

Hierarchical carbon-encapsulated FeCo Alloy nanoparticles bridging N-doped graphene nanoribbons for efficient bifunctional oxygen electrocatalysis and rechargeable Zn-air batteries

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Abstract

Rechargeable Zn-air batteries are promising for next-generation energy storage, yet their practical application is hindered by the sluggish kinetics of the oxygen reduction reaction (ORR) and oxygen evolution reaction (OER). Herein, a noble-metal-free hierarchical FeCo@C/N-GNRs composite is fabricated via a facile three-step strategy, in which FeCo alloy nanoparticles are encapsulated within carbon layers that bridge N-doped graphene nanoribbons. This unique architecture integrates hierarchical pores (micropores, mesopores, and macropores) with abundant defects, maximizing accessible surface area and active-site exposure. The catalyst exhibits an ORR half-wave potential of 0.830 V, an OER potential of 1.530 V at 10 mA cm⁻², and a small ΔE of 0.70 V, outperforming benchmark Pt/C + IrO₂ and most reported bifunctional catalysts. Chronoamperometric tests confirm its superior ORR/OER durability. When employed as the cathode in liquid and flexible Zn-air batteries, it delivers high peak power densities (165.83 and 53.92 mW cm⁻²) and maintains stable output under bending, demonstrating great potential for wearable energy devices. The synergistic interaction between the encapsulated FeCo alloy and N-GNRs enhances electron transfer and structural stability.

1. INTRODUCTION

The global pursuit of sustainable energy technologies has driven extensive research into efficient and durable electrochemical energy conversion and storage systems^[1–5]. Rechargeable Zn-air batteries (ZABs) are promising candidates owing to their high theoretical energy density (1086 Wh kg⁻¹), low cost, and intrinsic safety^[6–9]. However, their practical implementation is severely constrained by the sluggish kinetics of the oxygen reduction reaction (ORR) and oxygen evolution reaction (OER), both of which involve multi-step electron transfer and high energy barriers^[7–9]. Although noble-metal-based materials (e.g., Pt/C and IrO₂) exhibit excellent activity toward both reactions, their scarcity, high cost, and poor durability impede large-scale application^[10, 11]. Therefore, developing efficient, durable, and noble-metal-free bifunctional oxygen electrocatalysts is highly desirable^[12, 13].

Among various non-precious candidates, carbon materials coupled with transition metals—particularly metal alloys—have attracted considerable attention. Bimetallic alloy catalysts often outperform their single-component counterparts because the inherent polarity differences between the constituent metals induce internal electron redistribution and lower the reaction energy barrier, thereby generating a synergistic effect^[14–16]. For example, Hao's group prepared FeCo alloy nanoparticles encapsulated in N,P co-doped carbon-coated N-doped carbon nanotubes as bifunctional oxygen electrocatalysts^[17]. Chen Zuofeng's group synthesized nanoflower-like Co/CoFe nanoparticles supported on N-doped graphitic carbon via a coordination–cation exchange–pyrolysis route^[18]. Despite their favorable activity, suppressing alloy dissolution and preventing catalyst deactivation in alkaline media remain critical challenges. The rational selection of carbon support is equally important. Graphene nanoribbons (GNRs), as a one-dimensional derivative of graphene, not only possess comparable electrical and mechanical properties but also offer abundant defects and edge sites that are beneficial for OER/ORR. Thus, GNRs

are considered promising carbon candidates for oxygen electrocatalysis. However, their edge sites often become initiation points for carbon corrosion during electrochemical reactions, which undermines long-term stability^[10, 19–21].

To address these issues, we first prepared N-doped graphene nanoribbons (N-GNRs). Nitrogen doping provides two synergistic benefits: the strong C–N bonds enhance carbon durability, while the introduced N atoms induce local electron spin and charge density redistribution, thereby improving catalytic activity. Furthermore, encapsulating metal nanoparticles within carbon shells is an effective strategy to prevent dissolution^[22–24]. Based on this rationale, we designed and synthesized FeCo nanoparticles coated with carbon layers on N-GNRs (FeCo@C/N-GNRs) via an in-situ growth–pyrolysis strategy. Physical characterization confirms this structural feature. Electrochemical tests demonstrate that FeCo@C/N-GNRs exhibits excellent bifunctional oxygen electrocatalytic activity, with a bifunctional parameter ΔE ($\Delta E = E_{10} - E_{1/2}$) of 0.70 V, outperforming benchmark Pt/C + IrO₂ and most reported bifunctional electrocatalysts. When assembled into liquid and flexible Zn-air batteries, it delivers high catalytic activity and good stability.

2. EXPERIMENTAL

2.1. Materials

Carbon nanotubes were purchased from Changzhou Angxing Graphene R&D Center, while Fe(NO₃)₃·9H₂O, Co(NO₃)₂·6H₂O were sourced from Aladdin Reagent (Shanghai) Co., Ltd.

2.2 Synthesis of graphene nanoribbons

First, the synthesis of graphene nanoribbons begins by measuring 36 mL of concentrated sulfuric acid into a cleaned 100 mL beaker, followed by adding 150 mg of multi-walled carbon nanotube powder. The mixture is stirred at room temperature for 30 minutes and then subjected to ultrasonication for 10 minutes. The beaker is then transferred to an ice bath equipped with magnetic stirring, where 750 mg of pre-weighed potassium permanganate powder is slowly added while stirring. After the KMnO₄ powder is completely added, the reaction continues for another 10 minutes. The temperature is then raised to 65°C and maintained for 1 hour. The resulting product is poured into a 500 mL ice-water mixture, followed by the gradual addition of 9 mL of hydrogen peroxide while stirring the mixture for 10 minutes. The product is washed with dilute hydrochloric acid and excess deionized water via vacuum filtration, and dried by freeze-drying to obtain powdered graphene nanoribbons (GNRs).

2.3 Synthesis of N-GNRs

Nitrogen-doped graphene nanoribbons (N-GNRs) were prepared through mechanical grinding followed by pyrolysis. Graphene nanoribbons and urea with a mass ratio of 1:20 were ground together in a agate mortar for over 30 minutes. The uniformly ground mixture was then transferred to a tube furnace, heated

to 800°C at a rate of 5°C/min under a high-purity argon atmosphere, and maintained for 1 hour. The collected sample was labeled as N-GNRs.

2.4 Synthesis of Fe@C, Co@C, FeCo@C and FeCo@C/N-GNRs.

Take 200 mg of iron(III) nitrate nonahydrate, 200 mg of cobalt(II) nitrate hexahydrate, and 20 mg of N-GNRs, and disperse them together in 10 mL of methanol. Stir vigorously with a magnetic stirrer for over 30 minutes to ensure uniform dispersion of the precursors, and label the resulting solution as Solution A. Dissolve 500 mg of p-phenylenediamine in 10 mL of methanol to prepare Solution B. Pour Solution B into Solution A and stir for 30 minutes to achieve thorough mixing. Transfer the mixture into a reaction vessel and react at 120°C for 8 hours. After the reaction, allow the reaction vessel to cool to room temperature, then collect the product via centrifugation and ethanol washing. Dry the product overnight in a vacuum oven to obtain black powder. The pyrolysis conditions are identical to those for N-GNRs, and the collected product is labeled as FeCo@C/N-GNRs. The preparation processes for FeCo@C (FeCo nanoparticles coated with carbon layers), Fe@C and Co@C are similar to that of FeCo@C/N-GNRs. Fe@C, Co@C, and FeCo@C were all synthesized without the introduction of N-GNRs. Fe@C and Co@C were prepared using only their respective metal salts, i.e., iron(III) nitrate nonahydrate for Fe@C and cobalt(II) nitrate hexahydrate for Co@C, while FeCo@C was obtained by adding both metal salts simultaneously.

2.5 Physical characterization

The crystal structures of all prepared samples were characterized by X-ray diffraction (XRD) using a D8 ADVANCE diffractometer with Cu K α radiation ($\lambda = 1.5418 \text{ \AA}$). Morphological observations and microstructural analyses, including transmission electron microscopy (TEM), high-resolution TEM (HRTEM), selected-area electron diffraction (SAED), high-angle annular dark-field scanning TEM (HAADF-STEM), and corresponding elemental mapping, were performed on a Tecnai G2 F20 S-TWIN instrument. Raman spectroscopic measurements were carried out on a Thermo Fisher micro-Raman system equipped with a 532 nm laser source operating at 5 mW. Nitrogen adsorption–desorption isotherms were recorded on an ASAP 2460 automatic surface area and porosity analyzer, from which the Brunauer–Emmett–Teller (BET) specific surface areas and pore size distributions (derived via the density functional theory model) were determined. The surface chemical compositions, atomic percentages, and bonding states of the elements were examined by X-ray photoelectron spectroscopy (XPS) on an ESCALAB 250Xi spectrometer.

2.6 Electrochemical characterization

All electrochemical measurements were conducted on a CHI 760E electrochemical workstation (Shanghai Chenhua Instrument Co., Ltd., China) equipped with a Pine rotating disk electrode system, using a standard three-electrode configuration. To fabricate the working electrode, 4 mg of catalyst powder was dispersed in 1 mL of a mixed solvent (ethanol:Nafion = 99:1 by volume) under ultrasonication until a homogeneous suspension was obtained. Subsequently, 10 μL of the well-

dispersed catalyst ink was pipetted onto a polished rotating disk electrode (RDE, 5 mm in diameter), resulting in a catalyst loading of 0.204 mg cm⁻². The Hg/HgO electrode (in 1 M KOH) served as the reference electrode, while a graphite rod was used as the counter electrode. All measured potentials relative to Hg/HgO were subsequently converted to the reversible hydrogen electrode (RHE) scale using the Nernstian equation:

$$E_{(V \text{ vs. RHE})} = E_{(V \text{ vs. Hg/HgO})} + 0.059 \times \text{pH} + 0.098$$

For the oxygen reduction reaction (ORR) measurements, all electrochemical tests were conducted in a 0.1 M KOH aqueous solution. Prior to linear sweep voltammetry (LSV) acquisition, the catalysts were electrochemically activated by cyclic voltammetry (CV) scanning. Subsequently, CV profiles were recorded under both N₂-saturated and O₂-saturated conditions. LSV polarization curves were then obtained in the O₂-saturated electrolyte at a sweep rate of 10 mV s⁻¹, with the rotating disk electrode (RDE) operated at varying rotation speeds of 625, 900, 1225, 1600, 2025, and 2500 rpm. The electron transfer number (n) involved in the ORR process was derived from the Koutecký–Levich (K–L) equation as follows:

CV scanning was conducted to activate the catalysts prior to measuring LSV curves firstly, then CV curves were recorded under N₂-saturated and O₂-saturated conditions. Linear sweep voltammetry (LSV) test was carried out with a scanning rate of 10 mV/s at different RDE speeds (625, 900, 1225, 1600, 2025 and 2500 rpm) in O₂-saturated electrolyte. The number of transferred electrons (n) for ORR can be calculated according to the Koutecky–Levich (K–L) equation:

$$\frac{1}{J} = \frac{1}{J_k} + \frac{1}{J_l} = \frac{1}{J_k} + \frac{1}{B\omega^{1/2}}$$

$$B = 0.62nFC_0D_0^{2/3}\mu^{-1/6}$$

Where J , J_k and J_l are the measured current densities (mA/cm²), the kinetics current density (mA/cm²) and the diffusion-limiting current densities (mA/cm²), ω is the rotating speed (rad/s), F is the Faraday constant ($F = 96485 \text{ C/mol}$), C_0 and D_0 are the concentration of O₂ ($1.2 \times 10^{-6} \text{ mol/cm}^3$) and the diffusion coefficient of O₂ in 0.1 M KOH ($1.9 \times 10^{-5} \text{ cm}^2/\text{s}$). μ is the kinematic viscosity of the electrolyte 0.1 M KOH ($0.01 \text{ cm}^2/\text{s}$).

The Oxygen evolution reaction (OER) testing process is similar to ORR. Before measuring the LSV curve, a CV activation process is first performed to activate the catalyst, and then the OER polarization curve of the catalyst is recorded at a scan rate of 5 mV/s at a speed of 1600 rpm in an O₂ saturated electrolyte. It should be noted that no IR compensation was applied to the electrochemical data throughout this work.

The electrochemical surface area (ECSA) of the catalysts was evaluated by the double-layer capacitance (C_{dl}) measurement method. The C_{dl} was determined according to the CV curves recorded at different

scanning rates of (20, 40, 60, 80 and 100 mV/s) in the non-faradaic potential region. The specific calculation formula is as follows:

$$C_{dl} = \frac{J_a - J_c}{2\nu}$$

J_a and J_c are the anode current density (mA/cm²) and cathode current density (mA/cm²) at the intermediate potential in the select range, and ν is the scanning rate. The durability of ORR and OER for catalysts was characterized by the chronopotentiometric test.

2.7 Assembly and test of rechargeable liquid Zn–air batteries

The key parameters of the homemade rechargeable liquid zinc–air batteries (RLZABs) are summarized in Table S1. The air cathode was fabricated by coating the catalyst onto a nanocomposite substrate, which consisted of hydrophobic carbon paper integrated with a waterproof layer and a current-collecting layer via a rolling process, with a catalyst loading of 1.0 mg cm⁻². A polished zinc plate (0.5 mm thick) served as the anode, while a mixed electrolyte of 6 M KOH containing 0.2 M Zn(Ac)₂ was employed. Electrochemical characterization of the RLZABs included open-circuit voltage (OCV) and polarization curve measurements, which were conducted on a CHI 760E workstation at a scan rate of 10 mV s⁻¹. Galvanostatic discharge–charge cycling and specific capacity tests were performed on a LAND battery testing system (CT2001A) at a current density of 10 mA cm⁻², with each cycle consisting of a 10 min discharge, a 1 min rest, and a 10 min charge (total 21 min per cycle). All measurements were performed under ambient conditions.

2.8 Assembly and test of rechargeable flexible Zn–Air Batteries

Polished zinc foil with a thickness of 0.05 mm was cut into pieces of 6 × 2 cm² to serve as the anode, while the catalyst was loaded onto carbon cloth (WOS1009, 4 × 2 cm²) with a mass loading of 1 mg cm⁻² as the cathode. A piece of nickel foam with dimensions identical to those of the zinc foil was employed as the current collector and placed over the cathode. The gel polymer electrolyte was prepared as follows. First, a mixed solution containing 0.1 M Zn(Ac)₂ and 18.0 M KOH was prepared in a 50 mL volumetric flask. Subsequently, 2.0 g of polyvinyl alcohol (PVA) powder was dissolved in 20.0 mL of deionized water under continuous stirring at 95°C for 1 h using a partitioned heating method to ensure complete dissolution. Then, 2.0 mL of the aforementioned mixed solution was added, and the mixture was further stirred at 95°C for another 1 h. The resulting colloidal solution was poured into a mold (4 cm × 2 cm × 0.3 cm), frozen at – 20°C for more than 12 h, and subsequently thawed at approximately 5°C. The as-prepared gel electrolyte was sandwiched between the carbon cloth (cathode) and the zinc foil (anode), with the nickel foam placed on the outer side of the carbon cloth for current collection. The entire assembly was packaged with aluminum-plastic film to construct a rechargeable flexible zinc–air

battery. The testing protocol for the flexible zinc–air battery was similar with that used for the liquid-state zinc–air battery.

3. RESULTS AND DISCUSSION

The synthesis of FeCo@C/N-GNRs can be briefly divided into three main steps. Firstly, urea and pristine GNRs in a certain mass ratio were thoroughly mixed by mechanical grinding, followed by high-temperature pyrolysis to obtain N-GNRs. Using N-GNRs as the substrate, FeCo-based coordination frameworks were grown on the N-GNR surface via a facile solvothermal method, driven by the strong coordination interactions between $\text{Fe}^{3+}/\text{Co}^{2+}$ and the amine functional groups of p-phenylenediamine^[18]. The resulting mixture was then pyrolyzed at 800°C under a high-purity Ar atmosphere. During pyrolysis, the carbothermal reduction of the organic ligands reduces the metal ions to the metallic state, which in turn catalyzes the growth of carbon layers on the surface of the metal nanoparticles^[25, 26].

To determine the crystal structures of the prepared samples, XRD patterns were measured. As shown in Fig. S1, N-GNRs exhibits two typical diffraction peaks corresponding to the (002) and (101) planes of carbon (PDF#41-1487), consistent with previous reports^[12]. The crystal structure of Co@C is shown in Fig. 1a. Co^{2+} coordinates with p-phenylenediamine in alcohol solution to form complexes, and the Co species in the complexes aggregate during pyrolysis to form nanoparticles; consequently, the XRD pattern is dominated by the diffraction peaks of zero-valent Co (PDF#15-0806). When Fe and Co were simultaneously introduced into the precursor, a phase transition from Co to FeCo alloy occurred, and the XRD patterns confirm the formation of a mixed CoFe alloy phase (PDF#48-1817) in both FeCo@C and FeCo@C/N-GNRs. Since defects are non-negligible active sites for carbon-based bifunctional oxygen electrocatalysts, Raman spectra of the prepared samples were recorded to evaluate the defect degree. As shown in Fig. 1.b, all materials exhibit two prominent peaks at approximately 1350 cm^{-1} (D band) and 1580 cm^{-1} (G band), which can be attributed to disordered carbon and the $\text{E}_{2\text{g}}$ vibration of the sp^2 carbon network, respectively. Accordingly, the relative intensity ratio of the D band to the G band ($I_{\text{D}}/I_{\text{G}}$) is generally used to evaluate the degree of defects in carbon-based materials: a higher ratio indicates a greater degree of defects. The $I_{\text{D}}/I_{\text{G}}$ of N-GNRs is 1.30, indicating that N-GNRs are a defect-rich substrate material. Compared with Co@C ($I_{\text{D}}/I_{\text{G}} = 0.88$), FeCo@C exhibits an $I_{\text{D}}/I_{\text{G}}$ of 1.26, suggesting that the introduction of FeCo alloy can increase the defect degree of the material. Notably, FeCo@C/N-GNRs possesses the highest $I_{\text{D}}/I_{\text{G}}$ value, indicating that this material contains abundant defects—a structural feature that is beneficial for enhancing catalytic activity.

The micromorphology of the samples was observed by transmission electron microscopy. As shown in Fig. 1c, N-GNRs exhibit a typical ribbon-like structure, confirming the successful preparation of graphene nanoribbons, numerous long and narrow graphene nanoribbons are cross-linked with each other, forming a series of pores with varying apertures. For comparison, the typical structure of Co@C is presented in Fig. S2a and b, in which large wrinkled nanosheets are loaded with numerous nanoparticle structures. Upon the introduction of Fe (Fig. S2c and d), the nanosheet morphology transforms from large wrinkled

sheets into small fragmented pieces. The nanosheets remain loaded with metal nanoparticles, but the nanoparticle size increases noticeably. The microstructure of FeCo@C/N-GNRs is shown in Fig. 1d and e, where graphene nanoribbons are interwoven with wrinkled nanosheets derived from the FeCo-based coordination framework to form a substrate, on which uniformly dispersed nanoparticles are supported. To further identify the crystalline phases within the composite, the electron diffraction pattern was collected. From Fig. 1f, FeCo@C/N-GNRs exhibits a polycrystalline structure, and the calculated diffraction rings from inside to outside correspond to the C (002), CoFe (110), CoFe (200), and CoFe (211) planes, respectively, consistent with the XRD analysis. The HRTEM image (Fig. 1g) clearly demonstrates that the metal nanoparticles are encapsulated by carbon layers, and the lattice spacing in the core region is 0.203 nm, corresponding to the (110) plane of the FeCo alloy. Encapsulating metal nanoparticles within carbon shells can effectively prevent their aggregation and dissolution during electrochemical reactions under alkaline conditions. Finally, the elemental mapping images (Fig. 1h–m) reveal that the as-prepared sample contains uniformly distributed C, N, O, Fe, and Co.

The specific surface areas and pore structures of the synthesized samples were measured at 77 K. As demonstrated in Fig. 2a, all materials exhibit a sharp increase in N₂ uptake in the low relative pressure region (~ 0–0.05), which is associated with micropore filling and indicates the presence of micropores. A pronounced hysteresis loop appears in the relative pressure range of 0.4–1.0, characteristic of a typical type IV adsorption/desorption isotherm, reflecting the coexistence of mesopores and macropores. The specific surface area of FeCo@C/N-GNRs reaches 487.53 m² g⁻¹, superior to those of the control samples Co@C (214.69 m² g⁻¹), FeCo@C (277.03 m² g⁻¹), and N-GNRs (272.57 m² g⁻¹). The pore size distributions of the samples are shown in Fig. 2b; all samples possess hierarchical pore structures, among which FeCo@C/N-GNRs exhibits the most prominent microporous and mesoporous characteristics, consistent with the analysis of the adsorption–desorption curves. A large specific surface area is beneficial for the sufficient exposure of active sites, while the hierarchical pore structure facilitates the adsorption of gas molecules, the occurrence of electrochemical reactions, and the rapid transport of relevant reactive species, thereby accelerating the kinetics of both OER and ORR.

Beyond the textural properties, the surface chemical composition and elemental states of the catalysts also play a decisive role in electrocatalytic performance, particularly since both ORR and OER are typical three-phase interfacial reactions occurring at the catalyst surface. Therefore, XPS characterization was performed. As shown in Fig. 2c, the high-resolution C 1s spectrum can be deconvoluted into C–C/C = C, C–O, C = O/C–N, and O–C = O components, indicating the successful doping of N into the carbon framework. The high-resolution N 1s spectrum (Fig. 2d) can be divided into five peaks, corresponding to pyridinic-N (398.1 eV), metal-N (398.9 eV), pyrrolic-N (399.8 eV), graphitic-N (400.9 eV), and oxidized-N (402.8 eV)^[27]. The presence of metal-N species can be regarded as evidence of interfacial chemical bonding between the FeCo alloy and the carbon shell^[28]. In addition, it should be noted that different N configurations exhibit distinct activities: pyridinic N can stimulate the formation of active sites by modifying the local electronic structure of carbon, while graphitic N can promote the four-electron pathway in oxygen-related reactions [29]. To further elucidate the chemical states of the metal species,

the Fe 2p and Co 2p spectra were analyzed. As shown in Fig. 2e and f, both the Fe 2p and Co 2p spectra exhibit zero-valent metal peaks, while the other pair of XPS peaks and their satellite peaks correspond to the ionic states of Fe and Co. Previous studies have demonstrated that the presence of Fe and Co ionic states can facilitate electron transfer and reduce interfacial resistance^[18]. Moreover, the coexistence of metallic and ionic states of Fe and Co indicates a strong interaction between the FeCo alloy and the carbon layers, a feature that is beneficial for enhancing catalytic activity^[25, 22, 30].

To evaluate the electrocatalytic activity of the prepared samples, CV curves were first measured. As shown in Fig. S3, N-GNRs, Co@C, FeCo@C, and FeCo@C/N-GNRs exhibit no oxygen reduction peaks in N₂-saturated electrolyte, whereas oxygen reduction peaks appear in O₂-saturated conditions, indicating that all prepared samples possess certain ORR activity. The ORR activity can be roughly compared based on the peak potential in the CV curves: a more positive peak potential corresponds to higher ORR activity. Comparison of the CV curves of all catalysts illustrate that FeCo@C/N-GNRs exhibits the most positive peak potential (~ 0.844 V), close to that of commercial Pt/C. A more detailed comparison of ORR activity requires reference to the important ORR parameters E_{onset} , $E_{1/2}$, and J_L ; therefore, LSV curves of all catalysts were recorded. As shown in Fig. 3a and Table S2, FeCo@C/N-GNRs exhibits a large E_{onset} (0.922 V), and $E_{1/2}$ (0.830 V), surpassing the most control samples N-GNRs (E_{onset} = 0.921 V, $E_{1/2}$ = 0.751 V), Co@C (E_{onset} = 0.874 V, $E_{1/2}$ = 0.779 V), and FeCo@C (E_{onset} = 0.877 V, $E_{1/2}$ = 0.807 V), and approaching commercial Pt/C (E_{onset} = 0.938 V, $E_{1/2}$ = 0.840 V). The ORR kinetics of the catalysts were analyzed based on the Tafel slope. As shown in Fig. 3b, the Tafel slope of FeCo@C/N-GNRs is 83.2 mV dec⁻¹, which is not only superior to those of the control samples except FeCo@C but also surpasses that of the benchmark commercial Pt/C (83.8 mV dec⁻¹), indicating that FeCo@C/N-GNRs possesses a fast ORR kinetic process. The ORR pathway of the prepared catalysts was analyzed by LSV curves at different rotation speeds. As shown in Fig. 3c and Fig. S4a, c, e and g, as the rotation speed increases from 625 to 2500 rpm, the limiting current density of the catalysts increases steadily, consistent with the accelerated diffusion of O₂ molecules in the electrolyte toward the electrode surface. The inset of Fig. 3c and Fig. S4b, d, f and h demonstrate that the K–L plots fitted at different potentials (0.1 V, 0.2 V, and 0.3 V) exhibit good linearity and high consistency, implying first-order reaction kinetics with respect to dissolved oxygen concentration^[31]. Based on the average electron transfer number calculated from the K–L equation, FeCo@C/N-GNRs is the best among all synthesized catalysts, following a four-electron transfer pathway. Combined with the above analysis, it can be confirmed that FeCo@C/N-GNRs is an ORR catalyst comparable to commercial Pt/C.

The OER activity of the synthesized catalysts was further evaluated in an O₂-saturated electrolyte, and the corresponding curves and electrochemical parameters are presented in Fig. 3d and Table S3. Figure 3d shows the OER polarization curves of the catalysts. FeCo@C/N-GNRs exhibits an overpotential of 300 mV at 10 mA cm⁻², which is lower than those of the control samples N-GNRs (429 mV), Co@C (379 mV), and FeCo@C (345 mV), and also lower than that of the benchmark IrO₂ (362 mV). The OER kinetics of the catalysts were analyzed from the Tafel plots fitted from the LSV curves. From Fig. 3e, the Tafel slope of FeCo@C/N-GNRs is only 63.6 mV dec⁻¹, whereas those of N-GNRs, Co@C, FeCo@C, and

IrO₂ are 132.8, 111.1, 94.5, and 91.1 mV dec⁻¹, respectively. Combining the OER overpotential and Tafel slope (Fig. S5), it can be concluded that the prepared FeCo@C/N-GNRs catalyst possesses the best OER activity and OER kinetics in this work.

To further understand the underlying reasons for the enhanced electrocatalytic activity, the ECSA of the catalysts was analyzed. CV curves of all prepared catalysts were recorded in the non-faradaic region (Fig. 3f and Fig. S6), and the fitting lines of the current density difference (ΔJ) versus scan rate at a potential of 1.15 V were plotted. The calculated C_{dl} values are shown in Fig. 3g. The variation trend of C_{dl} values is similar to that of the specific surface areas, with FeCo@C/N-GNRs exhibiting the largest C_{dl} value of 12.88 mF cm⁻², implying that FeCo@C/N-GNRs provides the most abundant electrochemically accessible active sites in this work. The stability of the catalyst is also one of the important indicators for catalyst evaluation. To analyze the catalytic stability of FeCo@C/N-GNRs, chronoamperometry was employed to measure the time–current curves for ORR and OER at 0.7 V and 1.6 V, respectively. As shown in Fig. 3h, after 40,000 s of continuous testing, the ORR current density of FeCo@C/N-GNRs decreased by only 16.21%, whereas that of commercial Pt/C decreased by 34.66%. Similarly, after about 11 h of testing, the OER current density of FeCo@C/N-GNRs decreased by 15.95%, while the response current density of IrO₂ decreased by 63.69%. These results demonstrate that FeCo@C/N-GNRs possesses excellent ORR/OER durability.

The potential difference (ΔE) between the OER potential at 10 mA cm⁻² and the ORR half-wave potential is commonly used to evaluate the bifunctional activity of catalysts. Accordingly, the ΔE values of the prepared catalysts were calculated, and the results are shown in Fig. 3i. The ORR/OER potential difference of FeCo@C/N-GNRs is 0.70 V, which is considerably smaller than those of the other control samples in this work and also superior to that of Pt/C + IrO₂ ($\Delta E = 0.752$ V), indicating that FeCo@C/N-GNRs possesses excellent ORR/OER performance. Furthermore, as shown in Fig. 3j and Table S4, the bifunctional activity of FeCo@C/N-GNRs also outperforms that of most reported non-precious-metal bifunctional catalysts. Combining the physical characterization results, the high electrocatalytic activity and stability of FeCo@C/N-GNRs can be attributed to the following aspects: (1) GNRs with abundant edge structures were selected as the carbon substrate, endowing the resulting FeCo@C/N-GNRs composite with a high degree of defects, which creates more ORR/OER active sites in the catalyst. (2) The synergistic effect arising from the different polarities of the alloy components effectively induces electron redistribution within the uniformly distributed FeCo alloy nanoparticles, thereby lowering the reaction energy barrier [32]. (3) The composite pyrolysis of graphene nanoribbons with the FeCo-based coordination framework greatly increases the specific surface area and electrochemical surface area of the material and constructs a hierarchical pore structure with coexisting micropores, mesopores, and macropores, which not only facilitates the sufficient exposure of active sites but also promotes electron and mass transport during the reaction. (4) The high stability can be attributed to the nitrogen doping of the graphene nanoribbons and the encapsulation of FeCo nanoparticles within carbon layers: the former effectively enhances both the catalytic activity and stability of the material, while the latter effectively prevents the aggregation and detachment of metal nanoparticles during the reaction. The excellent

bifunctional oxygen electrocatalytic activity and durability of the FeCo@C/N-GNRs catalyst are conducive to its further application in rechargeable Zn-air batteries.

Based on the above analysis, rechargeable liquid Zn-air batteries (RLZABs) were assembled and tested using FeCo@C/N-GNRs and Pt/C + IrO₂ as air cathode catalysts. As shown in Fig. 4a, the open-circuit voltage of the FeCo@C/N-GNRs-based RLZABs is 1.49 V, higher than that of the Pt/C + IrO₂-based RLZABs (1.40 V) and closer to the theoretical voltage of Zn-air batteries (1.66 V). The charge–discharge polarization curves of the RLZABs based on different catalysts are shown in Fig. 4b. Compared with the Pt/C + IrO₂-based battery, the RLZABs using FeCo@C/N-GNRs as the air cathode catalyst exhibit a smaller voltage gap at different current densities, indicating superior electrocatalytic activity, consistent with the electrochemical test results. The power density curves calculated from the discharge polarization curves (Fig. 4c) show that the FeCo@C/N-GNRs-based RLZABs achieve a maximum power density of 165.83 mW cm⁻² at a current density of 264.11 mA cm⁻², outperforming the Pt/C + IrO₂-based battery (92.21 mW cm⁻² at 137.72 mA cm⁻²). The galvanostatic discharge test at a current density of 10 mA cm⁻² (Fig. 4d) shows that the specific capacity of the FeCo@C/N-GNRs-based RLZABs is 789 mAh g_{Zn}⁻¹, reaching 96.2% of the theoretical value (820 mAh g_{Zn}⁻¹), whereas the specific capacity of the Pt/C + IrO₂-based battery is only 739 mAh g_{Zn}⁻¹. The stability of the battery was evaluated by long-term charge–discharge cycling tests. The Pt/C + IrO₂-based battery failed due to over-discharge after approximately 330 cycles, whereas the FeCo@C/N-GNRs-based RLZABs continued to operate normally after 780 cycles (Fig. 4e), demonstrating a satisfactory stability.

Furthermore, given that wearable devices and flexible energy storage devices have become a research hotspot in recent years, FeCo@C/N-GNRs and Pt/C + IrO₂ catalysts were further assembled into rechargeable flexible Zn-air batteries (RFZABs) to explore their application in flexible devices. The typical structure of the RFZABs is shown in Fig. 5a, in which nickel foam serves as the current collector, flexible carbon cloth loaded with the bifunctional electrocatalyst serves as the air cathode, a KOH-containing PVA alkaline gel serves as the solid-state electrolyte in the middle, and zinc foil serves as the anode; all materials are encapsulated with an aluminum-plastic film in the order illustrated. The charge–discharge polarization curves (Fig. 5b) indicate that the FeCo@C/N-GNRs-based RFZABs exhibit a smaller voltage gap, consistent with the previous electrochemical and RLZAB tests. Based on the discharge polarization curves, the peak power density of the FeCo@C/N-GNRs-based RFZABs is 53.92 mW cm⁻², whereas that of the Pt/C + IrO₂-based RFZABs is only 30.95 mW cm⁻², demonstrating the superior performance of the former. The charge–discharge voltage curves in Fig. 5c confirm that the FeCo@C/N-GNRs-based RFZABs can maintain stable power output at various bending angles, indicating good flexibility and application prospects. Cyclic charge–discharge testing to verify the stability of the RFZABs is of great significance for their practical application. As shown in Fig. 5d, after 45 charge–discharge cycles, the Pt/C + IrO₂-based RFZABs exhibit significant performance degradation with a substantial drop in discharge voltage, whereas the FeCo@C/N-GNRs-based battery shows stable voltage performance under the same testing conditions with negligible voltage decay. Three FeCo@C/N-GNRs-based RFZABs connected in series (Fig. 5e) can stably light up an LED strip for more than 24 h without noticeable

brightness decay. The characterization results of both the RLZABs and RFZABs demonstrate that FeCo@C/N-GNRs is a stable and efficient bifunctional oxygen electrocatalyst with tremendous application potential in the field of Zn-air batteries.

4. CONCLUSION

In summary, FeCo alloy nanoparticles encapsulated within carbon layers bridging N-doped graphene nanoribbons (FeCo@C/N-GNRs) were successfully fabricated via a facile mechanical grinding and pyrolysis strategy. The resulting product possesses a hierarchical porous architecture with coexisting micropores, mesopores, and macropores, which maximizes the electrochemically accessible surface area and facilitates active-site exposure. Electrochemical measurements demonstrate that FeCo@C/N-GNRs functions as an excellent bifunctional oxygen electrocatalyst, delivering an ORR half-wave potential of 0.830 V, OER potential of 1.530 V at 10 mA cm⁻², and a small bifunctional parameter ΔE of only 0.70 V, surpassing benchmark Pt/C + IrO₂ and most reported bifunctional catalysts. Chronoamperometric tests confirm its superior ORR/OER durability. When employed as the cathode in both liquid and flexible rechargeable Zn-air batteries, FeCo@C/N-GNRs achieves higher peak power densities (165.83 and 53.92 mW cm⁻²) and enhanced stability relative to Pt/C + IrO₂-based batteries. Notably, the flexible batteries maintain stable power output under various bending angles, demonstrating substantial potential for portable and wearable energy devices.

Declarations

Conflict of interests

The authors declare that they have no conflict of interest.

Author Contribution

Chao Cheng: Conceptualization, Methodology, Software, Writing – original draft; Yongqiang Ming: Supervision, Writing – review & editing; Luping Liu: Supervision, Data curation; Yunjian Liu: Resources, Data curation; Juan Yang: Supervision, Writing – review & editing, Investigation.

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Figures

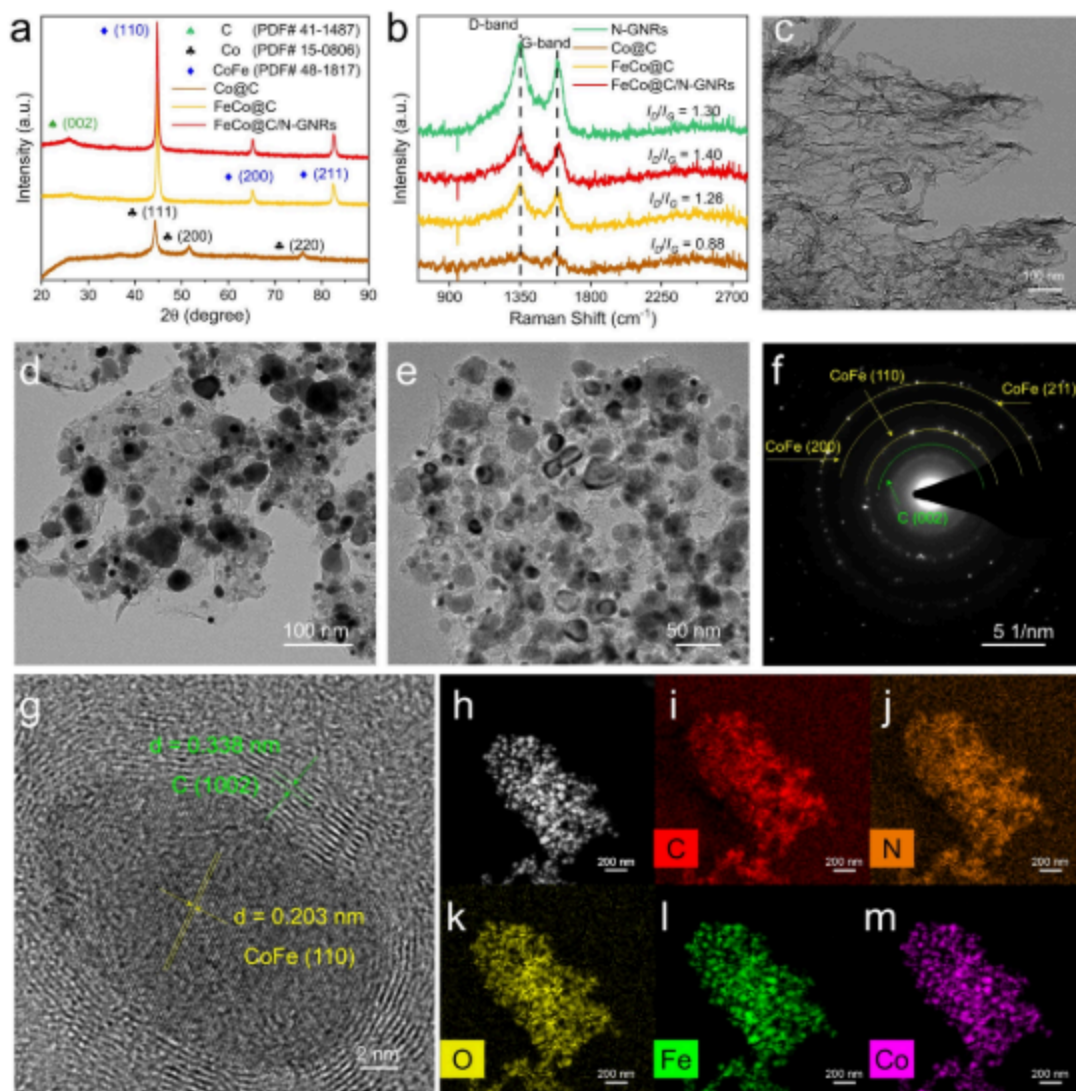


Figure 1

(a) XRD patterns; (b) Raman spectra; TEM images of N-GNRs (c) and FeCo@C/N-GNRs (d, e); the selected area electron diffraction (f) and the HRTEM image (g) of FeCo@C/N-GNRs; (h-m) high-angle annular dark-field scanning image and corresponding EDS elemental maps of C, N, O, Fe and Co in FeCo@C/N-GNRs

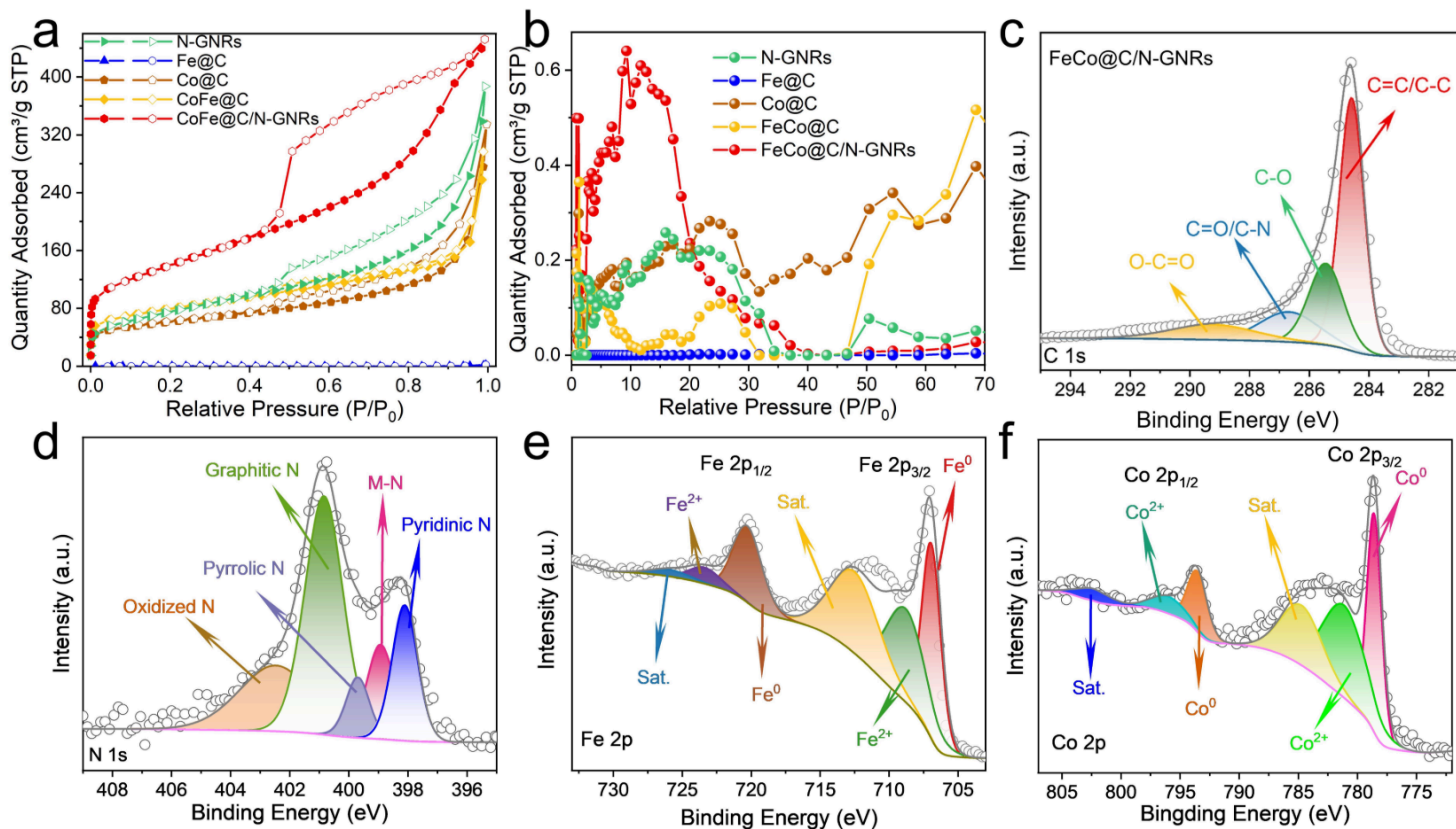


Figure 2

(a) N_2 adsorption–desorption isotherms; (b) Pore size distribution; XPS spectra of C 1s (c), N 1s (d), Fe 2p (e) and Co 2p (f) for FeCo@C/N-GNRs

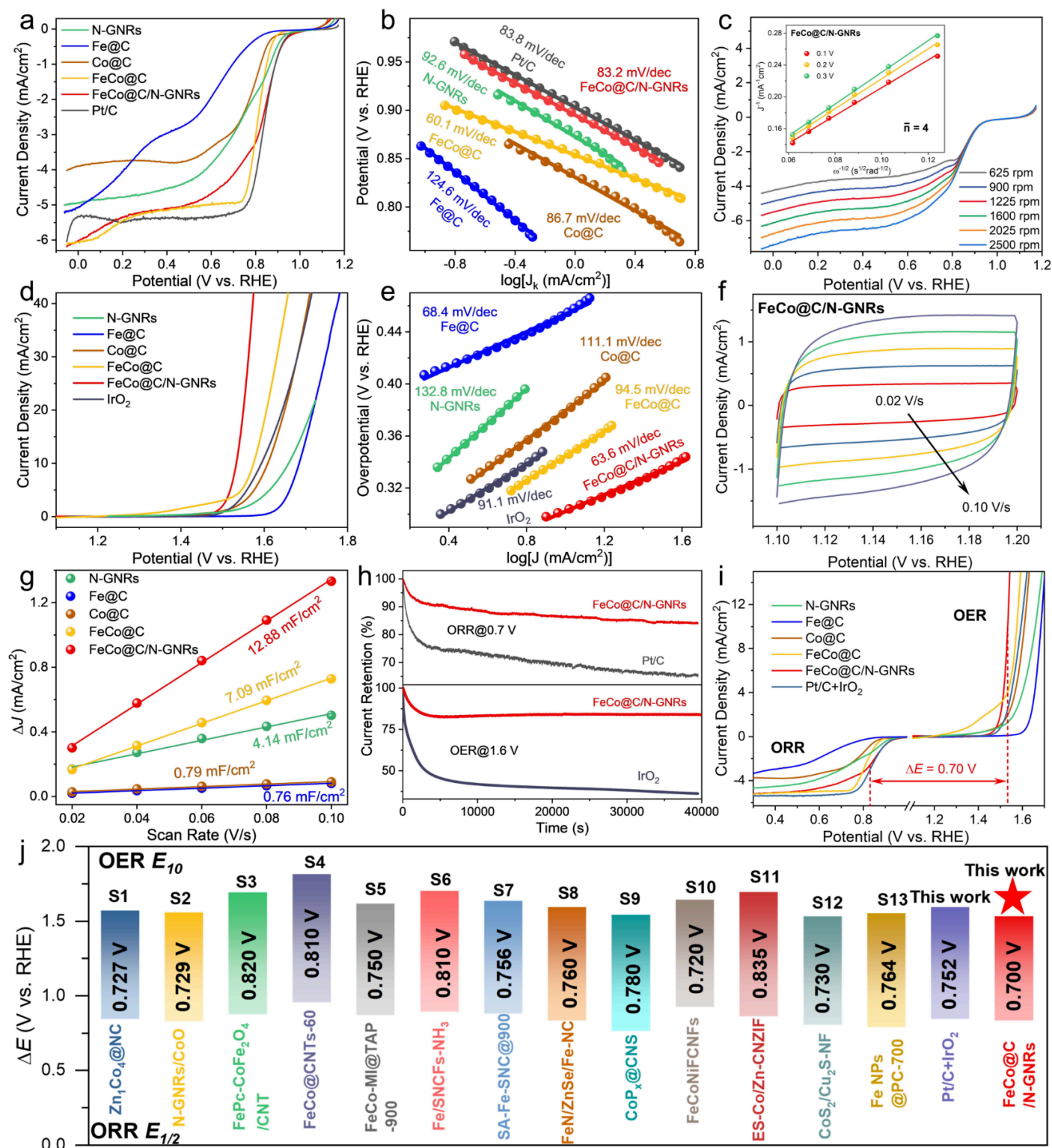


Figure 3

LSV curves (a) and Tafel slope (b) of ORR process for all catalysts; (c) LSV curves for ORR at the speed of 1600 rpm in O₂-saturated 0.1 M KOH and corresponding K–L plots (insert picture) for FeCo@C/N-GNRs; (d) LSV polarization curves for OER; (e) corresponding Tafel slopes for OER; (f) CV curves of FeCo@C/N-GNRs in no faradaic region at different scan rates; (g) current density difference (ΔJ) at 1.15 V plotted against scan rates; (h) durability tests of FeCo@C/N-GNRs for ORR and OER in comparison

with Pt/C and IrO₂ Comparison of ΔE values for (i) the prepared catalysts and (j) other reported bifunctional catalysts

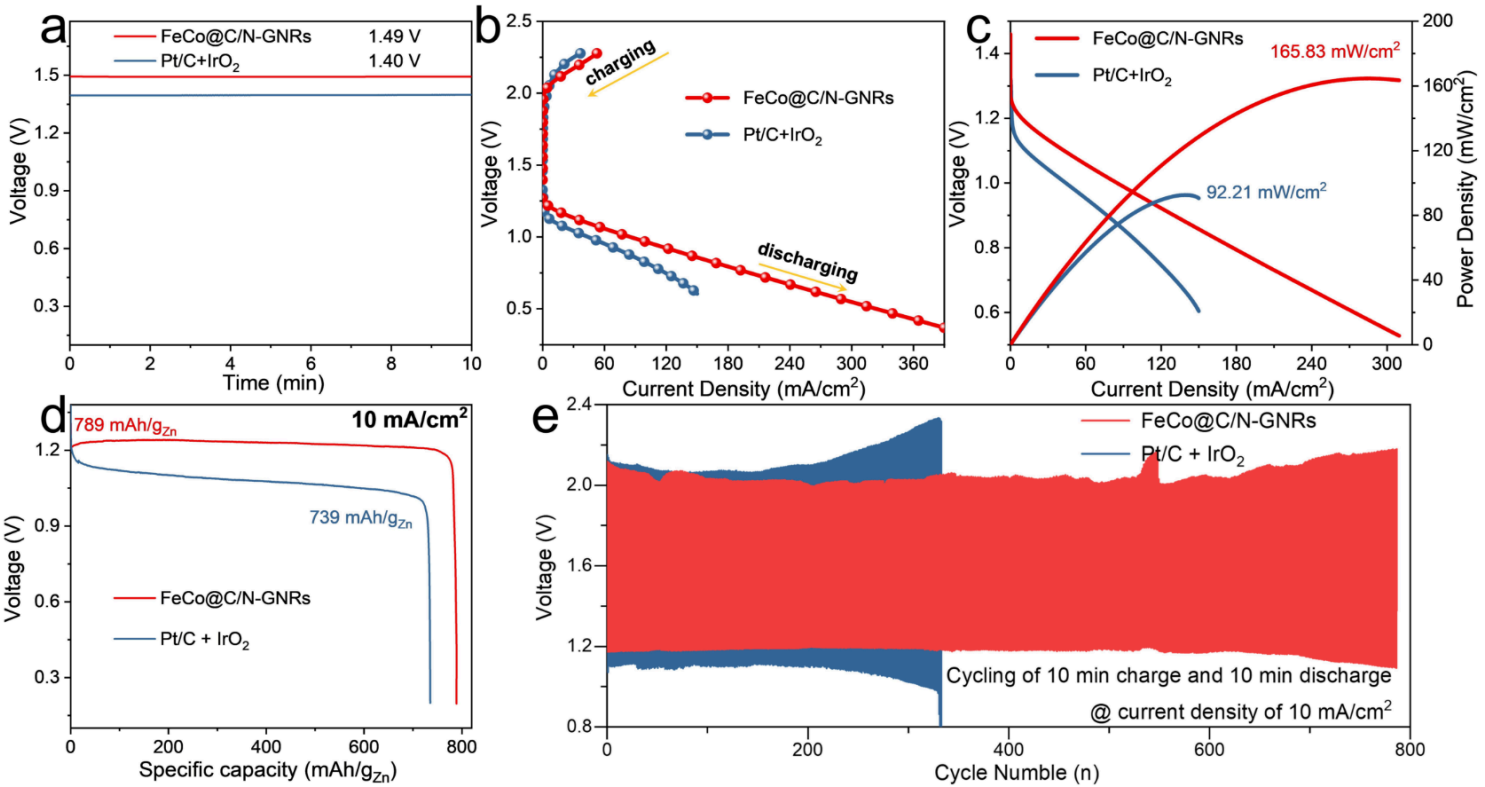


Figure 4

(a) OCV of FeCo@C/N-GNRs- and Pt/C+IrO₂-based RLZABs; (b) charging polarization curves and discharging polarization curves; (c) discharging curves and corresponding power density curves; (d) discharge curves at the constant current density of 10 mA/cm² for RLZABs with FeCo@C/N-GNRs and Pt/C+IrO₂ catalysts; (e) galvanostatic discharging-charging curves at 10 mA/cm²

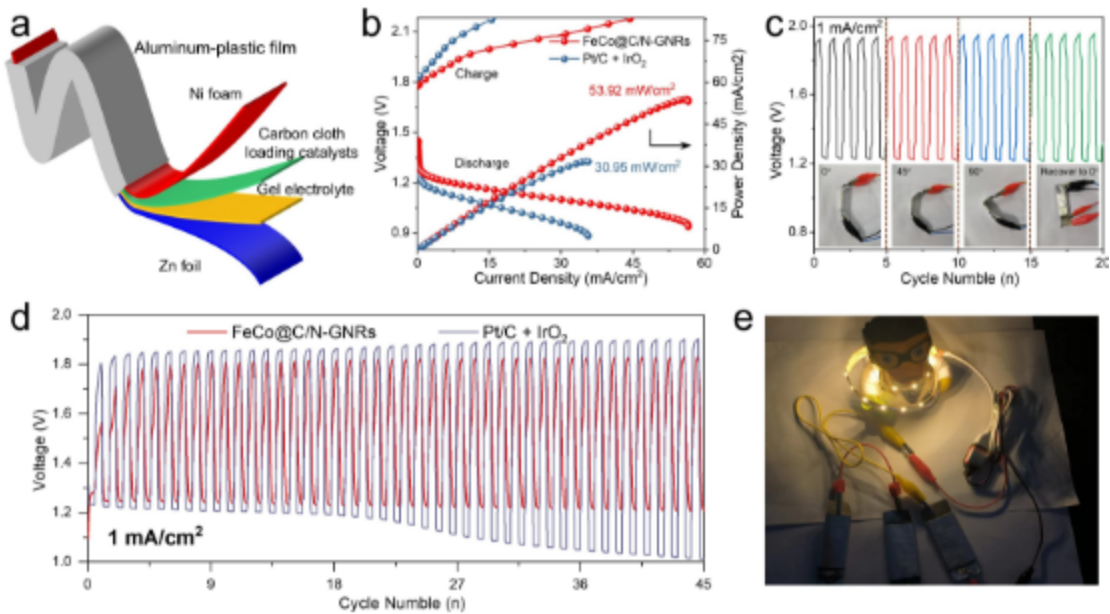


Figure 5

(a) Schematic diagram of RFZABs; (b) charging, discharging the corresponding power density curves of RFZABs based on FeCo@C/N-GNRs and Pt/C+IrO₂; (c) discharging–charging cycling curves of RFZABs at different bending states; (d) discharging–charging cycling stability tests at the constant current density of 1 mA/cm²; (e) three FeCo@C/N-GNRs-based RFZABs series lighting a LED light strip

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