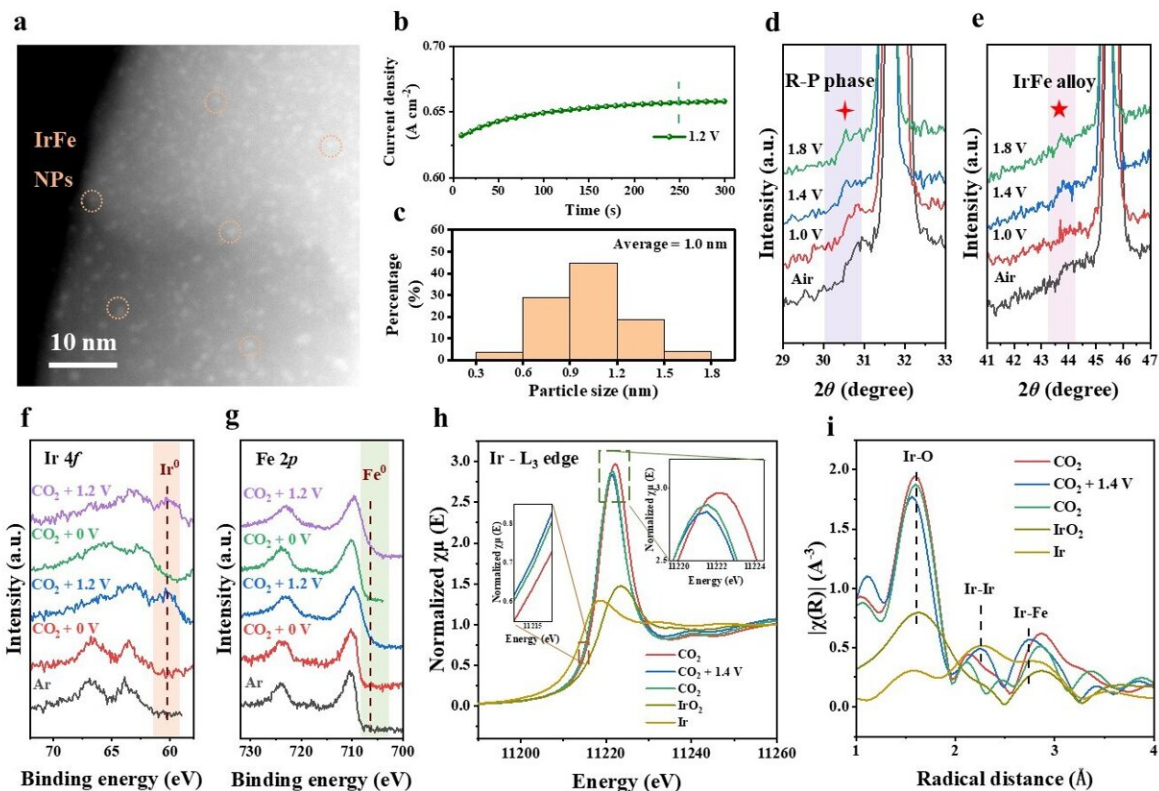


In situ investigation of the structure-activity correlation for carbon dioxide electrolysis in SOECs

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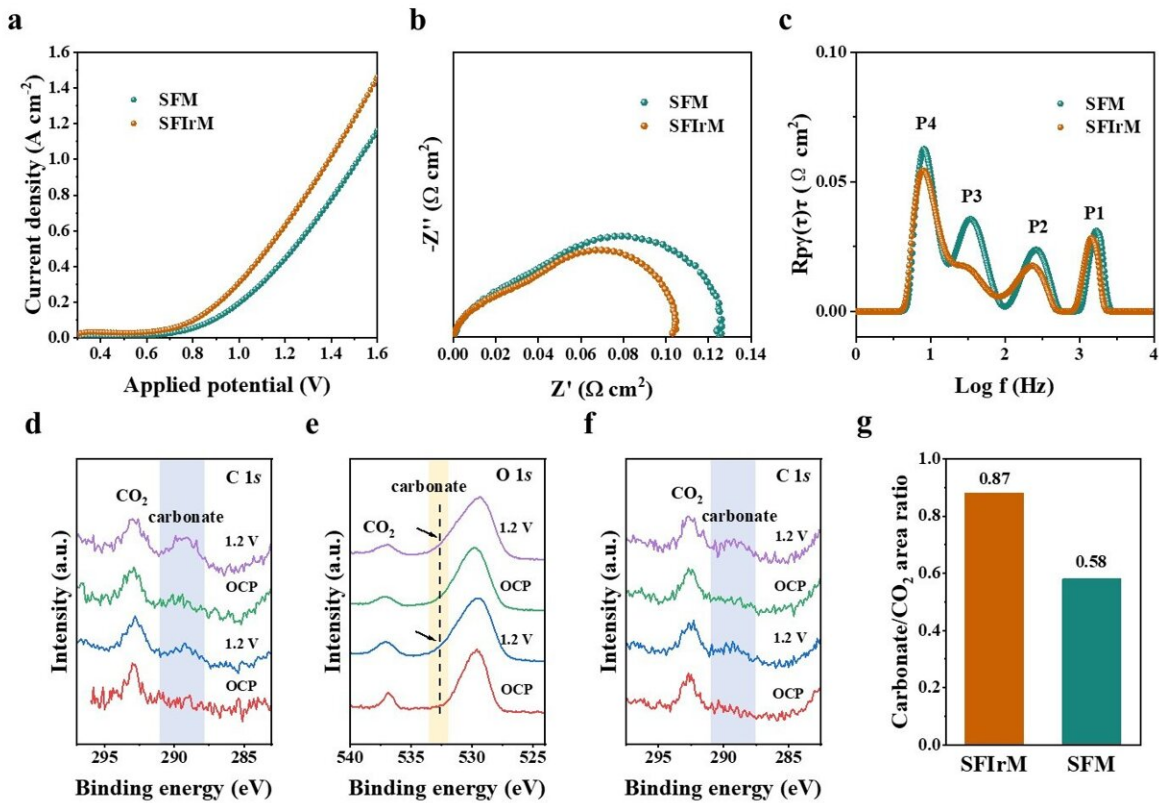
(a) Dark field-STEM image of the SFrM catalyst after (b) reconstruction at 1.2 V and (c) the corresponding diameter distribution of exsolved IrFe alloy NPs. (d-e) In situ XRD patterns at 29-33° and 41-47°. (f-g) In situ NAP-XPS spectra of Ir 4f and Fe 2p. (h-i) In situ Ir L₃-edge XANES spectra and the corresponding Fourier transformed EXAFS spectra. Credit: Science China Press

Solid oxide electrolysis cells (SOECs) provide a practical solution for direct conversion of CO₂ to chemicals; however, an in-depth mechanistic understanding of the dynamic reconstruction of active sites for perovskite cathodes during CO₂ electrolysis remains a great challenge.

In a study led by Dr. Houfu Lv, Prof. Guoxiong Wang and Prof. Xinhe Bao (Dalian Institute of Chemical Physics, Chinese Academy of Sciences), the team identify that iridium-doped Sr₂Fe_{1.45}Ir_{0.05}Mo_{0.5}O₆₋₈ (SFIrM) perovskite displays a dynamic electrochemical reconstruction feature during CO₂ [electrolysis](#) with abundant exsolution of highly dispersed IrFe alloy nanoparticles (NPs) on the SFIrM surface, which is well investigated using in situ electrochemical X-ray diffraction (XRD), near-ambient pressure X-ray photoelectron spectroscopy (NAP-XPS), and X-ray absorption spectroscopy (XAS).

During the electrochemical reconstruction and activation process under a constant voltage mode at 1.0 V for ~4 000 s, the [current density](#) gradually increased and finally approached a steady state.

The exsolved IrFe alloy NPs display high dispersion with an average diameter of ~1.0 nm and a density above 80 000 μm⁻² on the SFIrM surface. Moreover, the activation time was reduced rapidly with increasing [applied voltage](#) (constant voltage: 1.2 V, activation time: within 250 s), with similar particle size and density of the exsolved metal NPs during CO₂ electrolysis.



(a) LSV curves of the electrolysis cells with SFM and SFIrM cathodes. (b) EIS plots and (c) corresponding DRT analysis at 1.4 V. (d-e) In situ NAP-XPS spectra of C 1s and O 1s of the SFIrM cathode, and (f) in situ NAP-XPS spectra of C 1s of the SFM cathode. (g) The area ratio of carbonate/CO₂ deconvoluted from (d) and (f), respectively. Credit: Science China Press

In addition, in situ NAP-XPS measurements were employed to monitor their catalytic process during CO₂ electrolysis to reveal the intrinsic reaction mechanism. Upon application of electrochemical polarization, a broader peak appeared at ca. 290 eV in the C 1s spectra, which could be attributed to carbonate species and was most probably decisive for CO₂ electrolysis.

The peak area of carbonate species on the Sr₂Fe_{1.5}Mo_{0.5}O_{6-δ} (SFM)

cathode was weaker than that on the SFIrM cathode. The enhanced carbonate intermediate species signal confirmed that the in situ reconstructed IrFe@SFIrM interfaces facilitated CO₂ adsorption and activation. IrFe@SFIrM interfaces were thus proposed as the catalytically active sites that devoted to a higher CO₂ electrolysis performance than SFM.

The initial exsolved IrFe alloy NPs could be re-dispersed sufficiently into smaller nanoclusters via a brief oxidation treatment in air. IrFe alloy NPs were regenerated when the applied voltage was switched on again, and the appearance of the intermediate carbonate peak could be further obtained. The oxidative re-dispersion strategy could efficiently improve the stability by delaying particle aggregation.

The research is published in the journal *National Science Review*.

More information: Yuxiang Shen et al, In situ electrochemical reconstruction of Sr₂Fe_{1.45}Ir_{0.05}Mo_{0.5}O_{6-δ} perovskite cathode for CO₂ electrolysis in solid oxide electrolysis cells, *National Science Review* (2023). [DOI: 10.1093/nsr/nwad078](https://doi.org/10.1093/nsr/nwad078)

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