

## Supplementary Information

# PRED treatment mediated stable and efficient water oxidation performance of Fe<sub>2</sub>O<sub>3</sub> nano-coral structure

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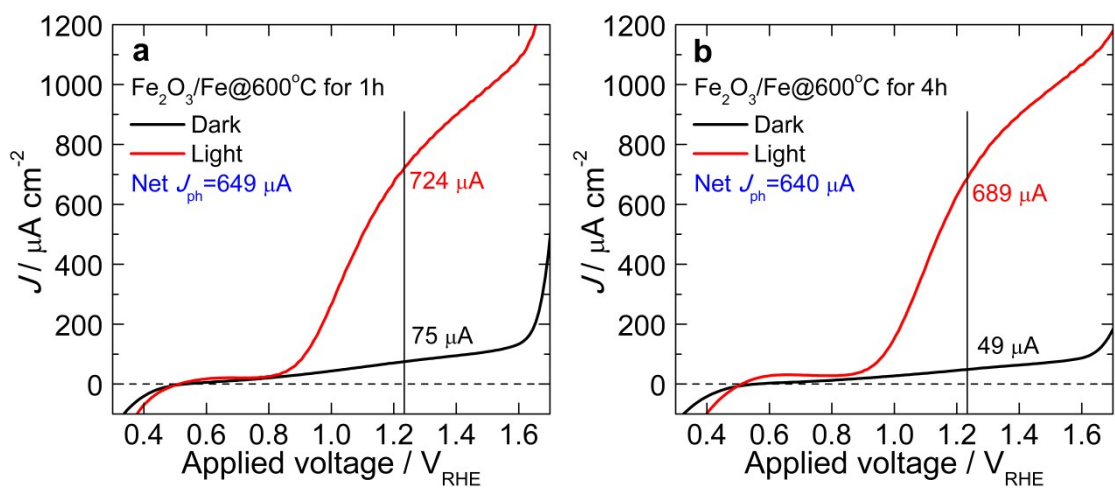
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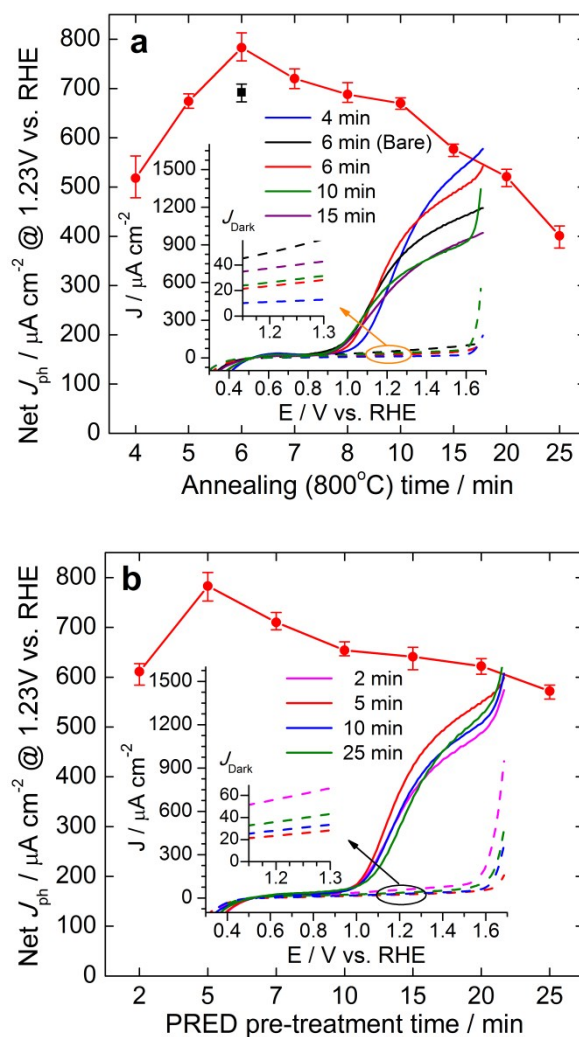
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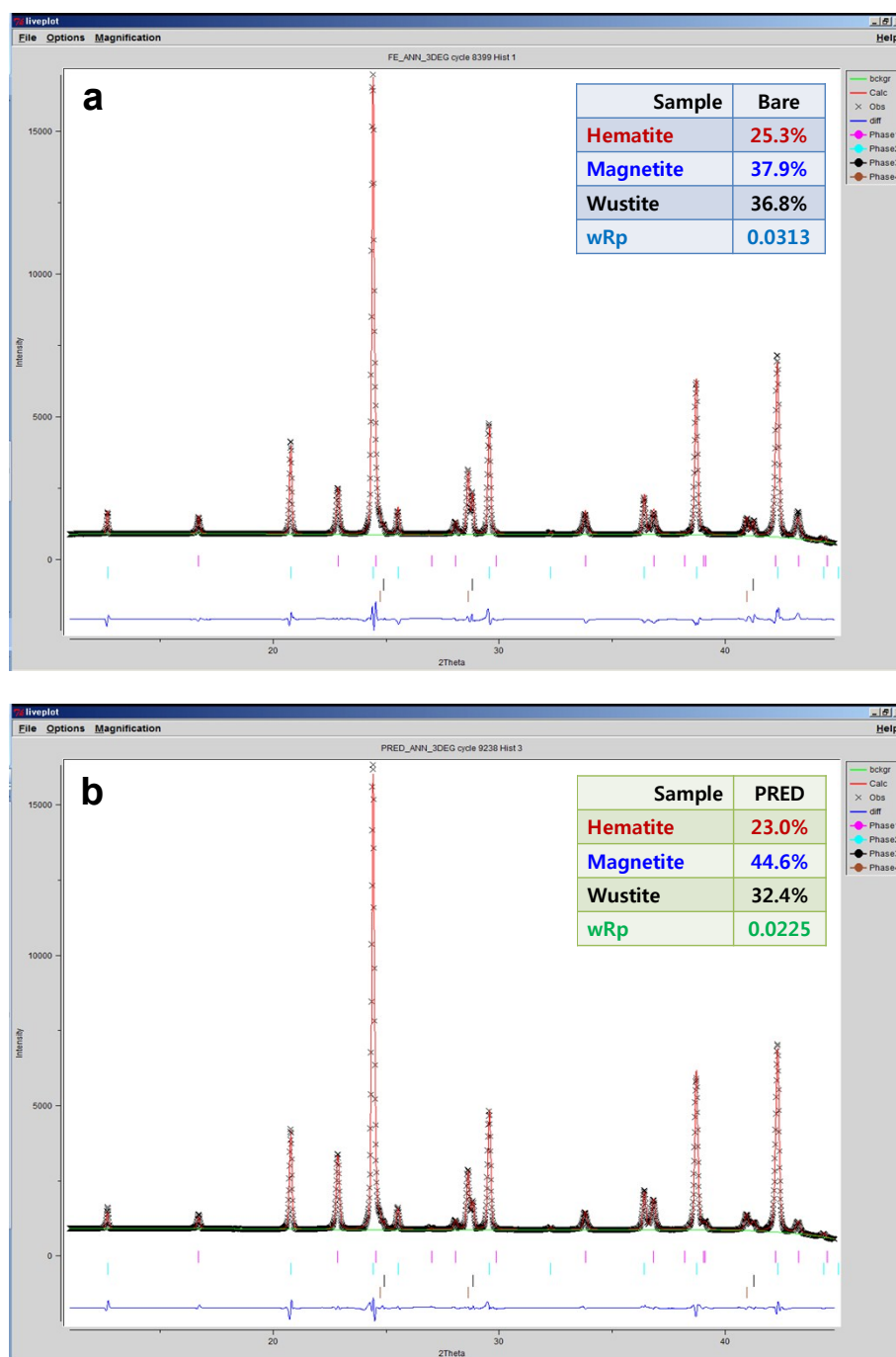
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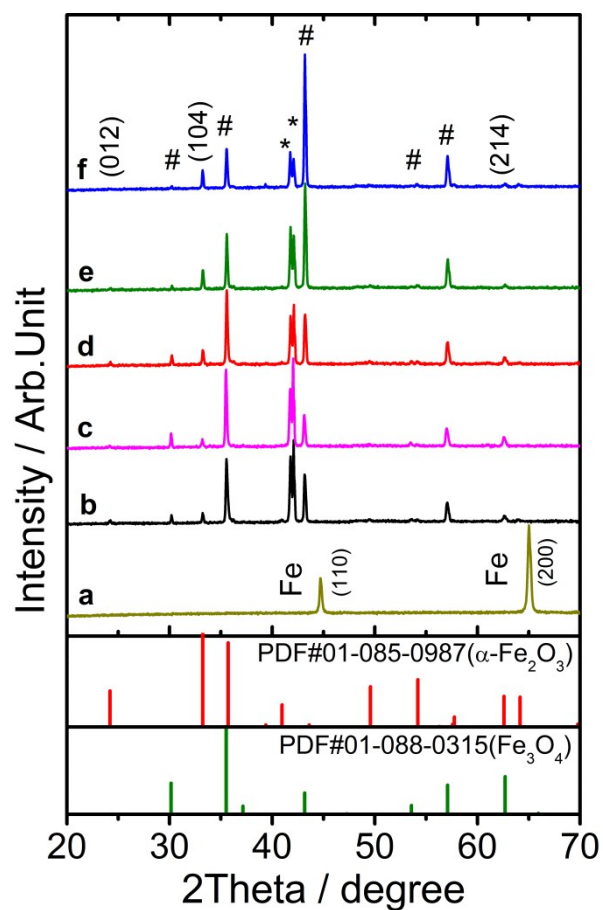
**Figure S1.** Photocurrent density-voltage curves of  $\text{Fe}_2\text{O}_3/\text{Fe}$  electrodes fabricated by conventional annealing (heating@ $5^\circ\text{C}$  and cooling naturally) of 5 min PRED-treated Fe foil at low temperature  $600^\circ\text{C}$  for (a) 1 h and (b) 4 h recorded under dark and light (1 sun) at the scan rate of  $50 \text{ mV s}^{-1}$ .



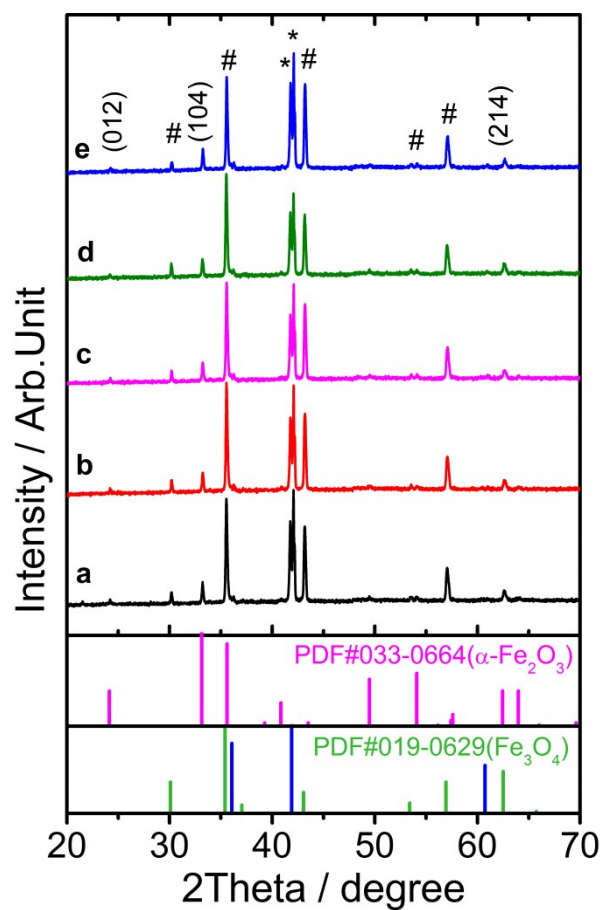
**Figure S2.** Photocurrent density ( $J_{ph}$ ) measured at 1.23 V<sub>RHE</sub> of Fe<sub>2</sub>O<sub>3</sub>/Fe electrodes fabricated (a) with 5 min PRED pre-treatment time and annealed at different annealing times and a bare Fe<sub>2</sub>O<sub>3</sub>/Fe electrode annealed at 800 °C for 6 min; (b) with different PRED pre-treatment times annealed at 800 °C for 6 min. Inset shows the  $J$ – $V$  curves of representative PRED-treated  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>/Fe electrodes with different PRED pre-treatment times. Light illumination, 1 sun; scan rate, 50 mV s<sup>–1</sup>.



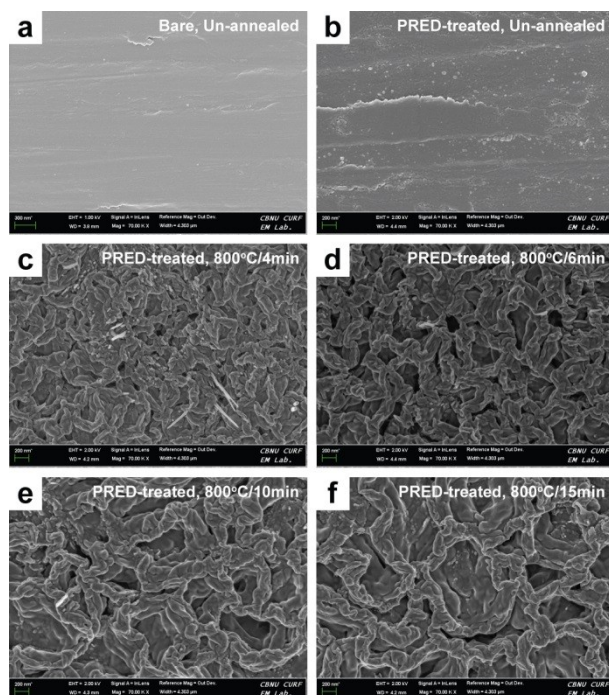
**Figure S3.** Rietveld analysis (GSAS program) from synchrotron X-ray diffraction patterns of (a) bare and (b) PRED-treated iron oxide/Fe electrodes prepared by annealing at 800 °C for 6 min. Insets show the phase fractions of different phases of iron oxide.



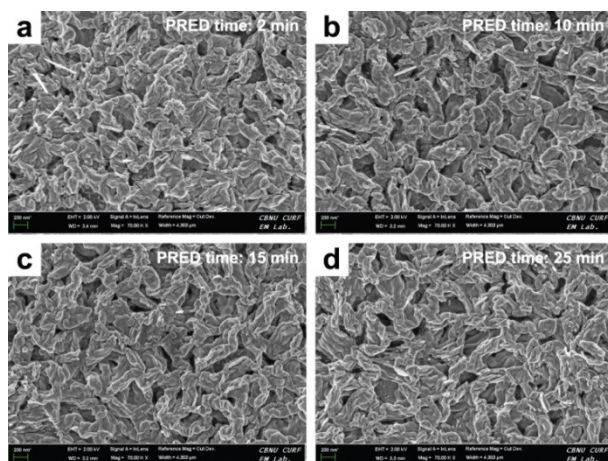
**Figure S4.** XRD patterns of (a) un-annealed PRED-treated Fe foil, (b) bare Fe foil annealed at 800 °C for 6 min, and PRED-treated Fe foils annealed at 800 °C for different annealing times: (c) 4 min; (d) 6 min; (e) 10 min; (f) 15 min. The symbols \* and # represent the Wustite (FeO) and magnetite (Fe<sub>3</sub>O<sub>4</sub>) phases of iron oxide.



**Figure S5.** XRD patterns of PRED-treated electrodes for different PRED times: (a) 2 min, (b) 5 min, (c) 10 min, (d) 15 min, and (e) 25 min, all annealed at 800 °C for 6 min. The symbols \* and # represent the Wustite (FeO) and magnetite (Fe<sub>3</sub>O<sub>4</sub>) phases of iron oxide.

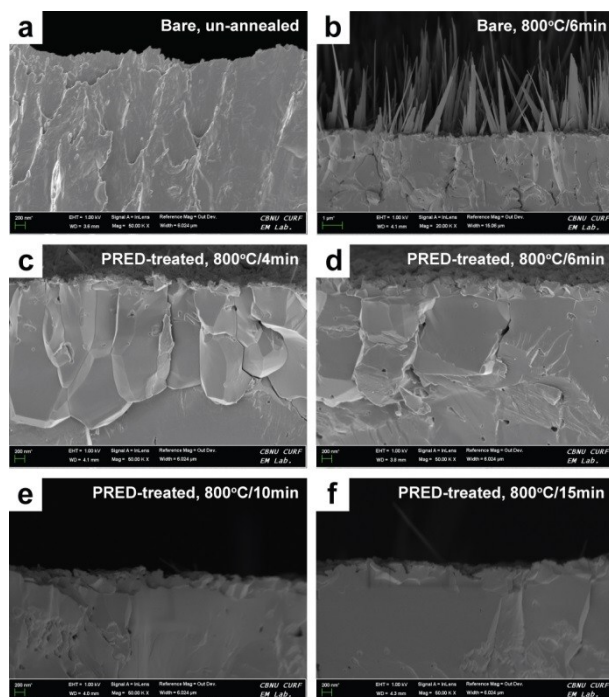


**Figure S6.** FESEM surface images of bare: (a) un-annealed Fe foil, PRED-treated  $\text{Fe}_2\text{O}_3/\text{Fe}$  electrodes for different PRED times: (a) 2 min, (b) 10 min, (c) 15 min, and (d) 25 min, all annealed at 800 °C for 6 min.

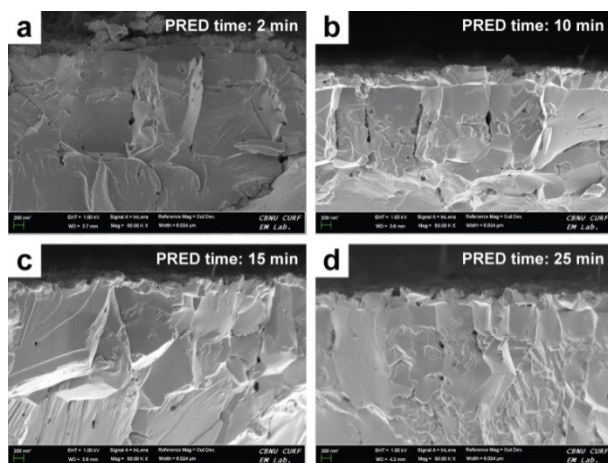


**Figure S7.** FESEM surface images of PRED-treated  $\text{Fe}_2\text{O}_3/\text{Fe}$  electrodes for different PRED times: (a) 2 min, (b) 10 min, (c) 15 min, and (d) 25 min, all annealed at 800 °C for 6 min.

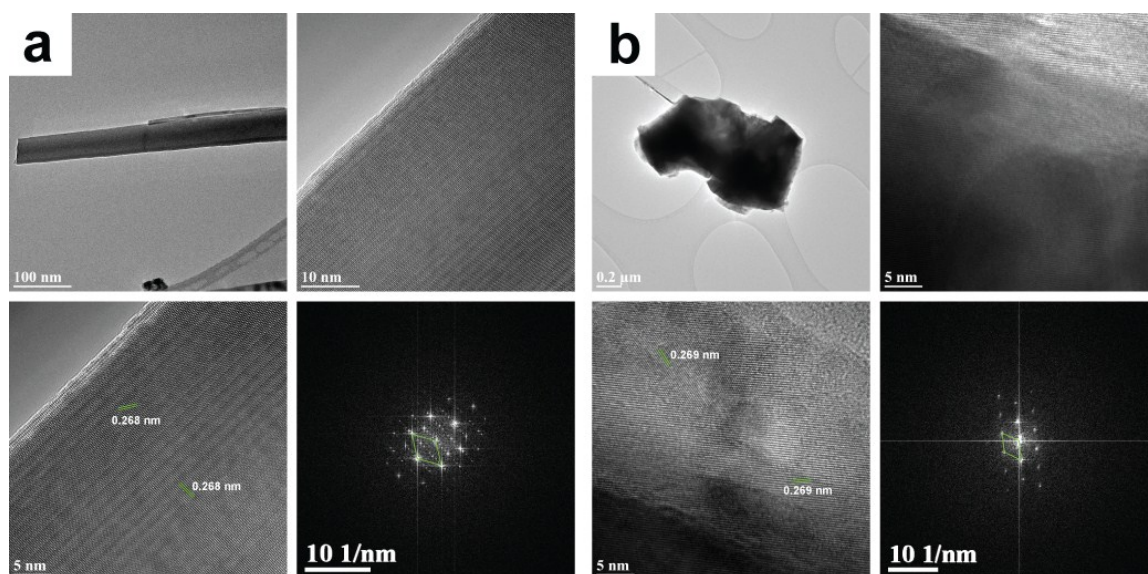




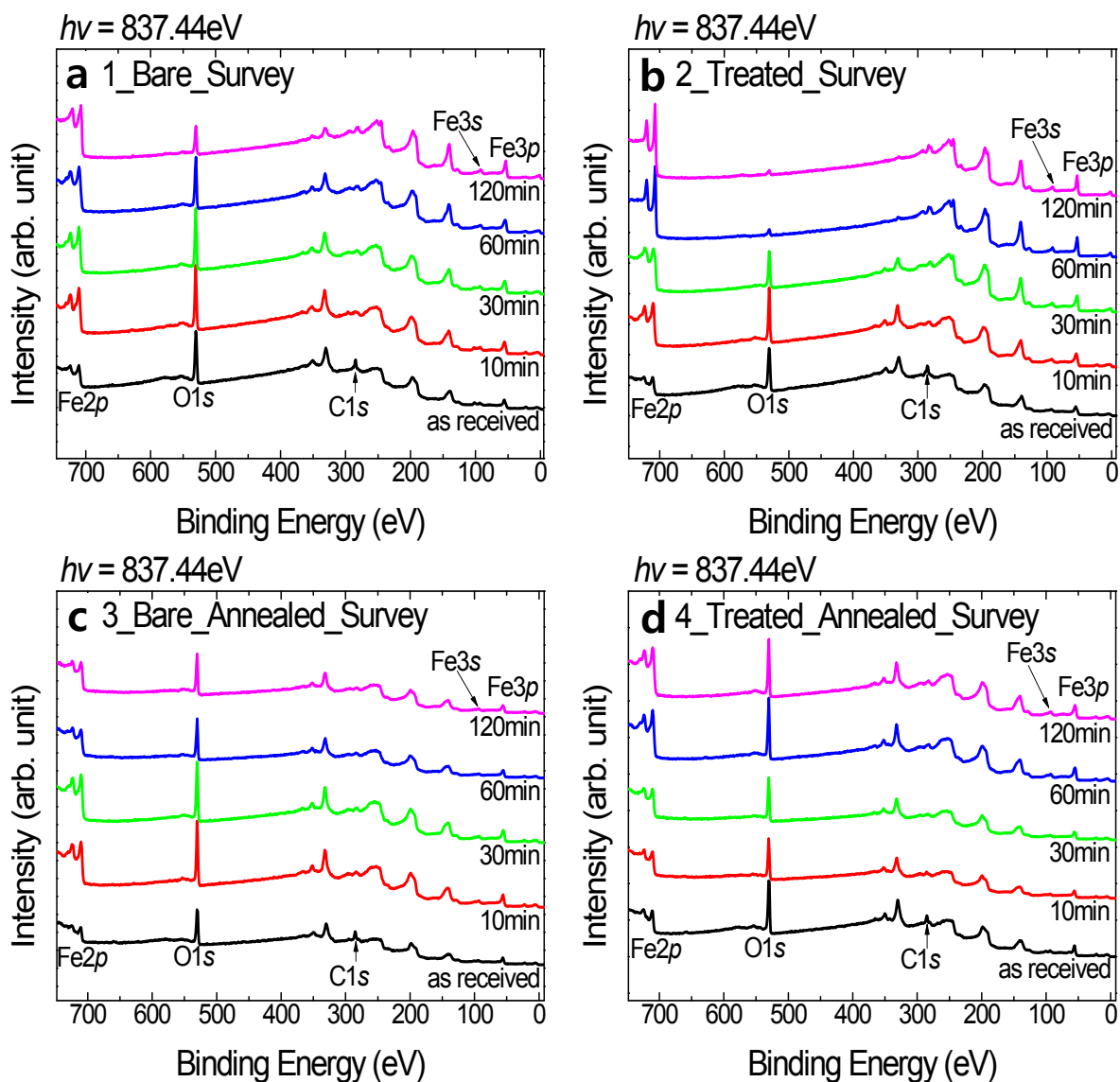
**Figure S8.** Cross-sectional FESEM images of  $\text{Fe}_2\text{O}_3/\text{Fe}$  electrodes, bare: (a) un-annealed Fe foil, (b) 6 min annealed, and PRED-treated: annealed at 800 °C for (c) 4 min, (e) 6 min, (f) 10 min, and (g) 15 min.



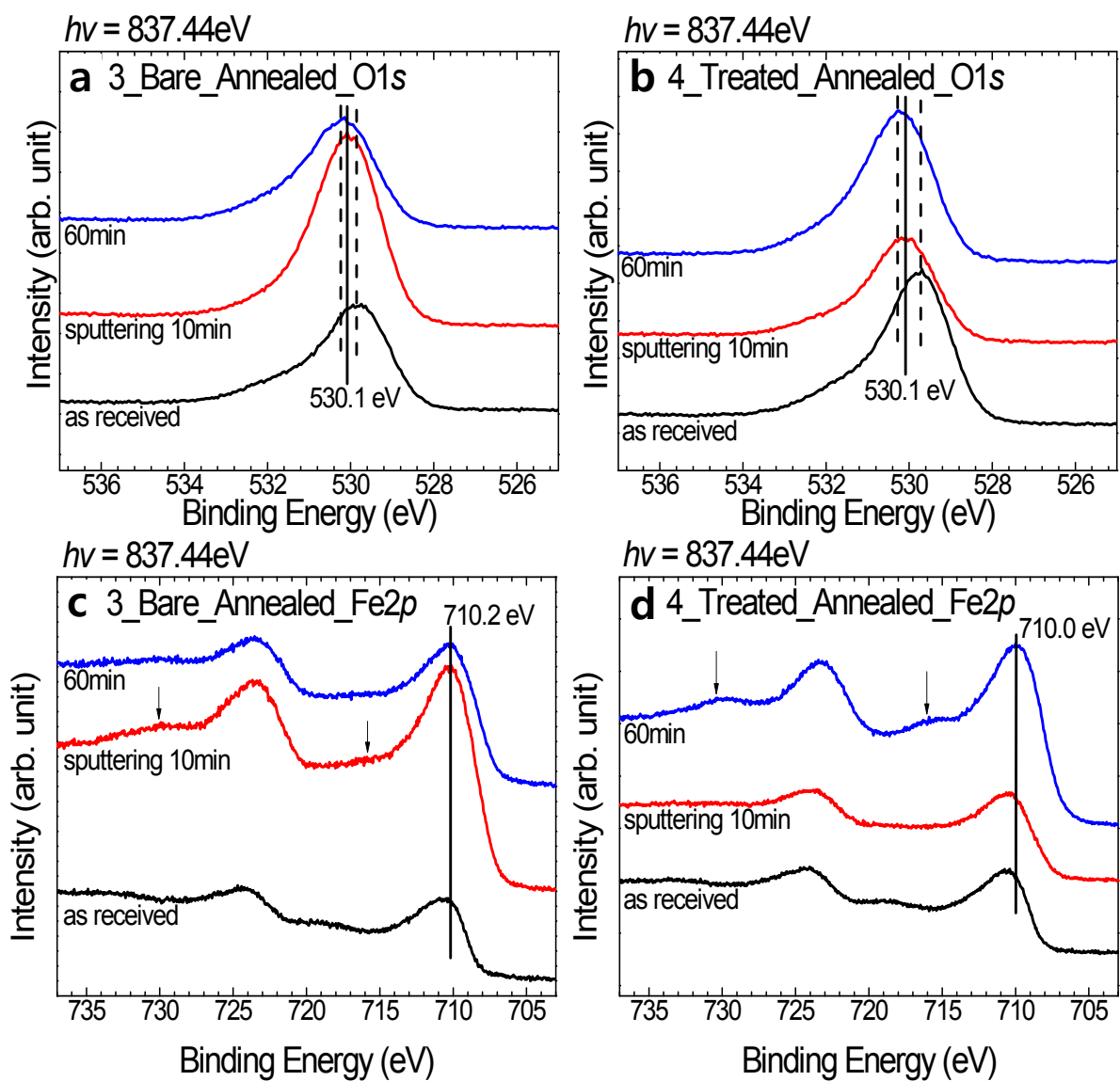
**Figure S9.** Cross-sectional FESEM images of PRED-treated  $\text{Fe}_2\text{O}_3/\text{Fe}$  electrodes for different PRED times: (a) 2 min, (b) 10 min, (c) 15 min and (d) 25 min, all annealed at 800 °C for 6 min.



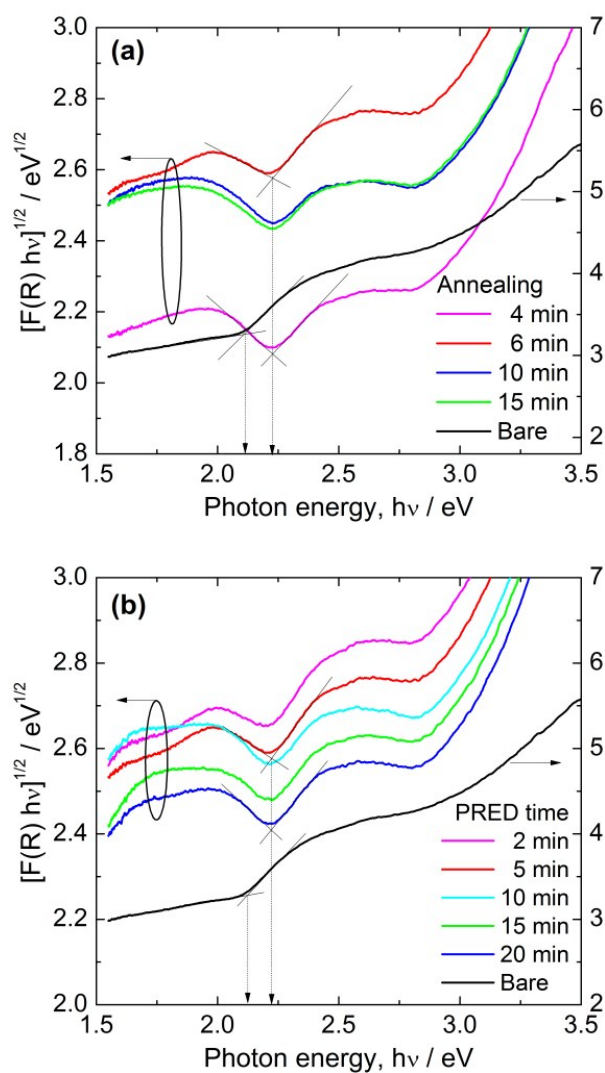
**Figure S10.** Low and high magnification HR-TEM images and corresponding FFTs of (a) bare and (b) PRED-treated iron oxide samples annealed at 800 °C for 6 min.



**Figure S11.** XPS survey spectra recorded during depth profiling of different samples (a) bare and (b) 5 min PRED-treated Fe foil, and (c) bare and (d) PRED-treated iron oxide samples annealed at 800 °C for 6 min. Sputtering depth time was varied as 0, 10, 30, 60 and 120 min.



**Figure S12.** High resolution XPS spectra of (a-b) O1s and (c-d) Fe2p regions during depth profile analysis of bare and 5 min PRED-treated Fe foil samples annealed at 800 °C for 6 min.



**Figure S13.** Kubelka–Munk plots obtained from the diffused reflectance (UV-vis) study of (a) untreated and PRED-treated  $\text{Fe}_2\text{O}_3/\text{Fe}$  electrodes annealed at 800 °C for different annealing times (b) untreated and PRED-treated  $\text{Fe}_2\text{O}_3/\text{Fe}$  electrodes annealed at 800 °C for 6 min with different PRED treatment times. The Kubelka–Munk plots revealed indirect band gap ( $E_g$ ) energies in the range of 2.11–2.23 eV.

## References

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