

Saline Water Electrolysis

Ultrafast Synthesis of IrB_{1.15} Nanocrystals for Efficient Chlorine and Hydrogen Evolution Reactions in Saline Water

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Abstract: The production of storable hydrogen fuel through water electrolysis powered by renewable energy sources such as solar, marine, geothermal, and wind energy presents a promising pathway toward achieving energy sustainability. Nevertheless, state-of-the-art electrolysis requires support from ancillary processes which often incur financial and energy costs. Developing electrolyzers capable of directly operating with water that contains impurities can circumvent these processes. Herein, we demonstrate the efficient and durable electrolysis of saline water to produce chlorine gas (Cl₂) and hydrogen using structurally ordered IrB_{1.15}, synthesized through ultrafast joule heating. IrB_{1.15} exhibits remarkable performance, achieving overpotentials of 75 mV for the chlorine evolution reaction (CER) and 12 mV for hydrogen evolution reactions (HER) at current densities of 10 mA cm⁻². Moreover, IrB_{1.15} displays a durability of over 90 h towards both CER and HER. Density functional theory reveals that IrB_{1.15} has adsorption energies significantly closer to 0 eV for Cl and H, compared to IrO₂ and Pt/C. Furthermore, in situ Raman investigations reveal that Ir in IrB_{1.15} serves as the active center for CER, while the introduction of B atoms to Ir lattices mitigates the formation of adsorbed hydrogen species on the Ir surface, thereby enhancing the performance of IrB_{1.15} in HER.

Introduction

Chlorine gas (Cl₂) is a vital chemical compound, with an annual production exceeding 70 million tons. It is extensively utilized in various applications such as water treatment, organic synthesis, and polyvinyl chloride manufacturing.^[1–3] The electrolysis of saline water under strongly acidic conditions (pH ~2) enables efficient chlorine evolution reaction (CER) at the anode, providing a viable approach for Cl₂ production.^[4–6] Simultaneously, the hydrogen gas generated at the cathode through the hydrogen evolution reaction (HER) can replace carbon-based energy sources.^[7–10] Achieving high activity, stability, and selectivity for both CER and HER necessitates the use of advanced electrocatalysts.^[11,12] Currently, Ru/Ir oxide and Pt/C represent the state-of-the-art electrocatalyst for CER and HER, respectively.^[13,14] However, noble materials incur substantial costs and exhibit limited long-term operational stability. For instance, Ru-based CER catalysts lack sufficient stability during prolonged operation due to the formation of soluble ruthenium chloride.^[15] Generally, compared to Ru-based materials, Ir-based materials exhibit superior catalytic stability but lower activity.^[16] Controlling the proper amount of Ir in the electrocatalysts can improve the catalytic activity while maintaining the operational stability of CER catalysts. It is crucial to concurrently mitigate the usage of Ir-based materials and enhance the CER performance to align with the sustainable development goals.

In recent years, Ir-based compounds, including oxides, phosphides (Ir₂P, IrP₂), chalcogenides (IrS₂, IrSe₂), and silicides (IrSi), have been investigated and showed high HER performance.^[17–24] Beyond noble metal materials, transition metal borides (TMBs) represent another distinct family. They integrate M–M metallic bonds, M–B ionic bonds, and B–B covalent bonds, offering a diverse range of properties, including exceptional hardness, high-temperature resistance, oxidation resistance, remarkable thermochemical stability, relatively low electrical resistivity, and good mechanical properties.^[25–28] To date, the systematic investigation of Ir-based ordered borides as electrocatalysts for CER and HER remains unexplored. The possible reason lies in the challenges associated with the catalyst fabrication under ambient pressure and moderate temperatures.^[29]

Fortunately, the technique of Joule heating offers a rapid flash heating and cooling capability to the samples, enabling ramp rates up to the order of 10⁵ K s⁻¹. This rapid and instantaneous temperature fluctuation completes the reaction within a few seconds. More importantly, the extremely

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short calcination time (in seconds) prevents the sintering and agglomeration of active components that can occur during prolonged heat treatment, ensuring rapid and effective dispersion of active atoms. This process facilitates precise regulation of the particle size, composition, and structure of the synthesized material.^[30] Recently, ultrafast Joule heating has been employed to synthesize intermetallic compounds with small nanoparticle sizes, including Pd₃Pb, metal carbides, metal chalcogenides, nanocrystals of high-entropy alloys and 2D materials.^[31–35]

In our work, using an ultrafast Joule heating-assisted strategy, we achieved the ordered incorporation of light boron (B) atoms into Ir lattices. Thanks to the interstitial effect mediated by B, the obtained intermetallic IrB_{1.15} demonstrated excellent CER and HER activity and stability in acidic media. Specifically, the IrB_{1.15} exhibited remarkable performance in CER and HER, achieving overpotentials of 75 mV and 12 mV, respectively, at current densities of 10 mA cm⁻². In situ detection techniques further unveiled that the presence of Ir in IrB_{1.15} served as the active center for CER, while the incorporation of B atoms into the Ir lattices facilitated the coordination of hydrogen species on the Ir surface, thereby enhancing the HER performance of IrB_{1.15}. Moreover, density functional theory (DFT) calculations indicate that IrB_{1.15} exhibits adsorption energies for Cl and H closer to 0 eV compared to commercial IrO₂. These factors contribute significantly to the enhanced catalytic activity of IrB_{1.15} in both CER and HER. Additionally, the strong chemical bonding and periodic atomic arrangement in the ordered IrB_{1.15} structure inhibit the dissolution and reconstruction of the active Ir, ensuring catalytic stability. Consequently, IrB_{1.15} displayed excellent durability for over 90 h in both CER and HER.

Results and Discussion

The synthesis mechanism for IrB_{1.15} is depicted in Figure 1a. First, the precursors of ammonium hexachloroiridate (IV) and sodium borohydride were thoroughly ground for approximately 10 minutes. The precursor mixture was then subjected to ultrafast Joule heating at 900 °C for 12 seconds under an argon flow. Finally, the mixture was cooled, and the byproducts (Na⁺, Cl⁻, etc.) were thoroughly rinsed with deionized water. Consequently, the product of IrB_{1.15} was successfully obtained.

The morphology of the IrB_{1.15} sample is initially characterized using high-angle annular dark-field scanning transmission electron microscopy (HAADF-STEM). The TEM images presented in Figures 1b–c demonstrate the presence of numerous nanoparticles within IrB_{1.15}, with particle diameters ranging from 2 to 16 nm and an average diameter of approximately 4 nm (Figure S1). Therefore, further control should be implemented in future research to enhance the homogeneity of the resulting nanoparticles. Additionally, the HRTEM image in Figure 1d reveals a well-defined lattice fringe measuring 2.71 Å, which closely corresponds to the observed diffraction pattern associated with the (101) crystal plane of the tetragonal phase IrB_{1.15}.

HAADF-STEM and EDS elemental mapping images reveal a uniform distribution of both Ir and B elements within the IrB_{1.15} particle (Figures 1e–g and Figure S2). Moreover, as demonstrated in Figures 2a and 2b, the atomic arrangement in specific regions exhibits remarkable conformity with the projected crystal structure along the [−110] orientation for IrB_{1.15} (Figure 2c), as observed through HADDF and bright-field (BF) microscopy. These characterizations fully confirm the formation of intermetallic IrB_{1.15} and the ordered filling of B atoms into Ir lattices.

The structure of IrB_{1.15} was determined using X-ray powder diffraction (XRD). As shown in Figure 2d, the observed peaks at 2θ angles of 33.0°, 34.9°, 41.6°, 49.2°, 55.5°, 66.4°, 72.9°, 73.9°, 76.4° and 77.4°, can be assigned to the (101), (004), (103), (112), (105), (200), (116), (008), (211) and (204) crystal planes, respectively, indicating an tetragonal IrB_{1.15} phase with the I4₁/amd space group [PDF No. 13–0363, I4₁/amd(141), a₀=2.81 Å, b₀=2.81 Å, c₀=10.26 Å]. The X-ray photoelectron spectroscopy (XPS) survey (Figure S3) reveals distinct signals corresponding to the presence of B and Ir elements in IrB_{1.15}. In the high-resolution Ir 4f spectra (Figure 2e), the observed binding energies at 60.6 and 63.5 eV in the Ir 4f spectrum are consistent with those expected for IrB_{1.15}, specifically corresponding to the Ir 4f_{7/2} and Ir 4f_{5/2} core levels, respectively.^[36] The B 1s spectrum of IrB_{1.15} is presented in Figure 2f, wherein the two distinct subpeaks at approximately 186.9 and ~188.5 eV are ascribed to the presence of Ir–B bond within IrB_{1.15}, while the peaks observed at around 189.9 and 191.8 eV for the B–O bond are attributed to BO_x species resulting from surface oxidation of boron.^[37] It is noteworthy that following the introduction of B, the binding energy of IrB_{1.15} exhibits a negative shift (~0.2 eV) in comparison to the metallic state Ir (60.8 eV).^[38] The aforementioned observation implies that the introduced B atom establishes a donor-acceptor architecture with Ir based on disparities in electronegativity, thereby generating an electron-rich environment for Ir atoms.

We evaluated the CER performance of IrB_{1.15} in solutions with different NaCl concentrations at pH=2, and also conducted on commercial IrO₂ for comparison purposes. We found that the current densities increase with the Cl⁻ concentrations (Figure S4). It is worth noting that Figure 3a also illustrates linear sweep voltammetry (LSV) of IrB_{1.15} in 0.1 M HClO₄ solutions (pH = 1.45, Figure S5). The selection of HClO₄ as the appropriate electrolyte is based on its suitability due to the presence of perchlorate ions (ClO₄⁻), which are fully oxidized oxyanions of chlorine. Under anodic bias, no further oxidation of these anions occurs except for the oxygen evolution reaction (OER). As depicted in Figure 3a, during anodic polarization in the presence of chloride containing 4.0 M NaCl, the onset potential for IrB_{1.15} is approximately 1.4 V (vs RHE, green curve), which exhibits a reduction of ~150 mV (red curve) compared to that recorded in the absence of chloride solutions (0.1 M HClO₄). In other words, the cathodic shift observed in the LSV under 4.0 M NaCl, as compared to that under 0.1 M HClO₄, suggests a significantly higher propensity for CER over OER in the chloride-containing electro-

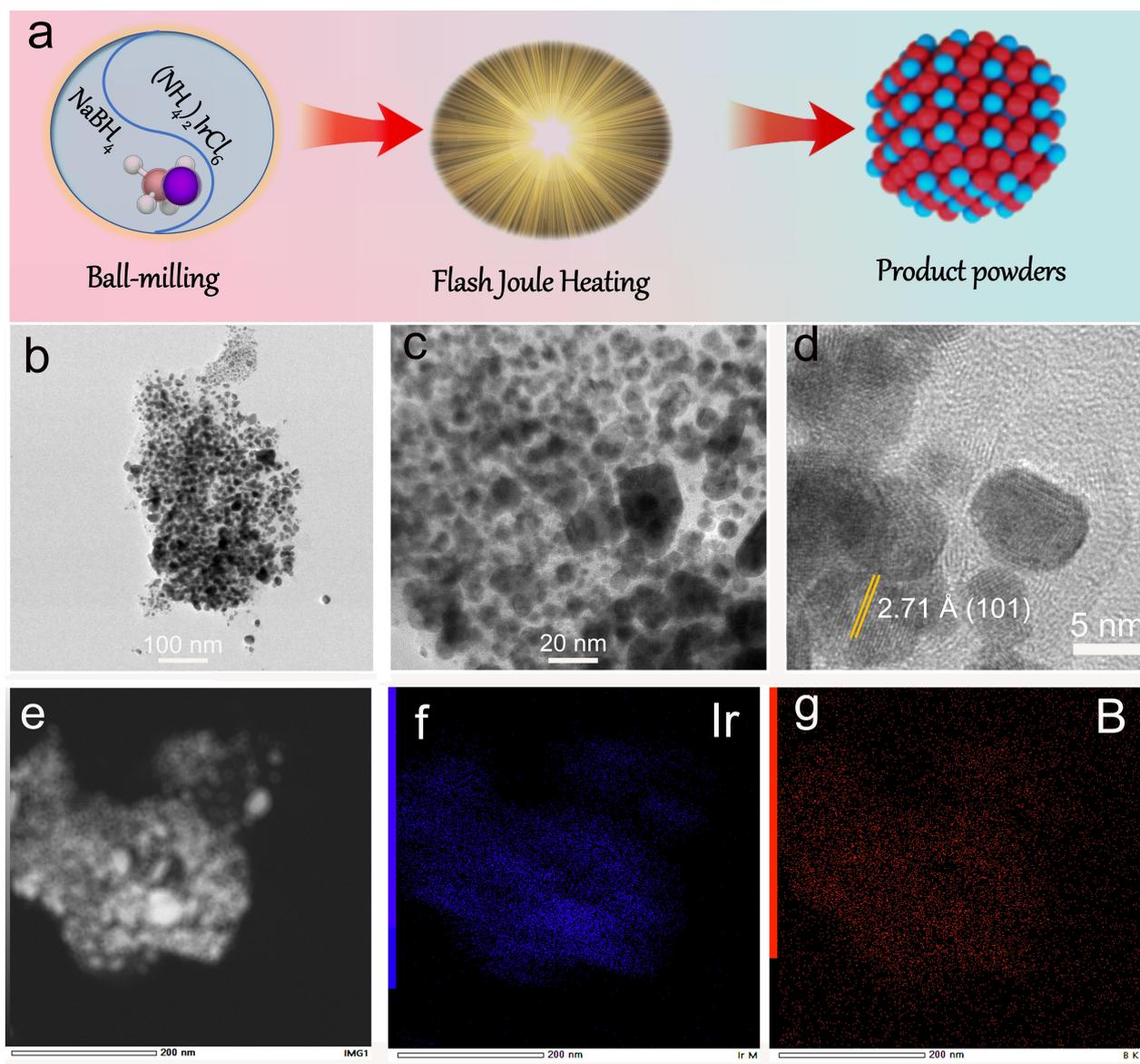


Figure 1. (a) Schematic illustration of the preparation of IrB_{1.15}. (b) Low and (c) high magnification TEM images of IrB_{1.15}. (d) HRTEM image of IrB_{1.15}. (e-g) HAADF images and EDX elemental mapping of Ir, and B, of IrB_{1.15}.

lyte. Additionally, as evidenced by the LSV curves, IrB_{1.15} exhibits a rapid initiation with a pronounced surge in the anodic current density. In detail, to reach a current density of 10 mA cm⁻², IrB_{1.15} only requires an overpotential of 75 mV, exhibiting a Tafel slope 62 mV dec⁻¹, significantly lower than that observed for the commercial IrO₂ (89 mV and 72 mV dec⁻¹) (Figures 3b and 3c). Furthermore, this value compares favorably with the behaviors of most Ir/Ru-based CER catalysts reported to date (Figure 3d and Table S1). The exceptional intrinsic activity of IrB_{1.15} is further supported by its reduced charge transfer resistance (R_{ct}) and superior electrochemical surface area (ECSA) (Figures 3e and 3f).^[4] Specifically, Nyquist plots presented in Figure 3e demonstrate that IrB_{1.15} has a lower R_{ct} compared to commercial IrO₂, providing further evidence for the enhanced CER charge transfer process facilitated by

B incorporation in IrB_{1.15}. Additionally, the cyclic voltammetry (CV) measurements conducted within a non-faradic potential range (0.75–0.95 vs RHE) reveal a higher ECSA of IrB_{1.15}, as indicated by the obtained value of double-layer capacitance (C_{dl}) (Figure 3f and Figure S6). This observation suggests that the incorporation of B can stimulate an increased number of accessible active Ir sites. More importantly, normalizing the polarization curves by ECSA and the mass of Ir further indicates IrB_{1.15} also possesses high intrinsic catalytic activity toward CER (Figure S7). It is noteworthy that the Cl₂ selectivity serves as another crucial criterion for CER electrocatalysts in the chlor-alkali industry, which was assessed here using the rotating ring-disk electrode (RRDE) technique. As depicted in Figure S8, IrB_{1.15} exhibits an impressive Cl₂ selectivity of 98.3%. The high Cl₂ selectivity of IrB_{1.15} can be attributed to its superior

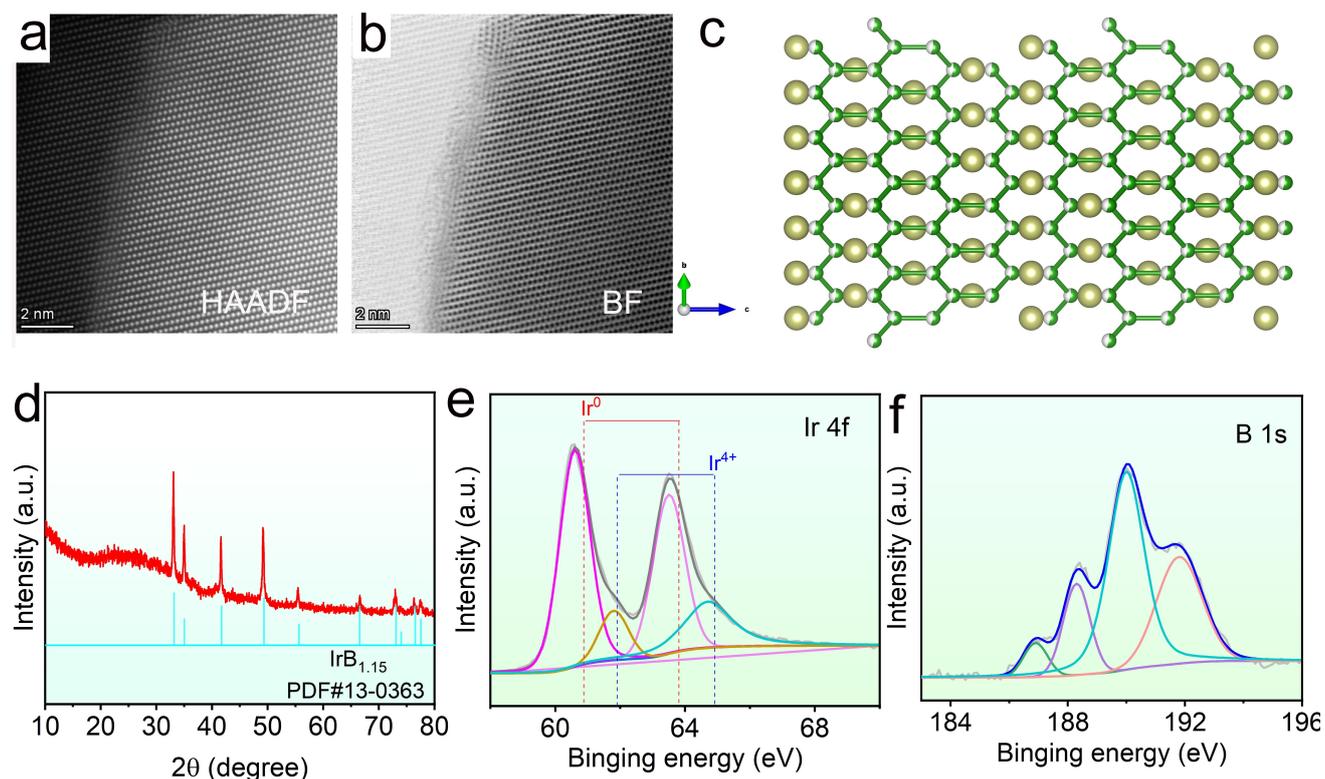


Figure 2. (a) HAADF-STEM and (b) BF and images of IrB_{1.15}. (c) Crystal structure viewed along the $[-110]$ direction for IrB_{1.15}. The yellow ball represents Ir, while blue and green ball represents B, respectively. (d) XRD pattern of IrB_{1.15}. (e) Ir 4f XPS spectrum of IrB_{1.15}. (f) B 1s XPS spectrum of IrB_{1.15}.

catalytic efficiency for CER, which surpasses that of the competing OER.

More importantly, the polarization curves before and after 1000, and 2000 CVs are illustrated in Figure 3g. After 2000 CV cycles, the polarization curve for IrB_{1.15} presents a slight degradation. For comparison, IrO₂ was also evaluated under identical conditions (Figure S9a). Notably, the LSV of IrO₂ exhibits a negative shift of approximately 16 mV after only 1000 CV cycles to achieve a current density of 10 mA cm⁻², indicating a substantial decline in its performance. Furthermore, the chronoamperometry curve of IrB_{1.15} (Figure 3h) and commercial IrO₂ (Figure S9b) demonstrates that IrB_{1.15} possesses accelerated degradation stability with minimal activity decay during extended operation over a period of 90 h. It is interesting to note that the current density of the IrB_{1.15} exhibits an initial increase followed by a subsequent decrease, which may be attributed to the electrochemically activation process of the IrB_{1.15}. As shown in Figure S10, the high-resolution Ir 4f and B 1s XPS spectra of the fresh sample and IrB_{1.15} after activation are similar, suggesting that the materials composition is preserved. However, a slight decrease in signal intensity at around 188.5 eV for the activated IrB_{1.15} catalyst compared to the fresh sample indicates possible oxidation of some B atoms during the CER activation process. Additionally, the slight decrease in current density can be attributed to the desorption of some particles from electrode surface. Due to

vigorous gas evolution, the stability of most powdered materials under CER conditions is typically low as effective strategies to securely immobilizing these materials on electrode surfaces remain limited.^[39] This good stability of IrB_{1.15} after CER testing is further confirmed by the TEM image, which reveals the preservation of its original morphology structure (Figure S11). Moreover, the XRD pattern obtained after the stability test reveals no evidence of any additional impurity phase, thereby confirming the exclusive presence of IrB_{1.15} (Figure S12). Thus, it is evident that IrB_{1.15} achieves a good balance between activity and stability for CER.

In general, the electrolysis of saline water under highly acidic conditions leads to the generation of Cl₂ at the anode, while simultaneously producing H₂ at the cathode. The evolved hydrogen gas was first confirmed using gas chromatography (GC-9790 Plus, Fuli) equipped with a thermal conductivity detector (TCD) (Figure S13). Therefore, we further evaluated the HER performance of IrB_{1.15} in 0.5 M H₂SO₄ solution. For comparison, commercially available 20 wt % Pt/C and IrO₂ were employed as reference materials. Before conducting the HER tests, the IrB_{1.15} catalyst was subjected to activation through multiple cycles of LSVs within the voltage window applied for HER. As displayed in Figure 4a, the IrB_{1.15}, IrO₂, and Pt/C materials demonstrate excellent HER performance. The overpotentials vs. RHE at a geometric current density of 10 mA cm⁻²

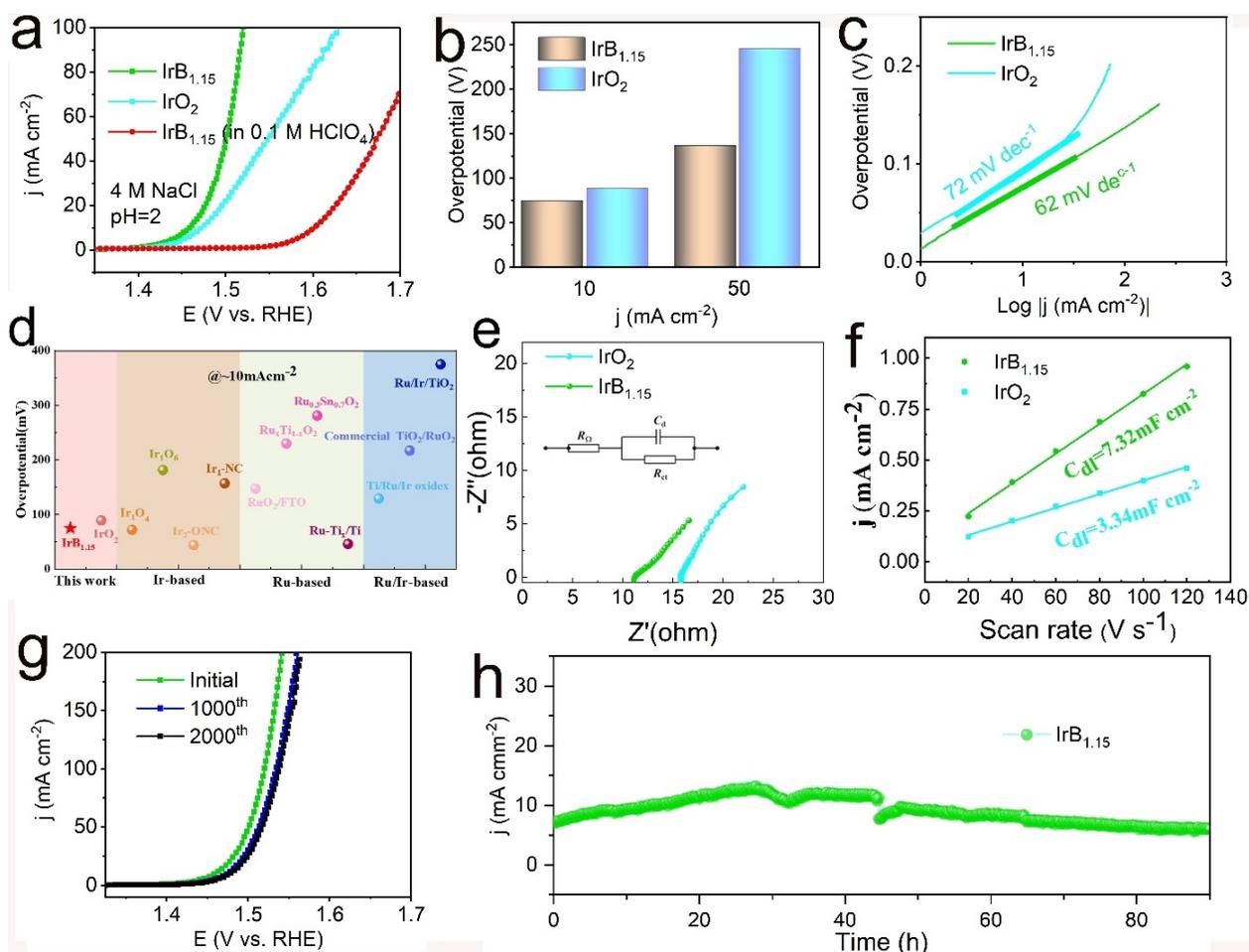


Figure 3. (a) CER polarization curves of IrB_{1.15} and IrO₂ in 4.0 M NaCl at pH = 2. (b) Corresponding overpotentials for IrB_{1.15} and IrO₂ at 10 and 50 mA cm⁻², respectively. (c) Tafel slopes for IrB_{1.15} and IrO₂ in 4.0 M NaCl at pH = 2. (d) Comparison of overpotential with different representative catalysts. (e) Nyquist plots for IrB_{1.15} and IrO₂ at an overpotential of 50 mV in the frequency range of 10⁵–0.01 Hz. (f) C_{dl} of IrB_{1.15} and IrO₂. (g) Polarization curves for IrB_{1.15} before and after 1000, 2000 CVs. (h) Chronoamperograms measurements of IrB_{1.15} at potential of 1.46 V vs. RHE (without iR correction).

for Pt/C, IrB_{1.15}, and IrO₂ are 22, 12, and 50 mV, respectively (Figure 4b). In other words, the catalytic activity of IrB_{1.15} in HER surpasses those of commercial Pt/C and IrO₂, indicating its superior performance in HER compared to the widely used catalysts. Additionally, the intrinsic catalytic activity of IrB_{1.15} is further demonstrated when normalizing the HER activity by the mass of Ir (Figure S14). The Tafel slope of Pt/C (27 mV dec⁻¹) is consistent with the reported value,^[40] indicating agreement with previous findings (Figure 4c). Moreover, IrB_{1.15} shows a slightly lower Tafel slope (25 mV dec⁻¹) compared to commercial IrO₂ (37.8 mV dec⁻¹). Notably, IrB_{1.15} exhibits a Faradaic efficiency (FE) exceeding 98% during the HER (Figures S15 and S16), implying that the observed current can primarily attributed to the electrocatalysis process of HER. To the best of our knowledge, IrB_{1.15} exhibits superior HER performance compared to most other noble-metal-based HER electrocatalysts in acidic aqueous solutions (Figure 4d and Table S2).

It is worth noting that we further investigated the HER performance of IrB_{1.15} under 4.0 M NaCl at pH = 2. As shown in Figure S17, compared to its performance in 0.5 M H₂SO₄ solution, IrB_{1.15} demonstrates moderate activity, with an overpotential of 68 mV at a geometric current density of 10 mA cm⁻² and a low Tafel slope of 59 mV dec⁻¹ in 4.0 M NaCl at pH 2 (Figures S17a and b). Furthermore, negligible degradation was observed in the LSV curves of IrB_{1.15} after 1000 CV potential cycles (Figure S17c). The chronopotentiometric test presented in Figure S17d further demonstrates the excellent HER stability of the IrB_{1.15} catalyst, as it maintains consistent performance over 40 h under 4.0 M NaCl at pH 2 with a current density of nearly 10 mA cm⁻². Furthermore, an electrolyzer was constructed in a two-electrode configuration, using IrB_{1.15} as both the anodic catalyst for CER and the cathodic catalyst for HER in 4.0 M NaCl at pH = 2 and room temperature. As depicted in Figure S18, the two-electrode potential of the IrB_{1.15} system achieves 10 mA cm⁻² at a cell voltage of only 1.51 V, demonstrating outstanding durability.

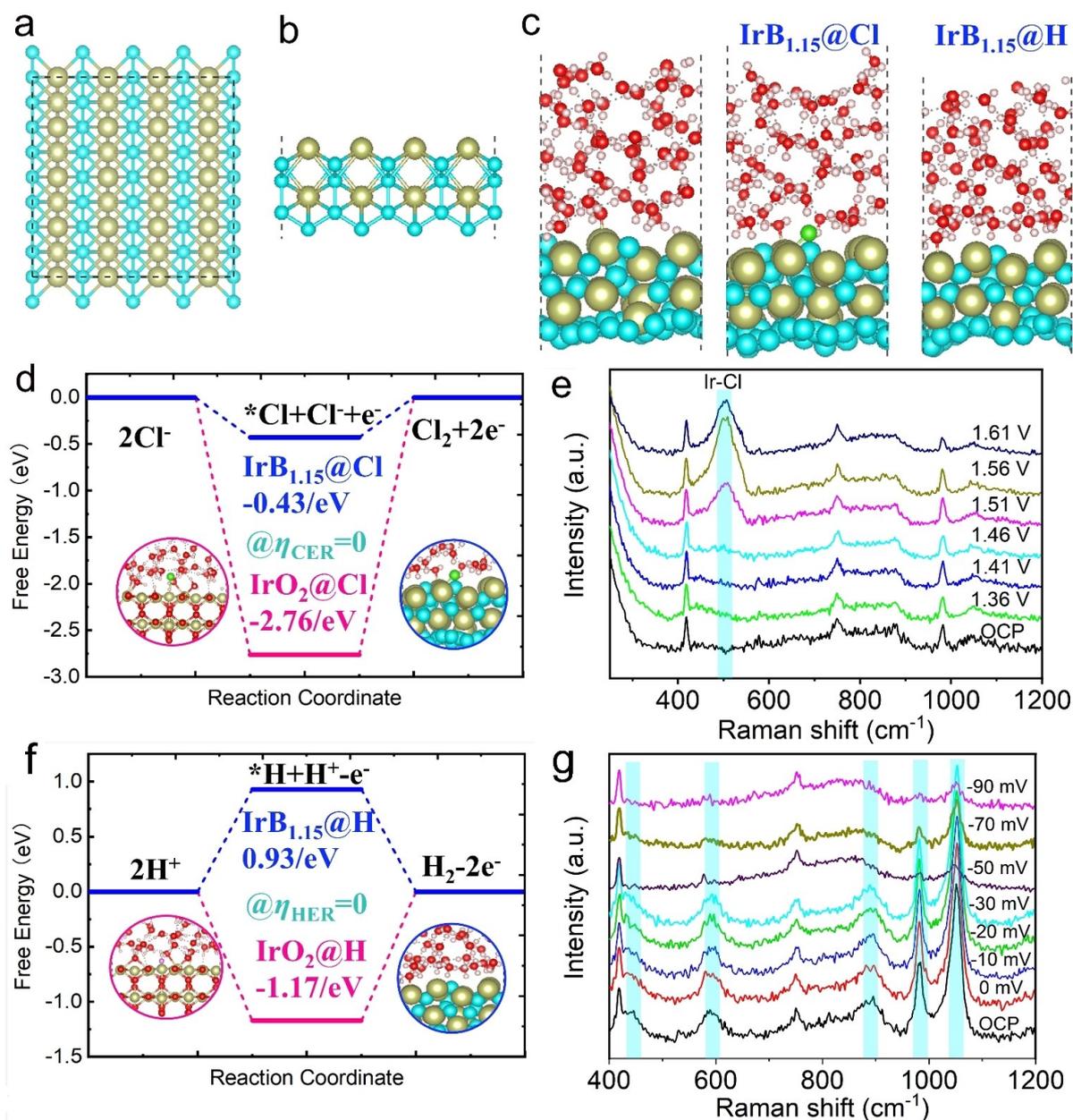


Figure 5. (a) Side-, and (b) top-down view of the IrB_{1.15}(110) surface. (c) DFT calculation models of the structures of bare IrB_{1.15} surface, IrB_{1.15}@Cl, IrB_{1.15}@H. (d, f) Free energy diagrams of the (d) CER and (f) HER process over IrB_{1.15} and IrO₂, respectively. (e and g) In situ Raman spectrum of IrB_{1.15} collected at (e) CER and (g) HER conditions.

of IrB_{1.15} during the HER process, thereby elucidating the catalytic active sites. The Raman peaks at approximately 432, 511, 882, 981, and 1052 cm⁻¹ observed in Figure 5g, corresponding to the H₂SO₄ electrolytes, exhibit a gradual decrease as the reduction potential increases. The observed phenomena may be attributed to the weakening of IrH_{ads} species with strong binding energy, resulting from the introduction of B atoms to Ir lattices. Consequently, an appropriate Gibbs free energy of hydrogen adsorption on the catalyst surface of IrB_{1.15} is achieved.^[17,43] It is worth noting that the Raman peaks at ~750 cm⁻¹ in Figures 5e and g can be attributed to solvent of water.^[44,45] Furthermore, the

Fermi level crosses the conduction band of IrO₂ as demonstrated by the projected density of states (PDOS) (Figure S21). The PDOS between commercial IrO₂ and IrB_{1.15} indicates enhanced electron mobility in the latter, which is highly significant for electrocatalytic CER or HER activity. As shown in Figures S21a and 21b, unlike the semiconducting behavior observed in IrO₂, IrB_{1.15} displays no band gap. Consequently, there is an evident electron transfer from B to Ir within the charge density of IrB_{1.15}, resulting in substantial enrichment of electrons. These investigations provide strong evidence that the coupling of B elements significantly influences the electronic structures of

Ir in intermetallic compounds of IrB_{1.15}, thereby enhancing electron mobility and electrocatalytic activity. In a word, the combined findings unequivocally validate the significant role of light B-induced interstitial effects in enhancing the catalytic performance of intermetallic IrB_{1.15} catalysts for both CER and HER. Additionally, this joule heating techniques is generally applied for the ultrafast synthesis of many other borides (e.g., RhB, Pd₂B) (Figure S22).

Conclusion

In summary, the intermetallic compounds of IrB_{1.15} have been successfully synthesized by Joule heating. The obtained IrB_{1.15} material exhibits outstanding catalytic activity and robust stability toward both CER and HER. It only needs overpotentials of 75 and 12 mV to produce an operationally relevant current density of 10 mA cm⁻² 4.0 M NaCl and 0.5 M H₂SO₄ solutions, respectively. These exceptional catalytic properties not only rival those of state-of-the-art commercial catalysts, such as IrO₂ and Pt/C but also exhibit superior long-term durability compared to these commercially available electrocatalysts. Mechanism studies showed that the presence of Ir in IrB_{1.15} serves as the active center for CER. Moreover, the introduction of B atoms to Ir lattices can attenuate the formation of adsorbed hydrogen species on the Ir surface, thereby enabling excellent performance in HER for IrB_{1.15} material. Furthermore, theoretical studies have confirmed that IrB_{1.15} demonstrates more favorable adsorption energies towards Cl and H, respectively, in comparison to IrO₂ and Pt/C. These findings serve as the primary factors contributing to the enhanced catalytic activity of IrB_{1.15} in both CER and HER. Undoubtedly, the catalytic activity exhibited by the intermetallic compounds of IrB_{1.15} paves a novel pathway for the design of metal boride materials, which were synthesized by Joule heating and exceptional performance toward CER, HER, and other related electrocatalytic applications.

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Conflict of Interest

The authors declare no competing financial interest.

Data Availability Statement

The data that support the findings of this study are available from the corresponding author upon reasonable request.

Keywords: intermetallic compounds · Joule heating · chlorine evolution reaction · hydrogen evolution reaction · saline water

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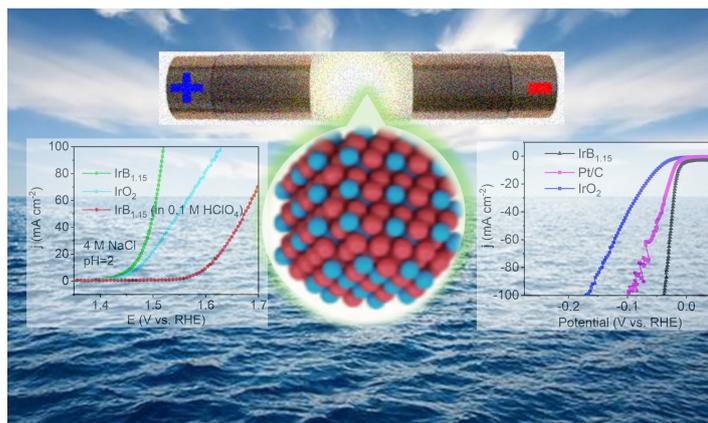
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Research Article

Saline Water Electrolysis

T. Liu, Z. Chen, S. Liu, P. Wang, Z. Pu,*
G. Zhang,* S. Sun* — e202414021

Ultrafast Synthesis of IrB_{1.15} Nanocrystals
for Efficient Chlorine and Hydrogen Evolution
Reactions in Saline Water



Intermetallic compounds of IrB_{1.15} have been successfully synthesized by ultrafast Joule heating at 900 °C for a duration of 12 seconds, showcasing their

robust electrocatalytic performance in acidic saline water media for both chlorine and hydrogen evolution reactions.