摘　要

开发先进的非贵金属析氧反应(OER)催化剂对实际电解水至关重要，而催化剂优化对于调控OER反应动力学、保证长期稳定性又起决定性作用。本研究采用水热法与热解法相结合的策略，成功制备了Fe_3O_4/Ni非贵金属复合催化剂，以提升OER性能。在1 M KOH电解液中测试显示：Fe_3O_4/Ni催化剂在10 mA·cm^{-2}电流密度下的过电位仅为242 mV，塔菲尔斜率低至61.04 mV·dec^{-1}，且具备优异的循环稳定性，综合性能显著优于单一的Ni与Fe_3O_4催化剂。本研究为高效非贵金属OER催化剂的设计提供了有效策略。

关键词

电催化水分解，析氧反应，Fe_3O_4/Ni催化剂

Fe_3O_4/Ni Composite for Enhanced Oxygen Evolution Reaction

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Abstract

Developing advanced non-noble metal catalysts for the oxygen evolution reaction (OER) is of great significance to practical water electrolysis, and material optimization plays a decisive role in regulating OER reaction kinetics and ensuring the long-term stability of catalysts. In this study, a strategy combining hydrothermal method and pyrolysis method was adopted to successfully synthesize the

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Fe_3O_4/Ni composite non-noble metal catalyst for enhancing OER performance. Tests in 1 M KOH electrolyte showed that the catalyst exhibited an overpotential of only 242 mV at a current density of 10 mA·cm^{-2}, a Tafel slope as low as 61.04 mV·dec^{-1}, and excellent cyclic stability. Its comprehensive performance was significantly superior to that of pure Ni and single Fe_3O_4 catalysts. This study provides an effective strategy for the design of high-efficiency non-noble metal OER catalysts.

Keywords

Electrocatalytic Water Splitting, Oxygen Evolution Reaction, Fe_3O_4/Ni Catalyst

1. 引言

电化学水分解是可持续制氢的重要过渡策略，可通过清洁水电解为替代化石能源基能源系统提供碳中和路径[1]。然而，其包含的两个半反应中，析氧反应(OER)因需经历四电子转移，动力学过程缓慢，成为制约整体能量转换效率的关键[2]。虽然贵金属氧化物(如 RuO_2、IrO_2)能为 OER 提供优异的催化活性，但高昂的生产成本与地缘政治导致的资源限制，严重制约着其广泛应用并阻碍商业化进程[3]。因此，开发经济高效的非贵金属 OER 催化剂以支撑清洁氢生产，已成为当前的迫切需求。

在新兴的非贵金属 OER 催化剂中，四氧化三铁(Fe_3O_4)与镍(Ni)材料因成本低廉、储量丰富，且催化活性与贵金属催化剂接近，成为极具潜力的替代选项[4] [5]。但单一组分存在明显局限，碱性电解环境中的活性位点暴露不足，以及长期运行中发生的不可逆腐蚀，都制约实际应用中的耐久性[6] [7]。因此，需要协同结合镍基材料的高催化活性与四氧化三铁材料的结构稳定性，开发 Fe_3O_4/Ni 复合催化剂，解决单一组分的固有缺陷。

本研究采用水热法与热解法相结合的策略，成功制备了 Fe_3O_4/Ni 复合材料并将其用作 OER 催化剂。实验结果表明，该催化剂表现出较低的过电位，较小的塔菲尔斜率和电荷转移电阻，以及优异的循环稳定性，综合性能显著优于单一的 Ni 与 Fe_3O_4 催化剂。其优异催化性能可归因于活性位点暴露量的有效提升，这一变化不仅直接增强催化活性，更进一步加速了 OER 反应动力学过程。

2. 实验部分

图 1 为 Fe_3O_4/Ni 催化剂的合成流程：首先将 0.01 mol $Ni(CH_3COO)_2{\cdot}4H_2O$、0.02 mol $Fe(NO_3)_2{\cdot}9H_2O$ 与 0.033 mol $CO(NH_2)_2$ 溶解于 75 mL 去离子水中，持续搅拌至体系混合均匀；随后将上述溶液转移至 100 mL 聚四氟乙烯内衬高压釜，于 180℃下水热 6 h，反应结束后缓慢冷却至室温；然后离心收集沉淀物，经去离子水与乙醇交替洗涤 3 次后，在 85℃真空干燥箱中干燥 12 h，得到前驱体粉末；最后将前驱体粉末置于管式炉，以 5℃/min 的升温速率升至 500℃煅烧 2 h，即得 Fe_3O_4/Ni 催化剂。

电化学性能采用 CHI 660E 电化学工作站在三电极体系下进行测试：工作电极为自制催化剂修饰电极，对电极为铂丝电极，参比电极为 Hg/HgO 电极。X'Pert PRO MPD 型 X 射线衍射仪(XRD)测试晶体结构；Axis Ultra DLD Kratos AXIS SUPRA 型 X 射线光电子能谱仪(XPS)表征元素组成及价态；Hitachi Regulus8100 场发射扫描电子显微镜(SEM)与 FEI Tecnai F20 透射电子显微镜(TEM)观察微观形貌与结构。

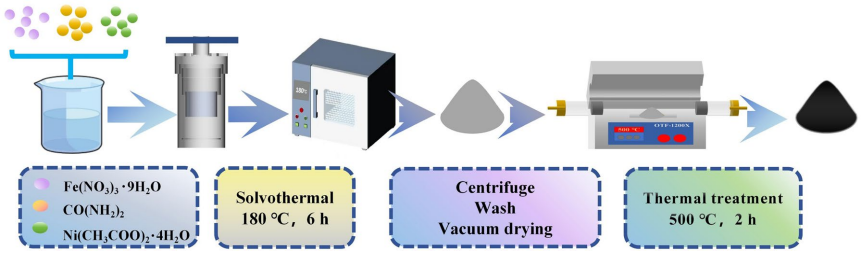

Figure 1. Preparation of Fe_3O_4/Ni catalyst
图 1. Fe_3O_4/Ni 催化剂的制备

3. 结果与讨论

通过 XRD 对 Fe_3O_4/Ni 催化剂的晶体结构和物相信息进行分析。由图 2(a)可知，在 43.7˚、52.1˚和 76.5˚处出现的衍射峰，分别对应于 Ni 的(111)、(200)和(220)晶面(PDF: 04-004-3634) [8]，而位于 18.1˚、30.3˚、35.7˚、37.1˚、42.8˚、48.1˚、53.2˚、57.1˚、62.7˚和 66.8˚处的衍射峰分别对应于 Fe_3O_4 的(111)、(220)、(311)、(222)、(400)、(331)、(422)、(511)、(440)和(531)晶面[9]。EDS 图谱(图 2(b))进一步证实了 O (51.73 wt%)、Fe (25.90 wt%)和 Ni (22.37 wt%)的共存和大致含量。

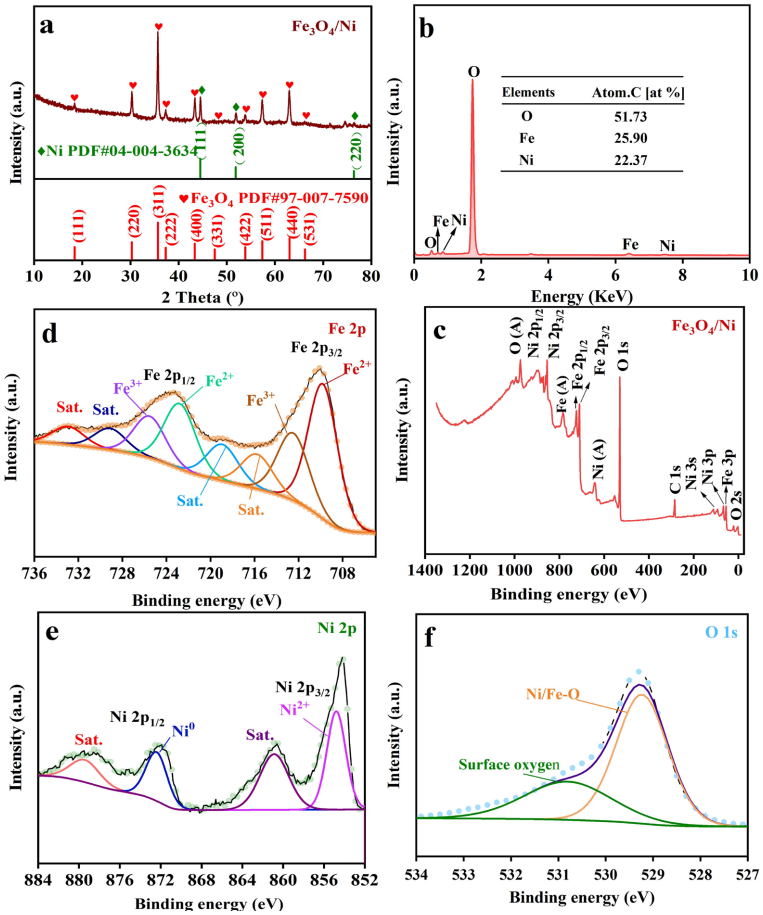

Figure 2. Spectra of Fe_3O_4/Ni catalyst: (a) XRD pattern, (b) EDS spectrum, (c) XPS survey spectrum, (d) Fe 2p spectrum, (e) Ni 2p spectrum, (f) O 1s spectrum
图 2. Fe_3O_4/Ni 催化剂的光谱图：(a) XRD 图谱，(b) EDS 光谱，(c) XPS 全谱，(d) Fe 2p 图谱，(e) Ni 2p 图谱，(f) O 1s 图谱

此外，XPS 全谱图(图 2(c))揭示了氧、镍和铁元素，其分别位于 978.7 eV (O)、786.4 eV (Fe)和 647.5 eV (Ni)。由图 2(d)可知 Fe 2p 谱图显示六个峰：四个卫星峰(734.1、729.2、720.3 和 717.1 eV)以及两个分别位于 724.3 (Fe $2p_{1/2}$)和 710.2 eV (Fe $2p_{3/2}$)的主峰[6]。对于 Ni 2p (图 2(e))，Ni $2p_{3/2}$ 和 Ni $2p_{1/2}$ 的特征峰分别在 886.5~877.4 eV 和 867.5~861.4 eV 处出现一个卫星峰，其中 Ni^0出现在 873.1 eV 处，Ni^{2+}则出现在 854.9 eV 处[10]。在 O 1s 光谱(图 2(f))中，可识别出表面吸附氧(531.5 eV)和晶格氧(Ni/Fe-O, 529.3 eV) [6]。

采用 SEM 与 TEM 分析 Fe_3O_4/Ni 催化剂的微观形貌。由图 3(a)和图 3(b)可见，该催化剂呈颗粒状结构，存在轻微的团聚，平均颗粒粒径约为 25 nm。高分辨透射电子显微镜(HR-TEM)图像(图 3(c))进一步显示，Fe_3O_4/Ni 催化剂存在 0.203 nm 与 0.241 nm 的晶格间距，分别对应 Ni (111)晶面与 Fe_3O_4 (222)晶面[11] [12]。此外，SEM 元素图像(图 3(d))表明 Fe、O、Ni 三种元素在催化剂表面均匀分布，进一步印证了 XRD 和 XPS 表征结果，证实所制备材料为 Fe_3O_4/Ni 复合材料。

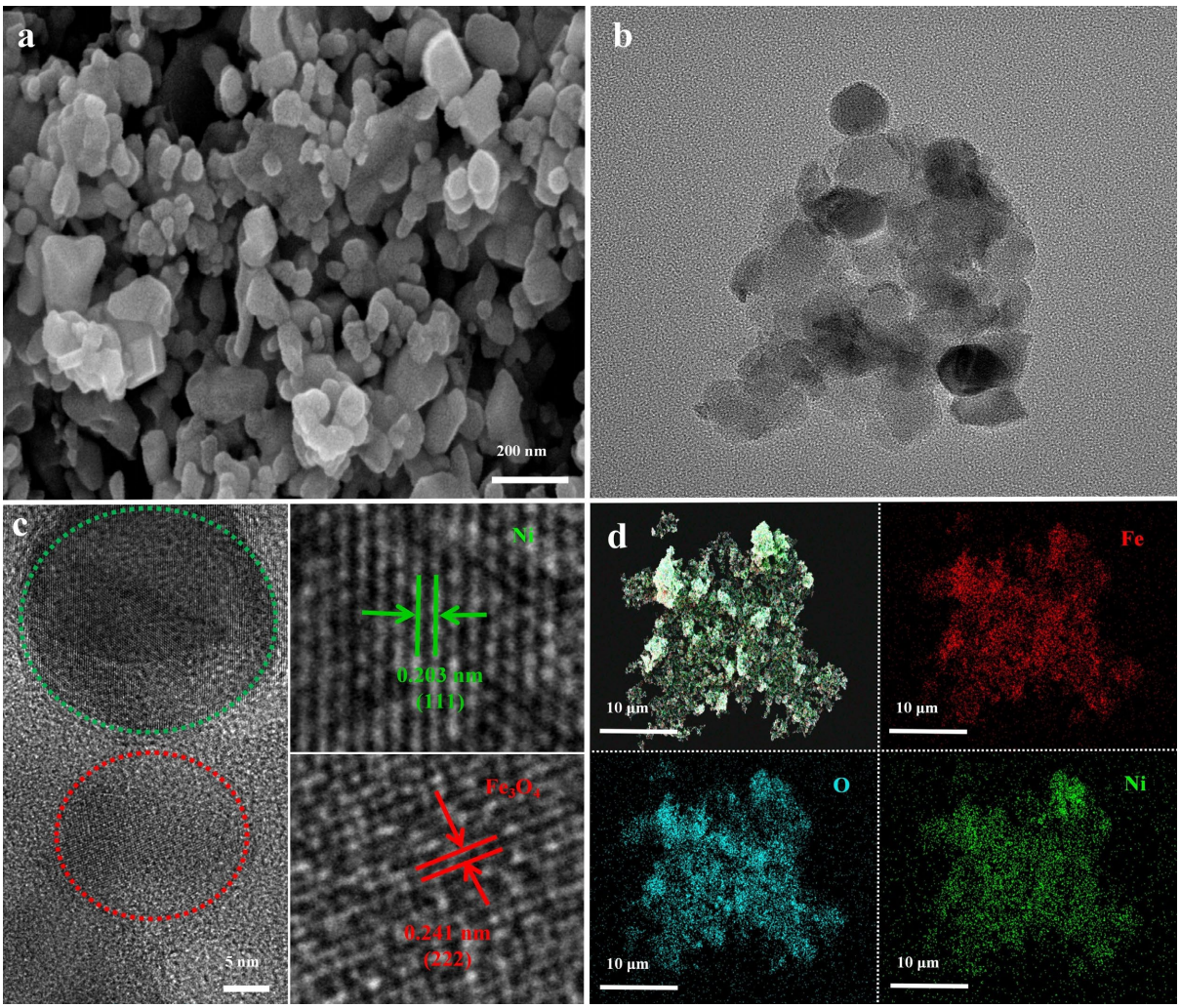

Figure 3. Electron microscopy images and elemental distribution maps of the Fe_3O_4/Ni catalyst: (a) SEM, (b) TEM, (c) HRTEM and (d) SEM-mapping of Fe_3O_4/Ni catalyst

图 3. Fe_3O_4/Ni 催化剂的电镜图和元素分布图：(a) SEM，(b) TEM，(c) HRTEM 和(d) 元素分布图

在 1 M KOH 电解液中评估 Fe_3O_4/Ni 的 OER 活性，并以 Fe_3O_4 和 Ni 为对照样，同步测试其线性扫描伏安(LSV)曲线。图 4(a)显示，Fe_3O_4/Ni 在 10 mA·cm^{-2} 电流密度下的过电位为 242 mV，显著低于 Fe_3O_4

(309 mV)和 Ni (339 mV)，表明其具有最优 OER 催化活性[13]。值得注意的是，当电流密度升至 50 和 100 mA·cm^{-2} 时(图 4(d))，Fe_3O_4/Ni 的性能优势进一步凸显。此外，对比近期报道的 NCS-P、COFeO@BP、CoP/CSNSs 等催化剂[10] [14]-[20]，发现 Fe_3O_4/Ni 的 OER 活性同样具有一定的优势(图 4(b)和表 1)。

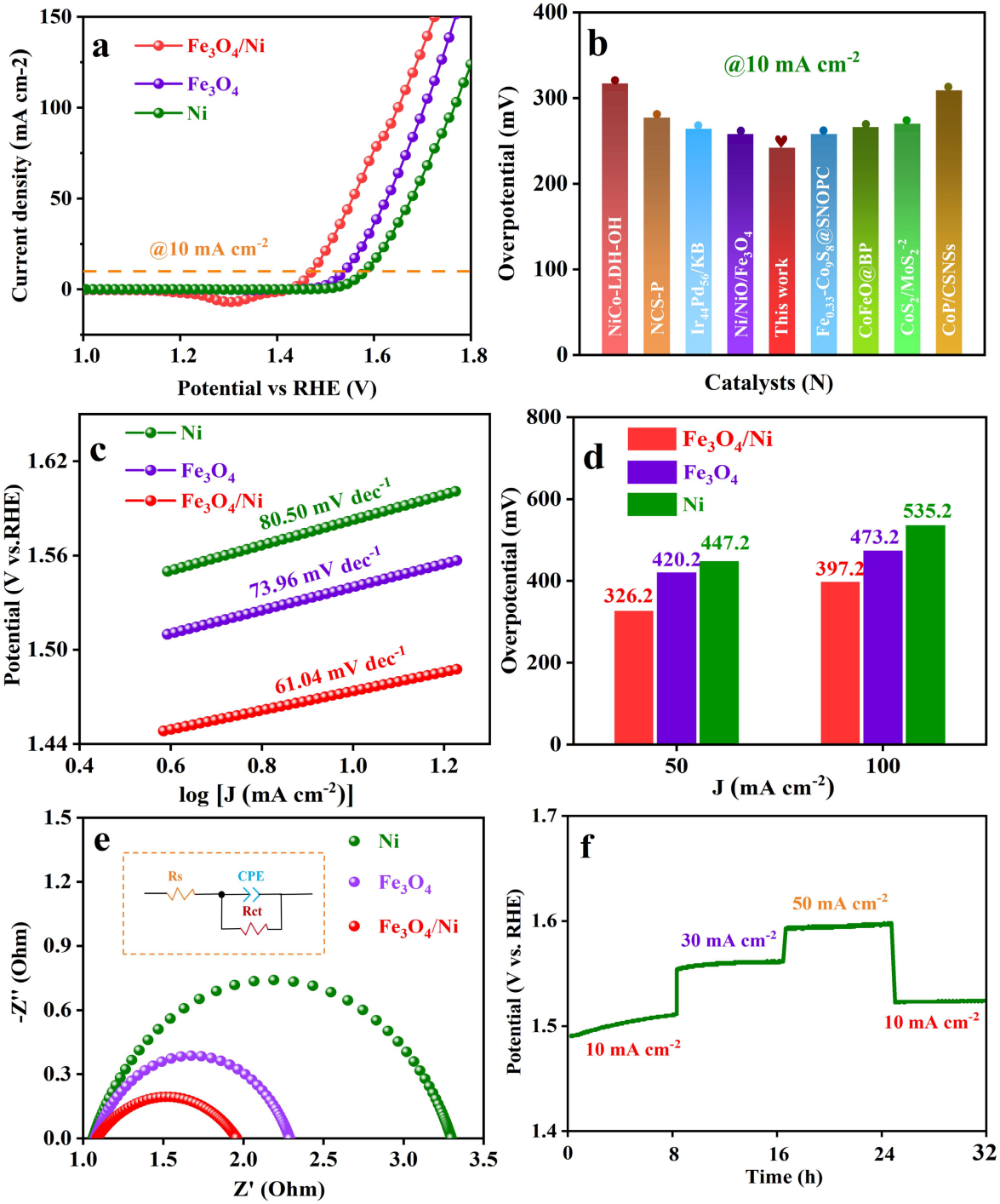

Figure 4. Electrochemical performance diagrams: (a) LSV curves, (b) comparison diagrams of Fe_3O_4/Ni with other catalysts, (c) Tafel Slopes, (d) overpotentials at 50 and 100 mA cm^{-2}, (e) EIS plots, (f) stability tests at different current densities

图 4. 电化学性能图：(a) LSV 曲线，(b) Fe_3O_4/Ni 与其他催化剂的对比图，(c) Tafel 斜率，(d) 50 和 100 mA·cm^{-2} 时对应的过电位，(e) EIS 图谱，(f) 不同电流密度下稳定性测试

Table 1. Comparison of the performance of Fe-Ni-based catalysts
表 1. Fe-Ni 基催化剂性能对比表

催化剂	电解液	η_{10} 过电位(mV)	塔菲尔(mV·dec^{-1})	参考文献
$NiFeO_xH_y$	1 M KOH	250	30	[23]
$Ni_{0.6}Fe_{0.4}$-pH	1 M KOH	263	55.6	[24]
$Ni_2Fe_1S_4$ NWs	1 M KOH	260	45	[25]
$NiFe_2O_4$-HNP/CNTs	1 M KOH	260	40	[26]
$Ni_{80}Fe_{20}$	1 M KOH	269	43	[27]
Ni-Fe/Nis-10	1 M KOH	265	38.8	[28]
P-S-NiFe NCs	1 M KOH	270	35	[29]
2Ni1Fe-MFS	1 M KOH	270	41	[30]
Ni-Fe LDH	1 M KOH	270	32	[31]
$Fe_2O_3/Fe_{0.64}Ni_{0.36}$@C-800	1 M KOH	274	83	[32]

塔菲尔斜率是反映OER反应动力学的关键指标。图4(c)显示，Fe_3O_4/Ni的塔菲尔斜率为61.04 mV·dec^{-1}，小于 Fe_3O_4 (73.96 mV·dec^{-1})和 Ni (80.50 mV·dec^{-1})，表明其反应动力学更快[21]。电化学阻抗谱(EIS)测试用于评估催化剂与电解质界面的电荷转移电阻，等效电路中 Rs、CPE 和 Rct 分别代表溶液电阻、恒相元件和电荷转移电阻。结果显示(图 4(e))，Fe_3O_4/Ni 的 Rct 值(0.88 Ω)小于 Fe_3O_4 (1.22 Ω)和 Ni (2.24 Ω)，证实其电荷转移速率更快[22]。稳定性测试表明(图 4(f))，Fe_3O_4/Ni 在 10、30 和 50 mA·cm^{-2} 电流密度下连续运行 32 h 后，电位保持相对稳定，证明其在碱性环境中具有较好的耐久性。

4. 总结

本研究采用水热和煅烧相结合的方式，将 Fe_3O_4 与 Ni 复合，成功制备出 Fe_3O_4/Ni 非贵金属 OER 催化剂。在碱性条件下，该催化剂展现出优异的电催化析氧性能，10 mA·cm^{-2} 电流密度下过电位仅 242 mV，塔菲尔斜率为 61.04 mV·dec^{-1}，且具备良好的催化稳定性。本研究为高效非贵金属 OER 催化剂的设计与开发提供了参考。

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