

Octahedral and Tetrahedral Site Substitution in Spinel Mn_3O_4 Regulates Oxygen Evolution in Acid

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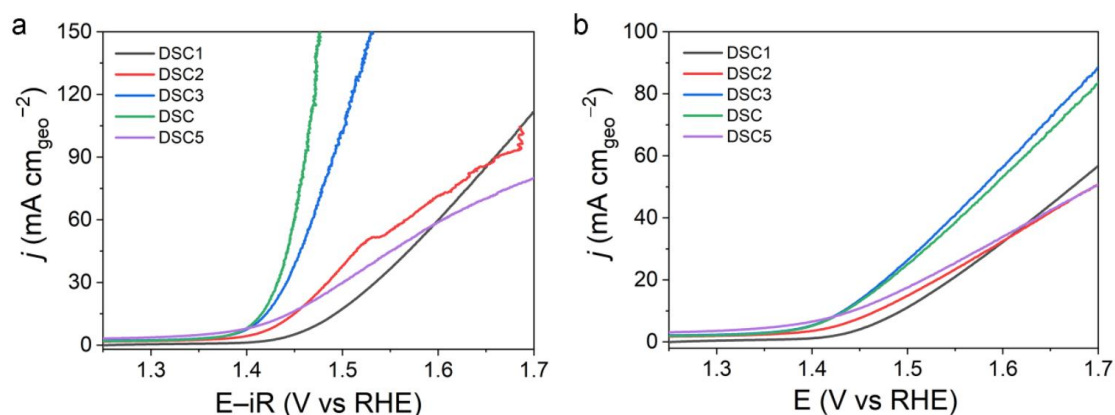


Figure S1. (a) iR-corrected polarization curves of DSC1, DSC2, DSC3, DSC, and DSC5. (b) Corresponding non-iR-corrected polarization curves. The uncompensated resistance values used for iR correction were 1.1, 2.0, 1.4, 1.7, and 1.5 Ω for DSC1, DSC2, DSC3, DSC, and DSC5, respectively. All measurements were performed using a geometric electrode area of 2 cm², and the current densities were normalized to this area.

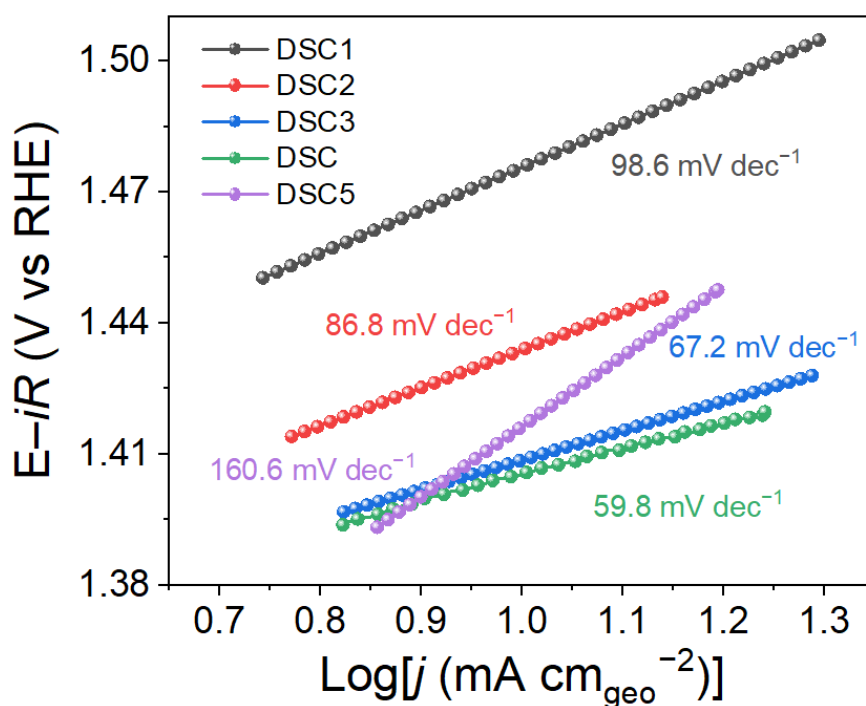


Figure S2. Tafel plots based on the iR-corrected polarization curves of DSC1, DSC2, DSC3, DSC, and DSC5.

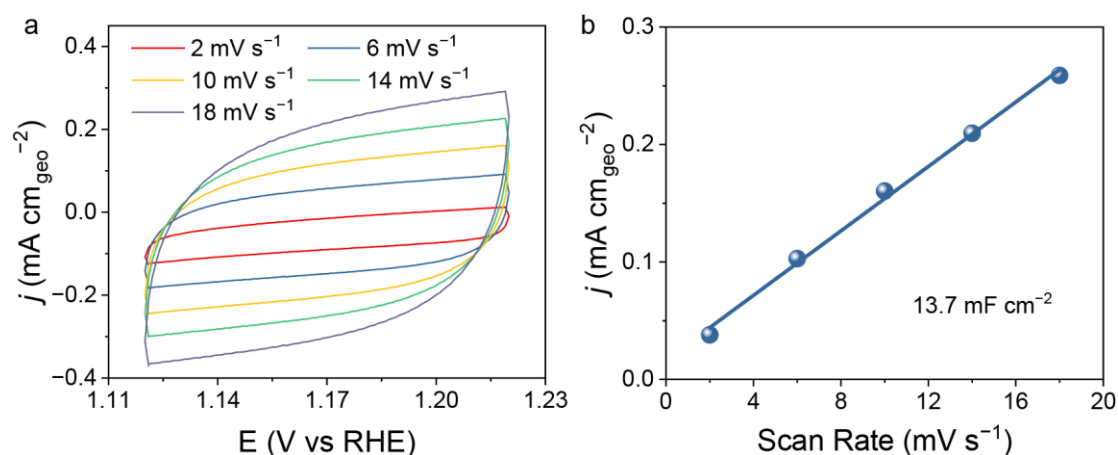


Figure S3. (a) CV curves at non-Faraday potential ranges of the DSC1 with different scan rates. (b) Calculated specific C_{dl} plot of the DSC1 derived from CV curves.

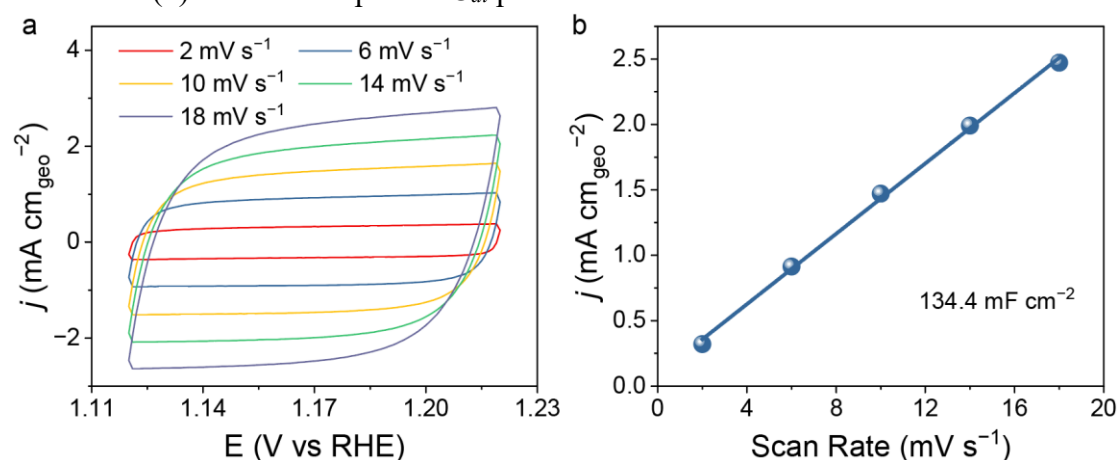


Figure S4. (a) CV curves at non-Faraday potential ranges of the DSC2 with different scan rates. (b) Calculated specific C_{dl} plot of the DSC2 derived from CV curves.

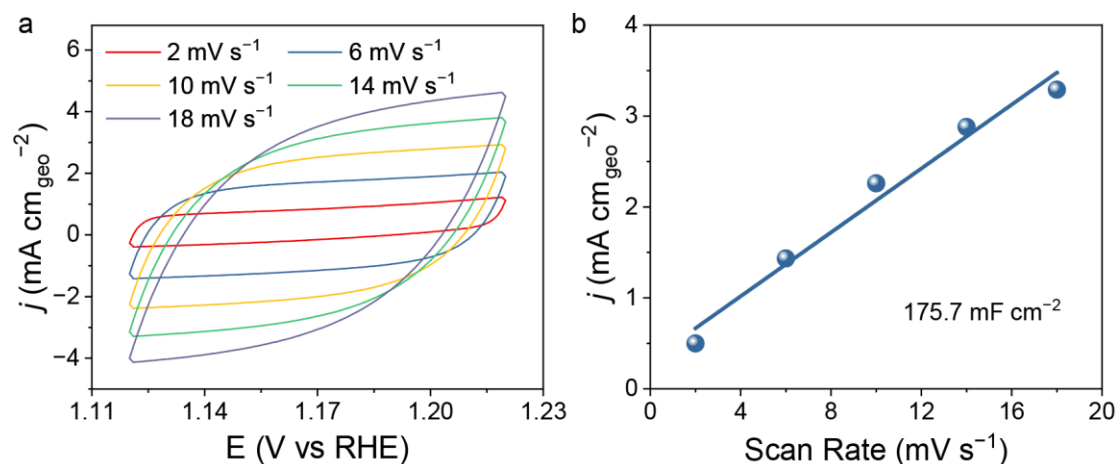


Figure S5. (a) CV curves at non-Faraday potential ranges of the DSC3 with different scan rates. (b) Calculated specific C_{dl} plot of the DSC3 derived from CV curves.

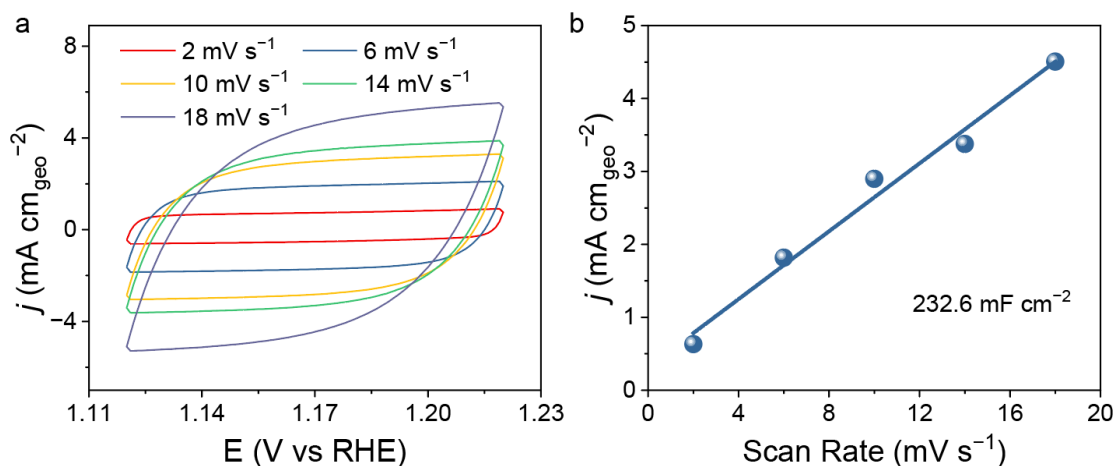


Figure S6. (a) CV curves at non-Faraday potential ranges of the DSC with different scan rates. (b) Calculated specific C_{dl} plot of the DSC derived from CV curves.

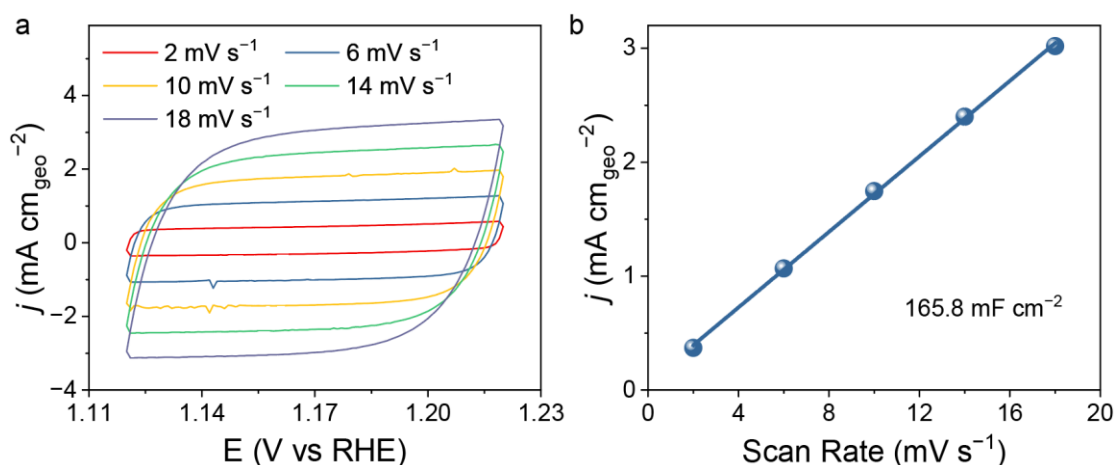


Figure S7. (a) CV curves at non-Faraday potential ranges of the DSC5 with different scan rates. (b) Calculated specific C_{dl} plot of the DSC5 derived from CV curves.

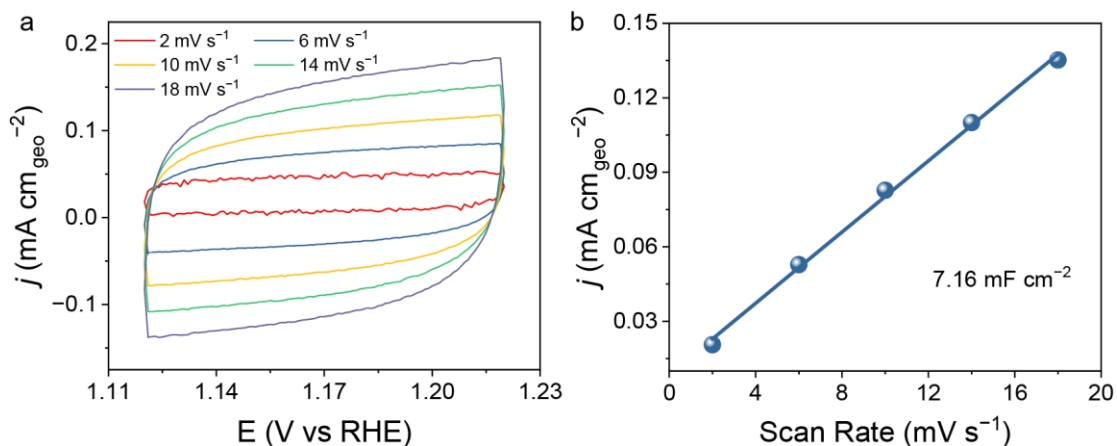


Figure S8. (a) CV curves at non-Faraday potential ranges of the commercial RuO_2 with different scan rates. (b) Calculated specific C_{dl} plot of the commercial RuO_2 derived from CV curves.

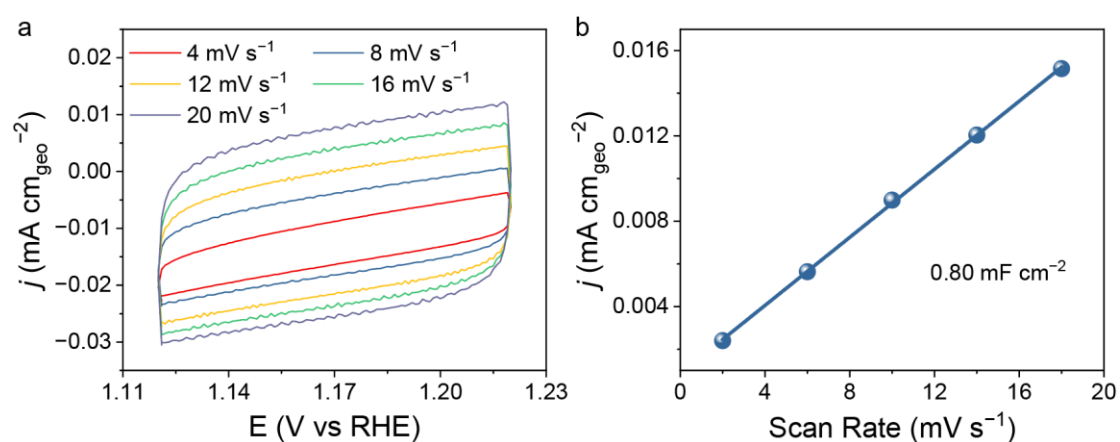


Figure S9. (a) CV curves at non-Faraday potential ranges of the SSC with different scan rates. (b) Calculated specific C_{dl} plot of the SSC derived from CV curves.

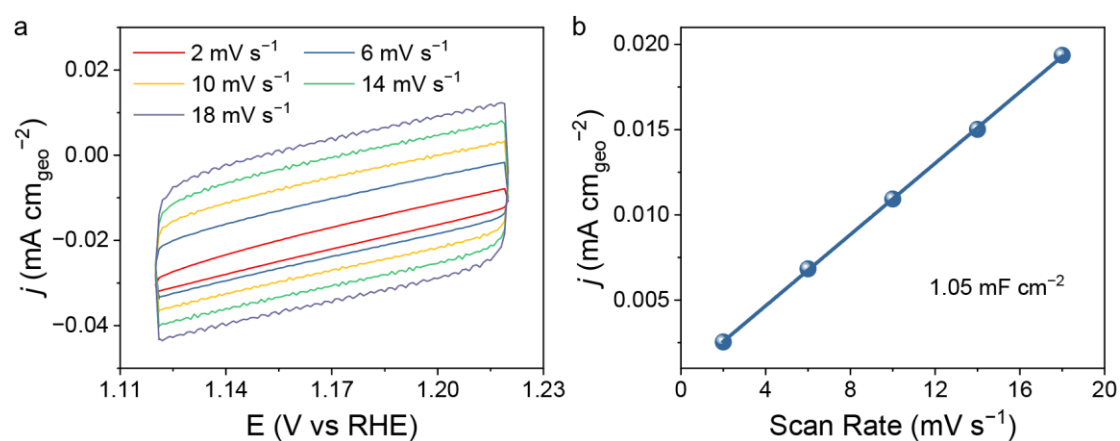


Figure S10. (a) CV curves at non-Faraday potential ranges of the Mn₃O₄ with different scan rates. (b) Calculated specific C_{dl} plot of the commercial Mn₃O₄ derived from CV curves.

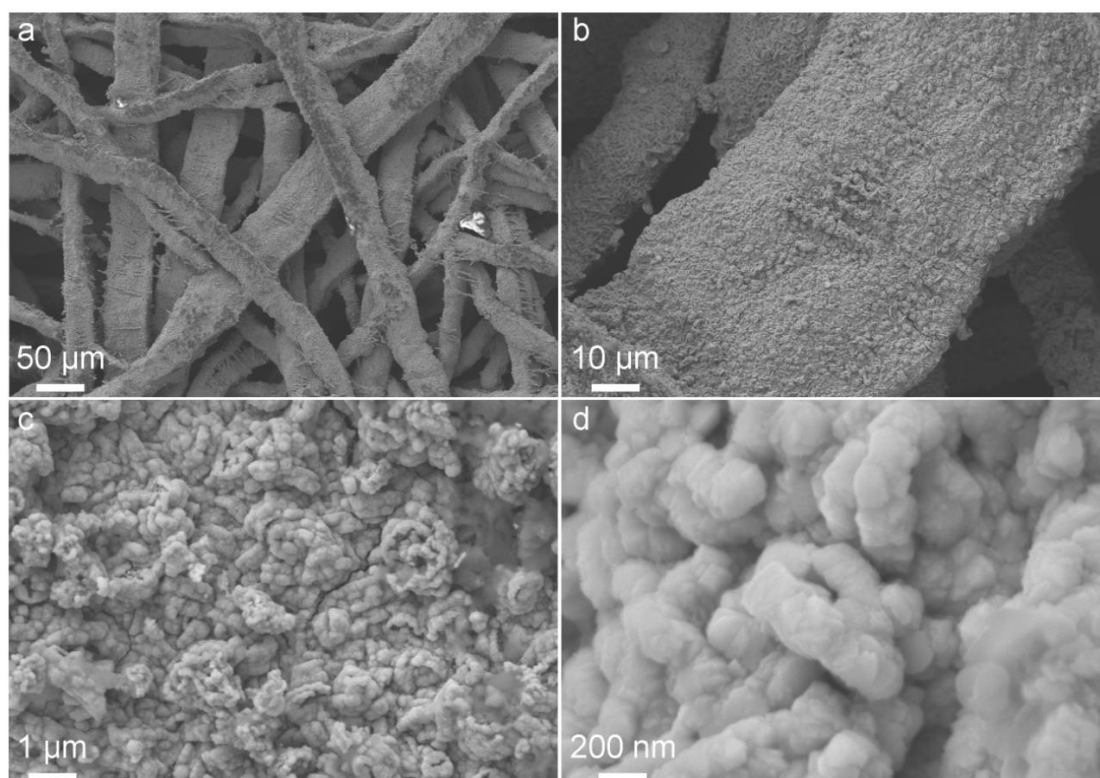


Figure S11. Low and high-resolution SEM images of the Mn_3O_4 .

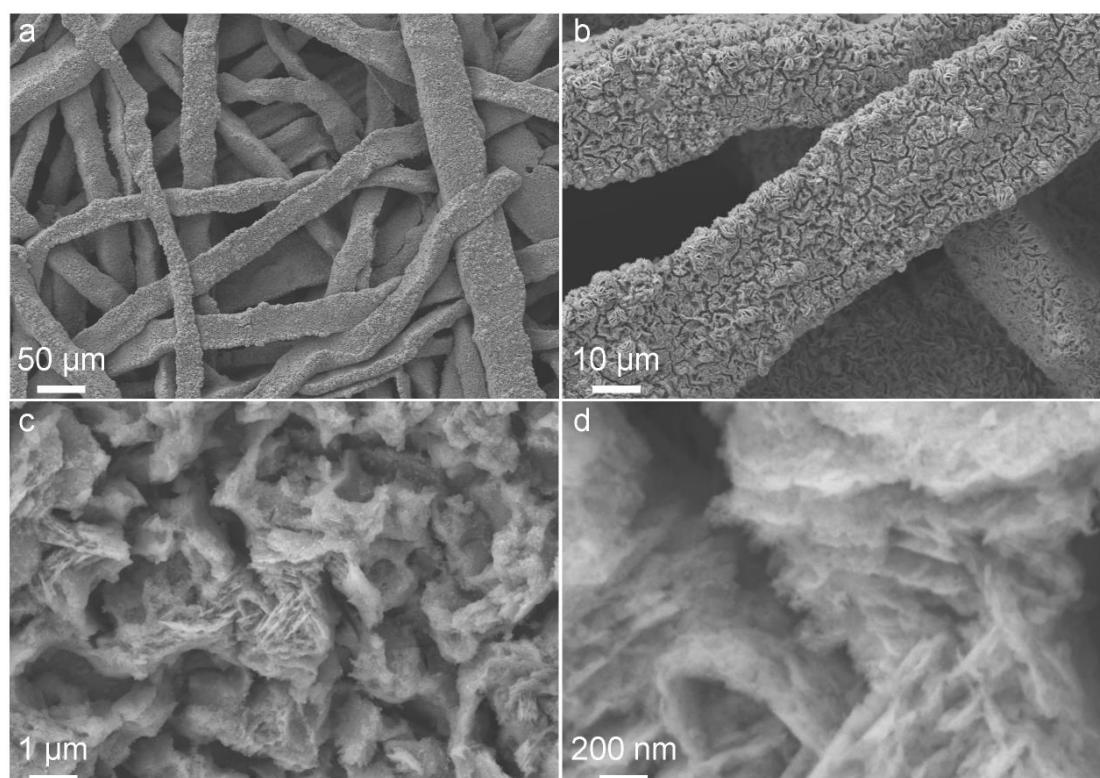


Figure S12. Low and high-resolution SEM images of the SSC.

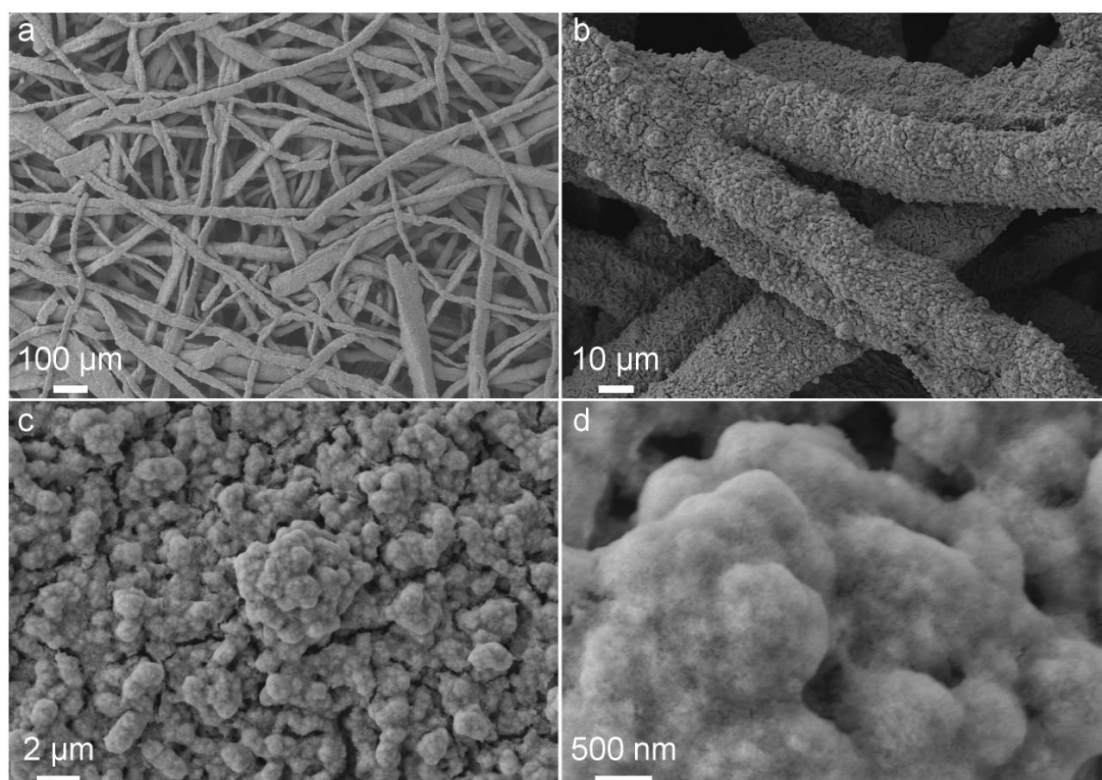


Figure S13. Low and high-resolution SEM images of the DSC1.

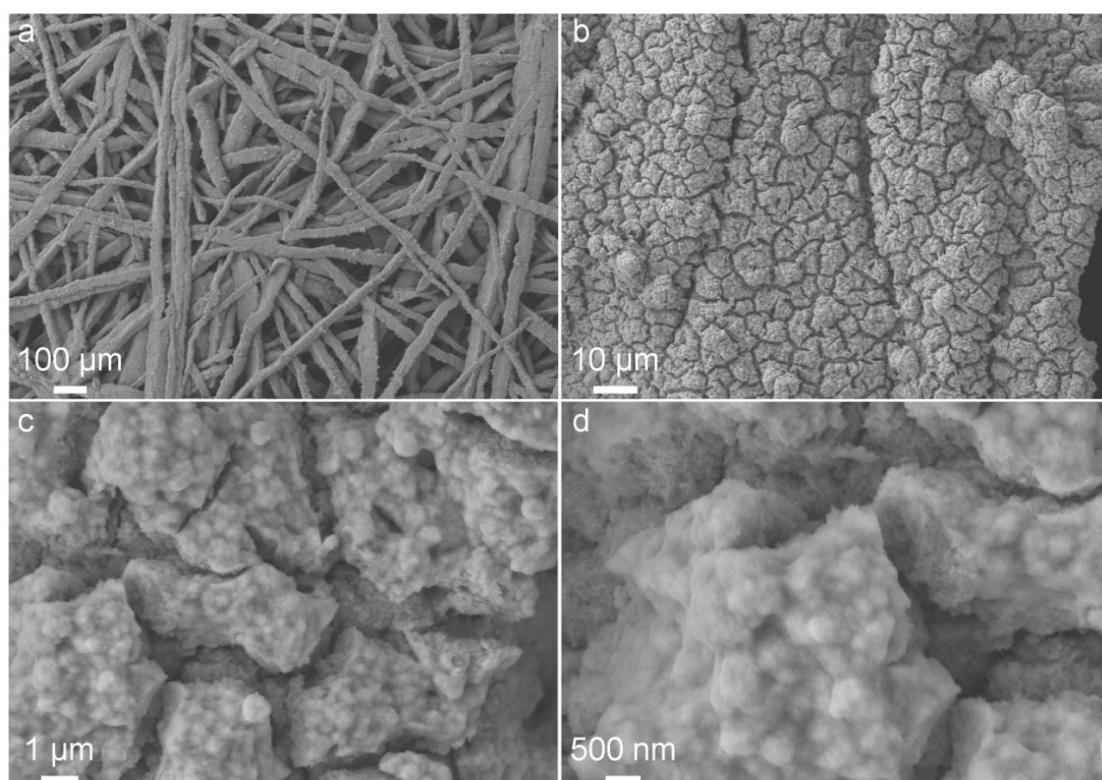


Figure S14. Low and high-resolution SEM images of the DSC2.

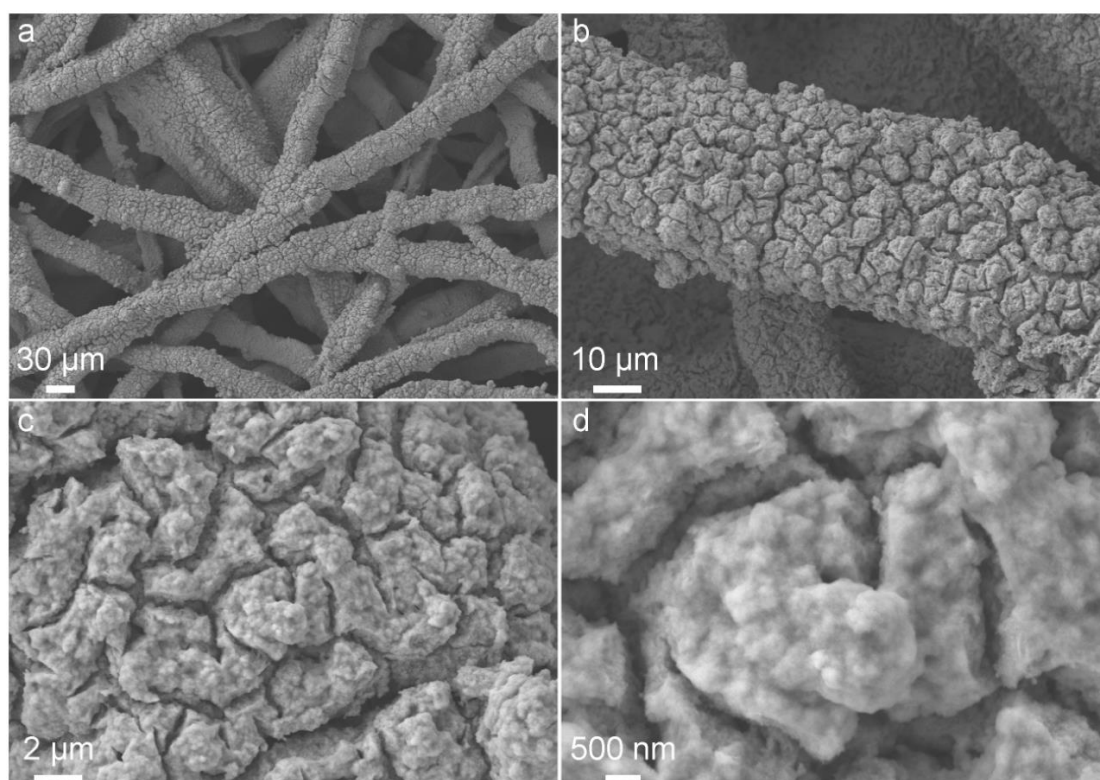


Figure S15. Low and high-resolution SEM images of the DSC3.

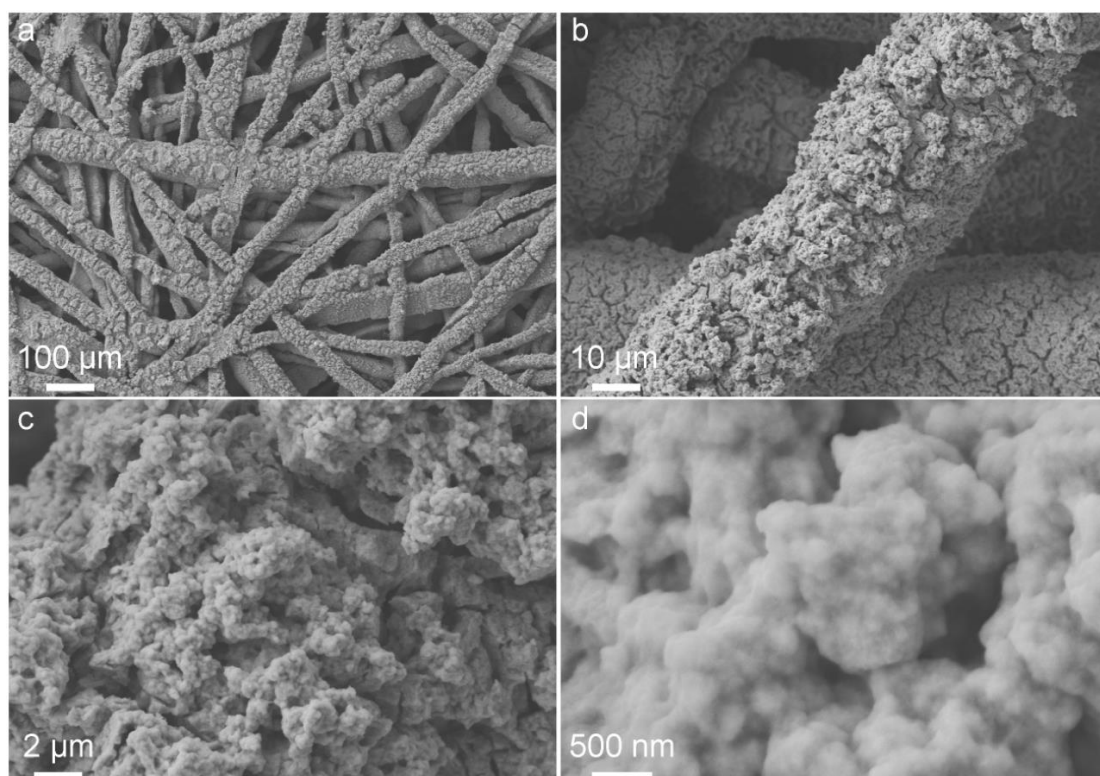


Figure S16. Low and high-resolution SEM images of the DSC.

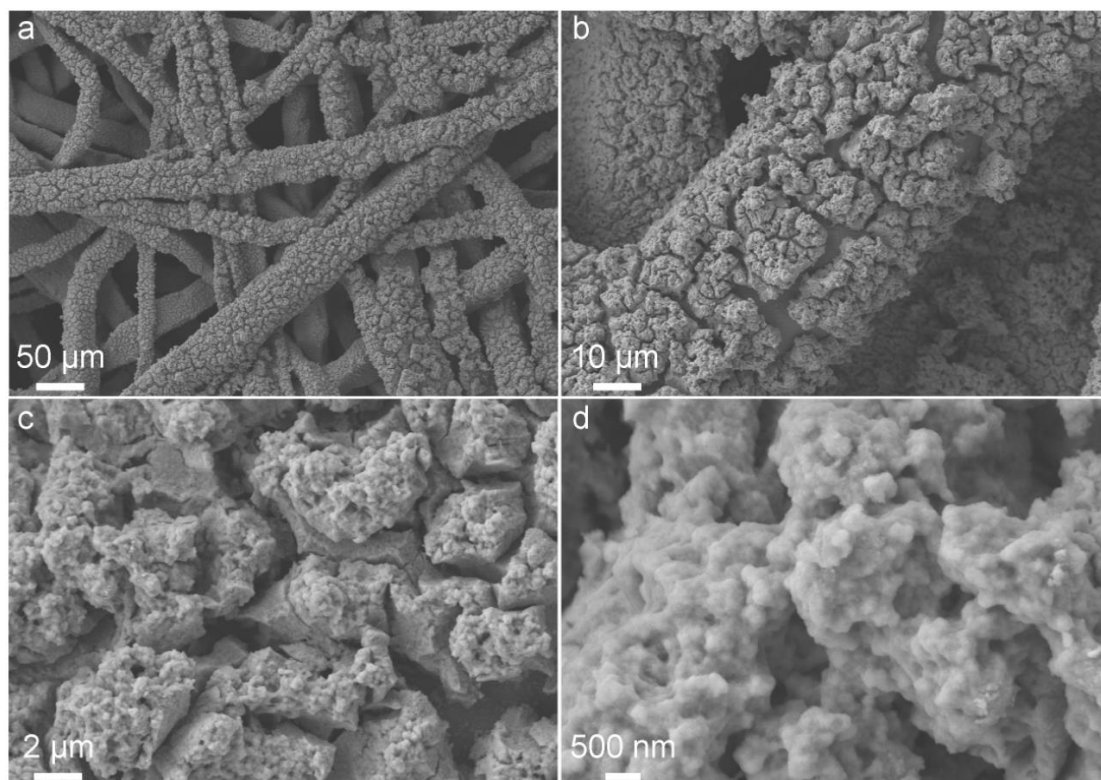


Figure S17. Low and high-resolution SEM images of the DSC5.

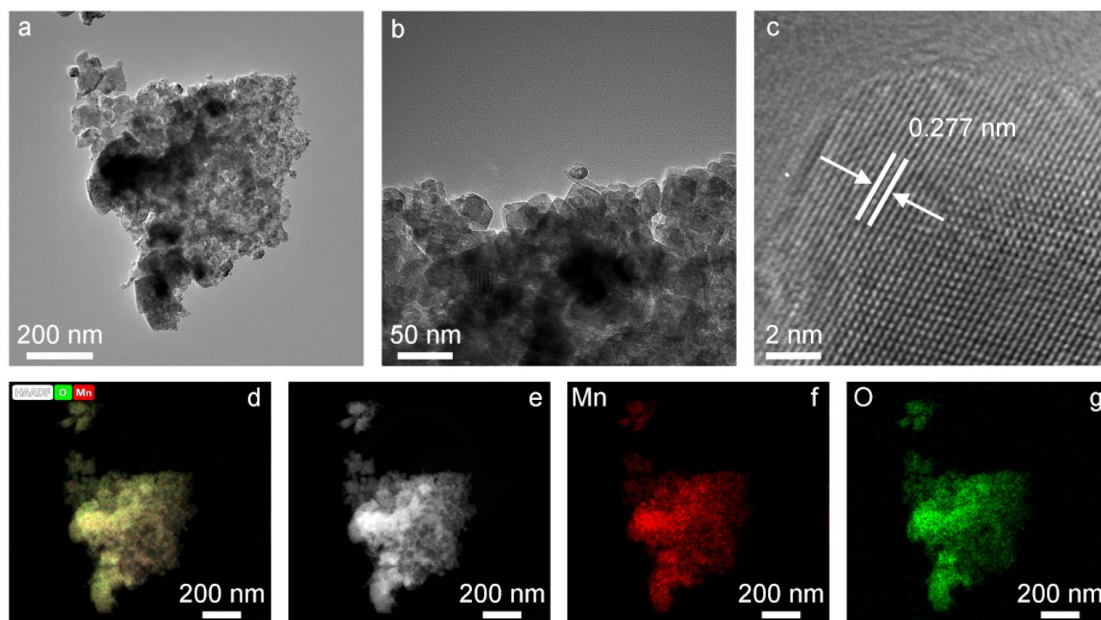


Figure S18. Low and high-resolution TEM and the corresponding elemental mapping images of the Mn_3O_4 .

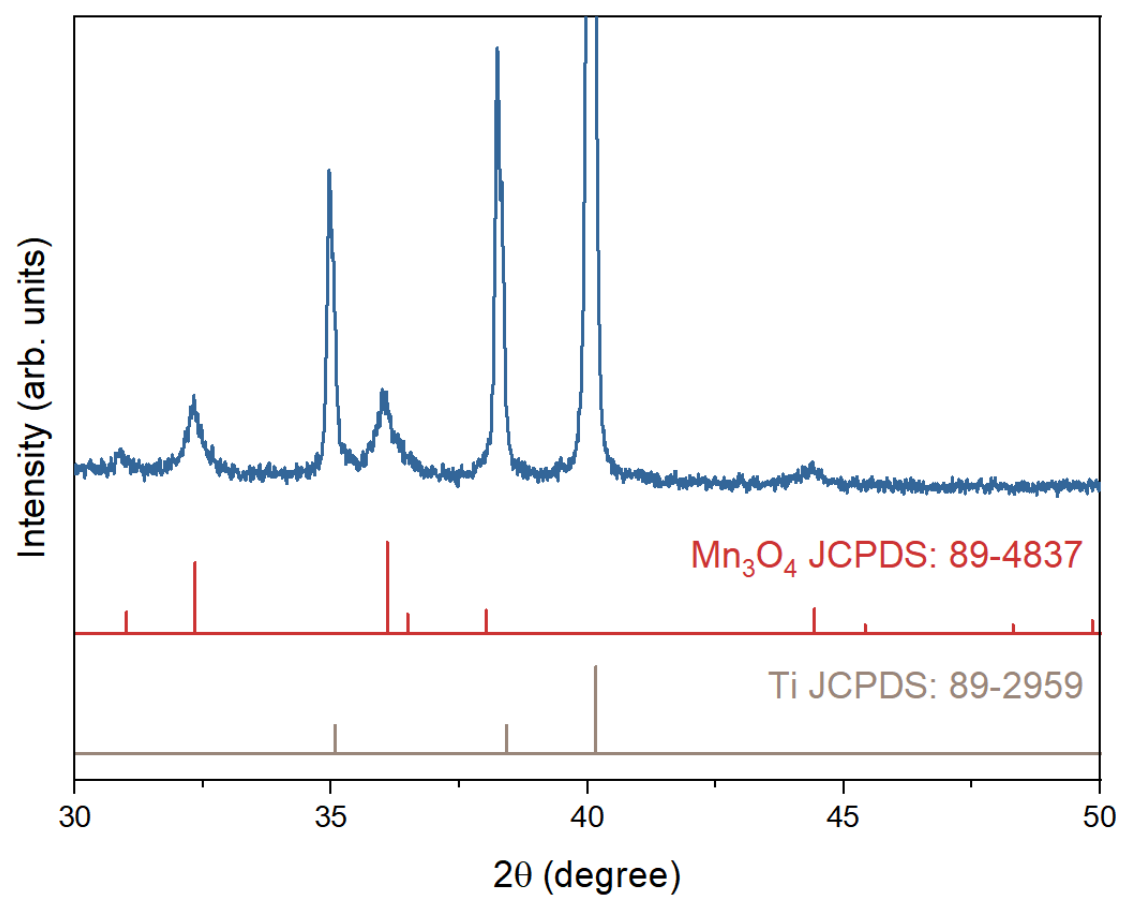


Figure S19. XRD pattern of Mn₃O₄.

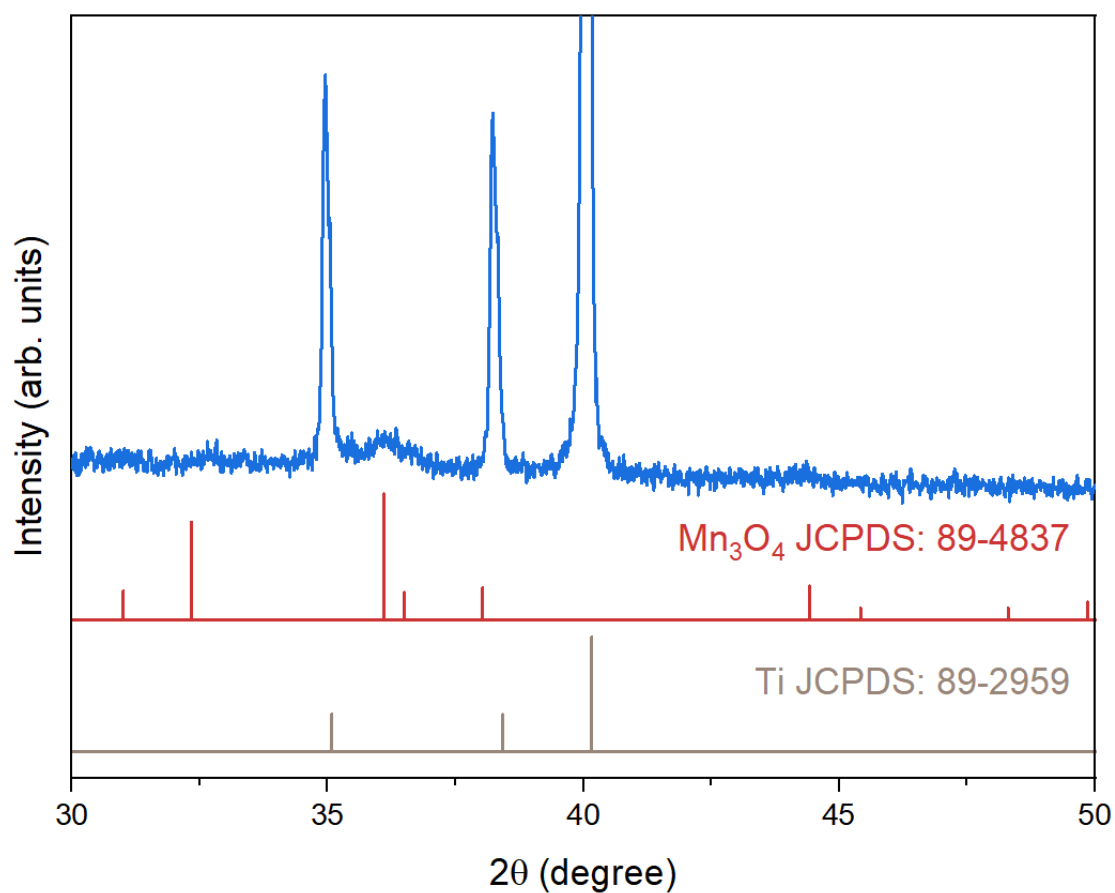


Figure S20. XRD pattern of SSC.

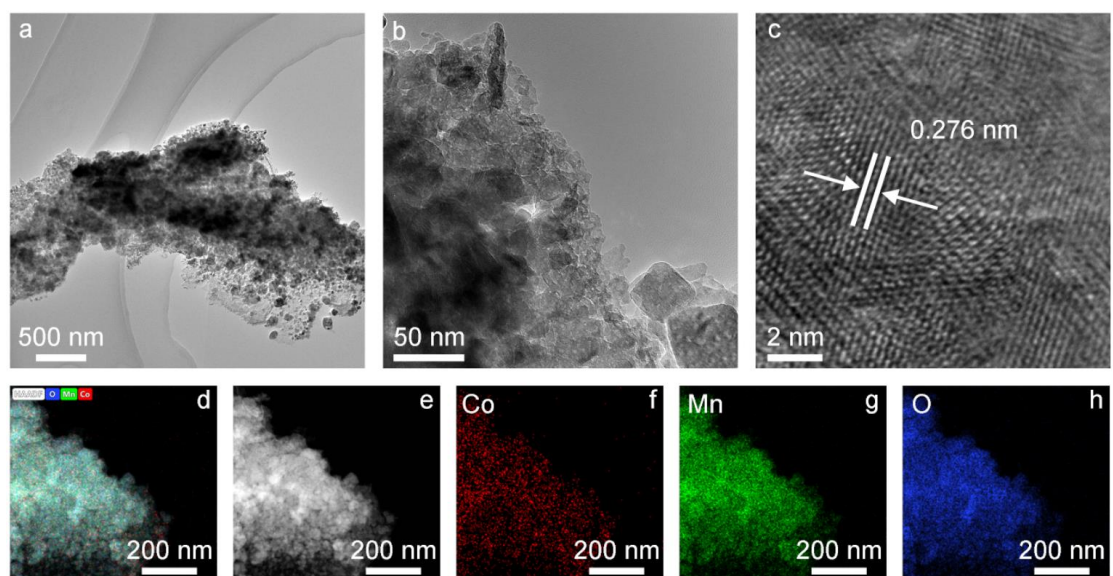


Figure S21. Low and high-resolution TEM and the corresponding elemental mapping images of the SSC.

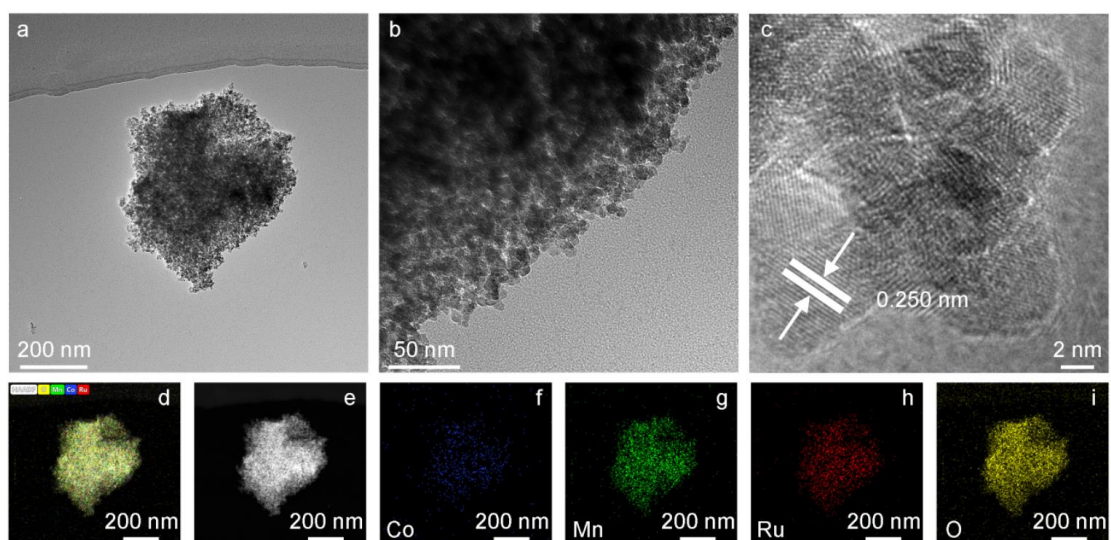


Figure S24. Low and high-resolution TEM and the corresponding elemental mapping images of the DSC2.

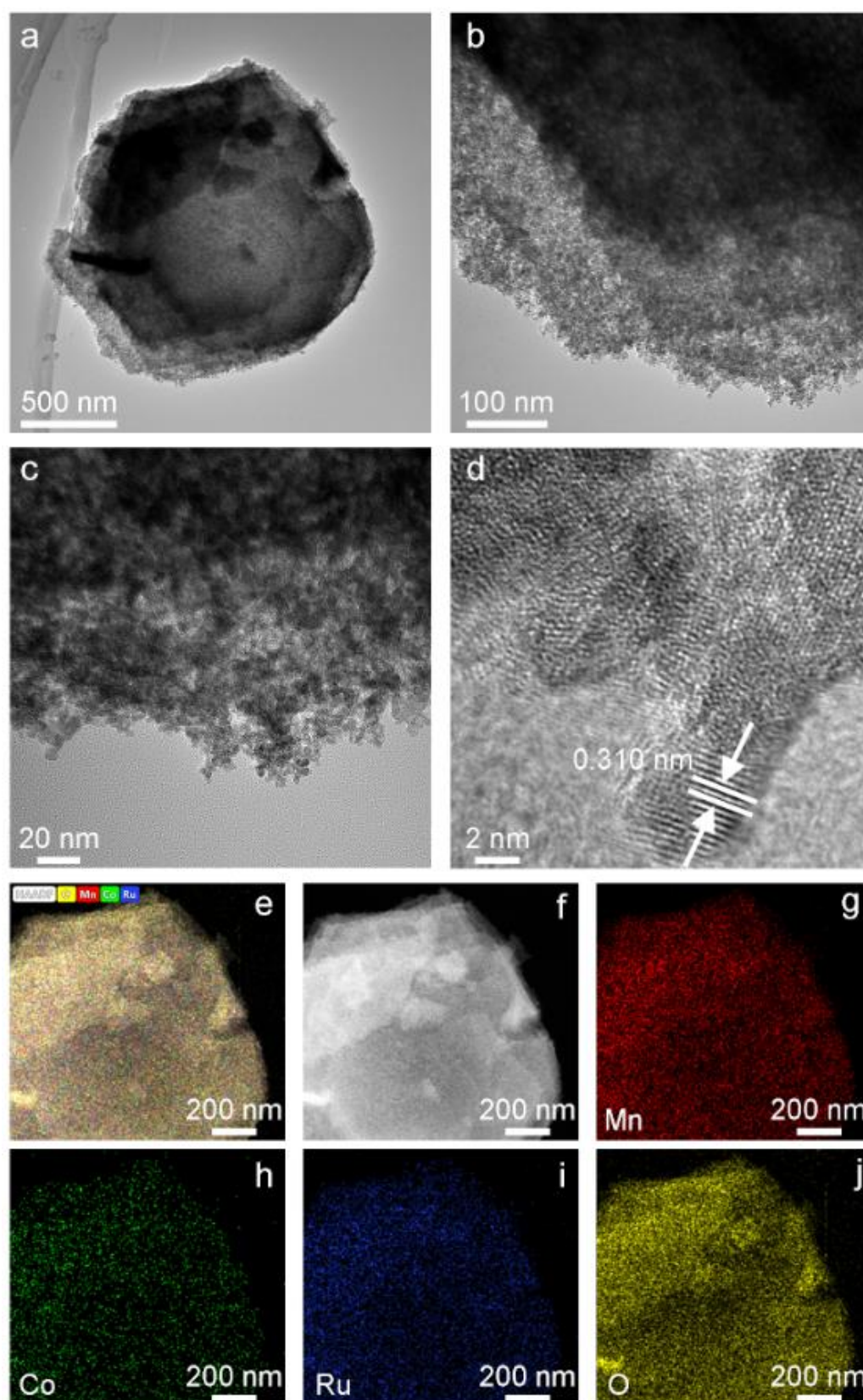


Figure S25. Low and high-resolution TEM and the corresponding elemental mapping images of the DSC3.

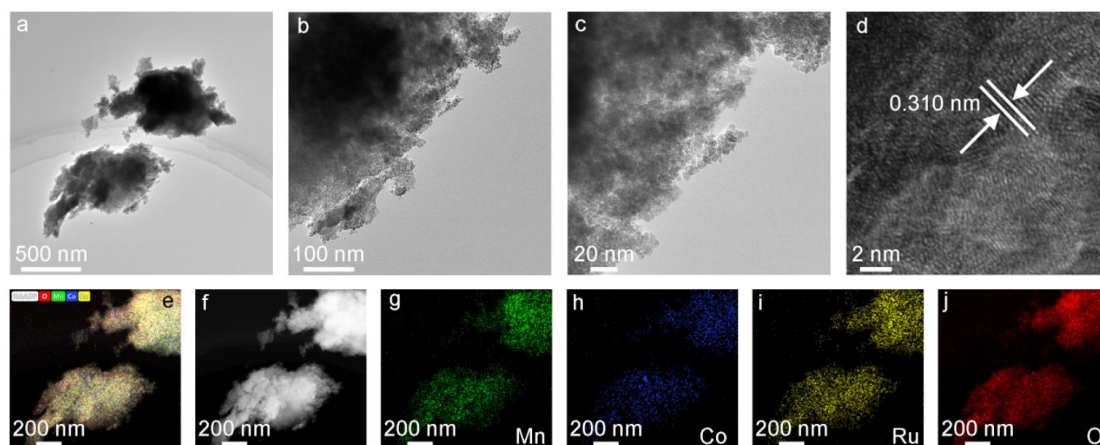


Figure S26. Low and high-resolution TEM and the corresponding elemental mapping images of the DSC.

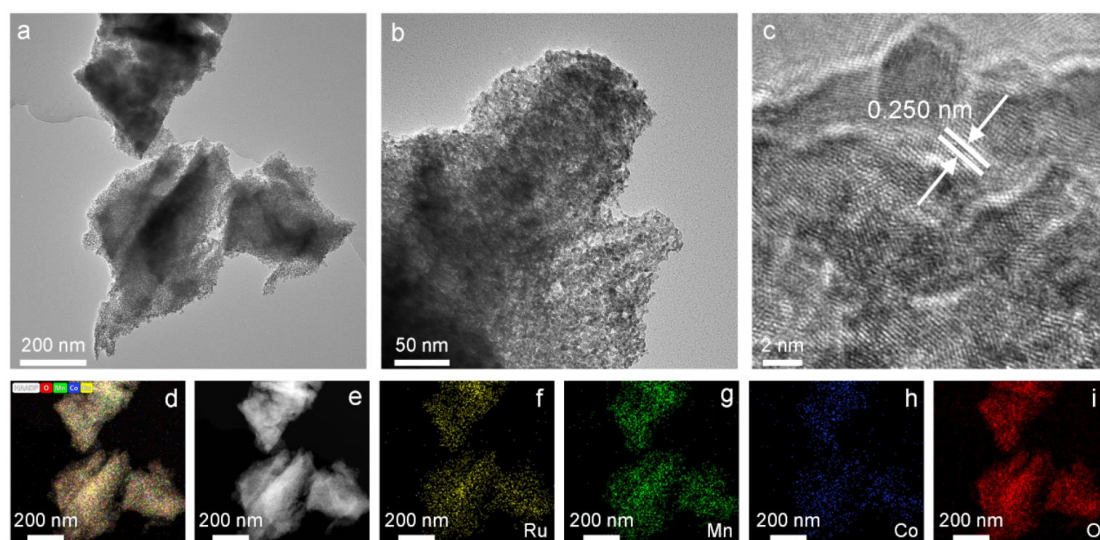


Figure S27. Low and high-resolution TEM and the corresponding elemental mapping images of the DSC5.

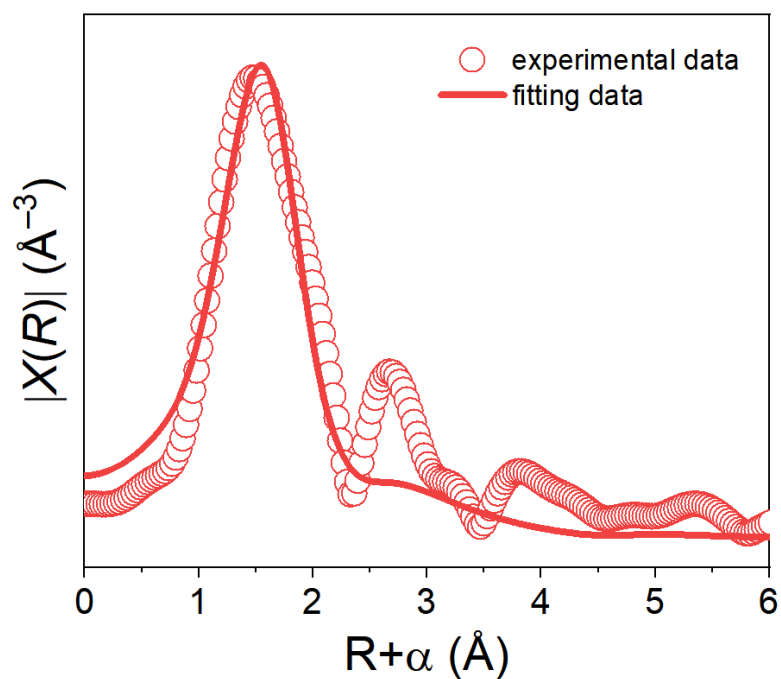


Figure S28. Experimental (symbols) and fitted (solid line) R-space FT-EXAFS spectra of Ru K-edge for DSC sample. $R+\alpha$ denotes the phase-uncorrected radial distance from the absorbing atom.

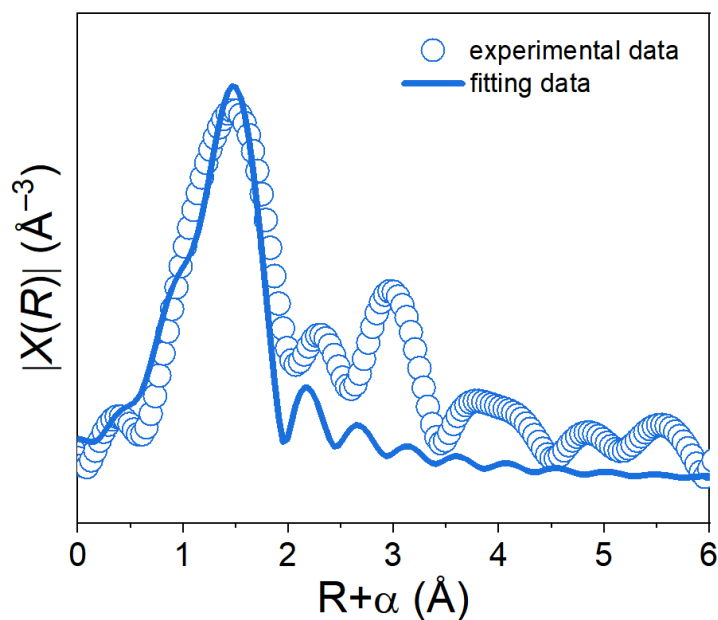


Figure S29. Experimental (symbols) and fitted (solid line) R-space FT-EXAFS spectra of Co K-edge for DSC sample. $R+\alpha$ denotes the phase-uncorrected radial distance from the absorbing atom.

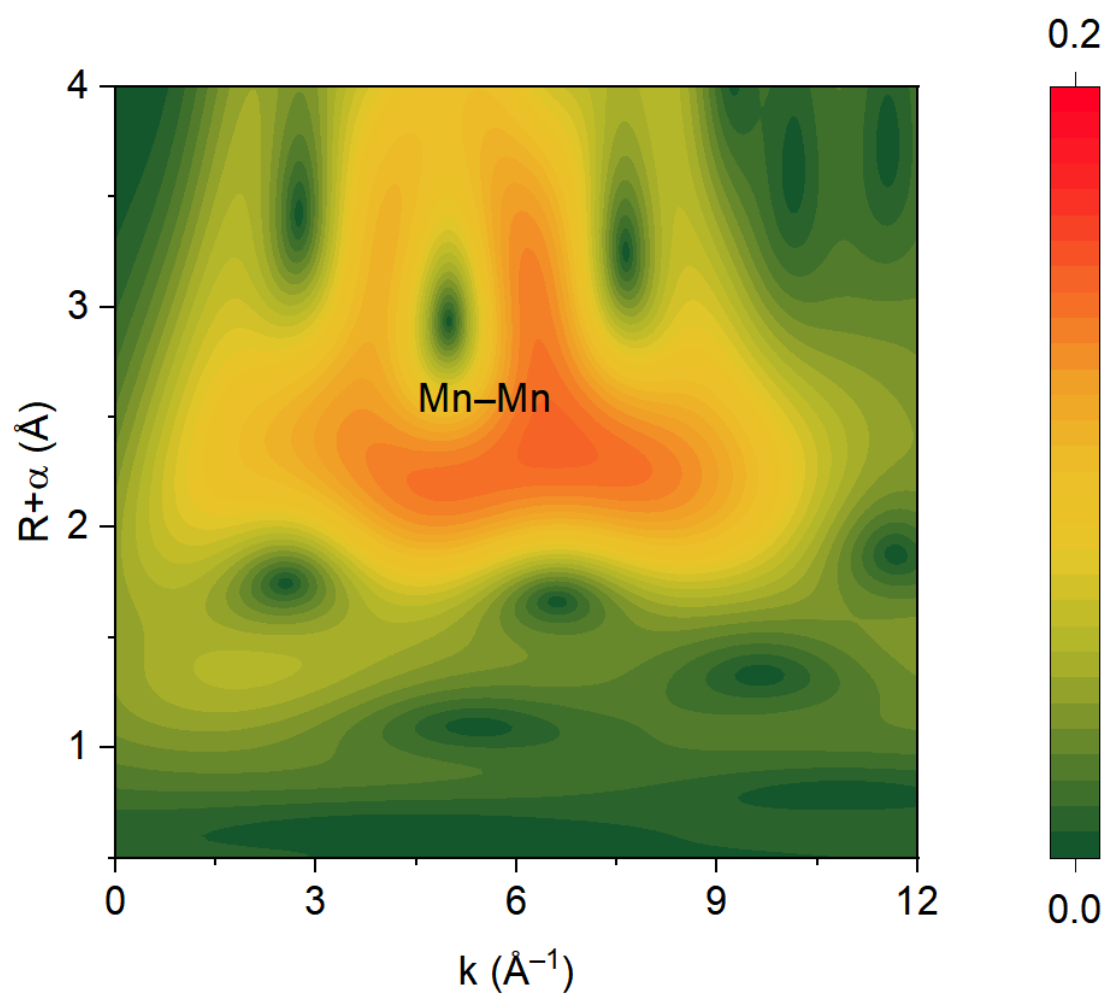


Figure S30. WT of Mn K-edge EXAFS spectra for Mn-foil. $R+\alpha$ denotes the phase-uncorrected radial distance from the absorbing atom.

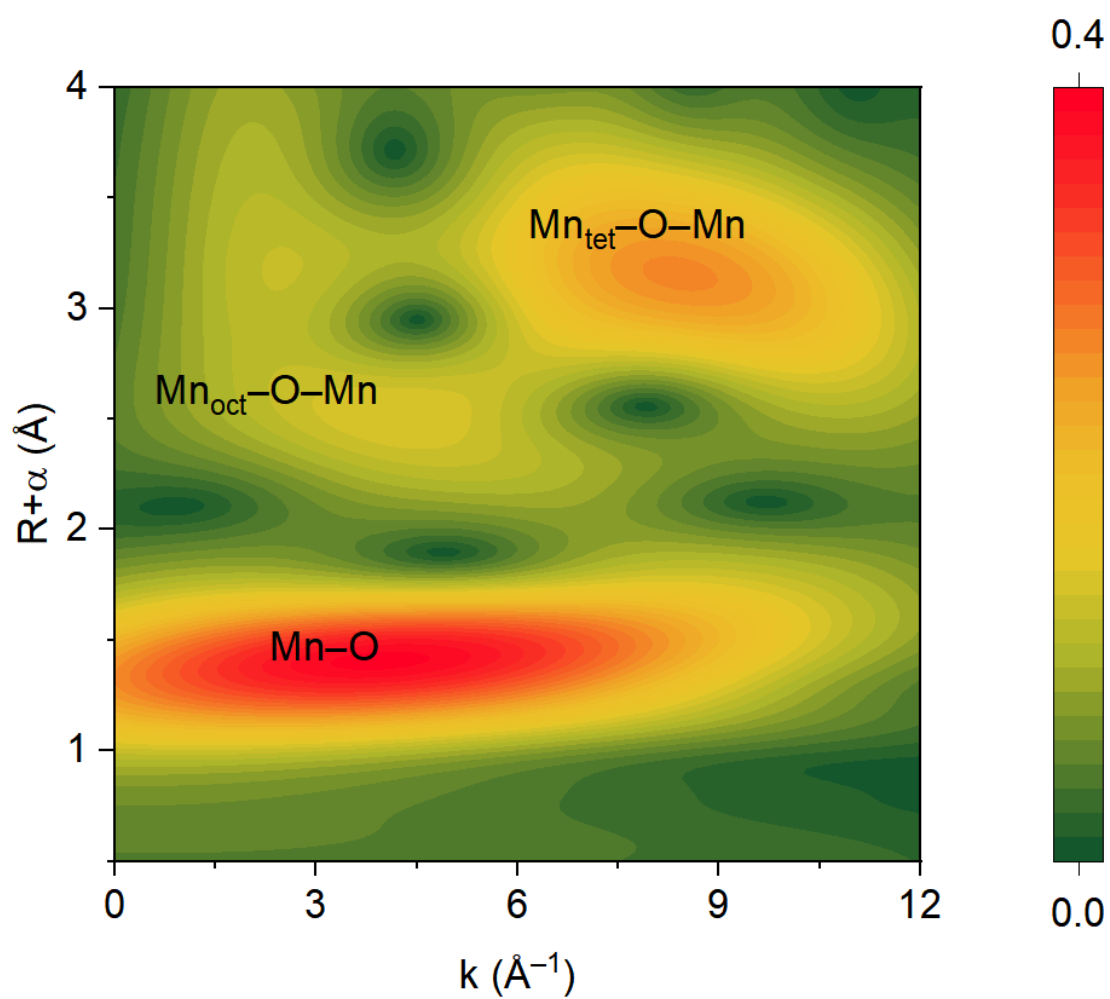


Figure S31. WT of Mn K-edge EXAFS spectra for Mn_3O_4 . $R+\alpha$ denotes the phase-uncorrected radial distance from the absorbing atom.

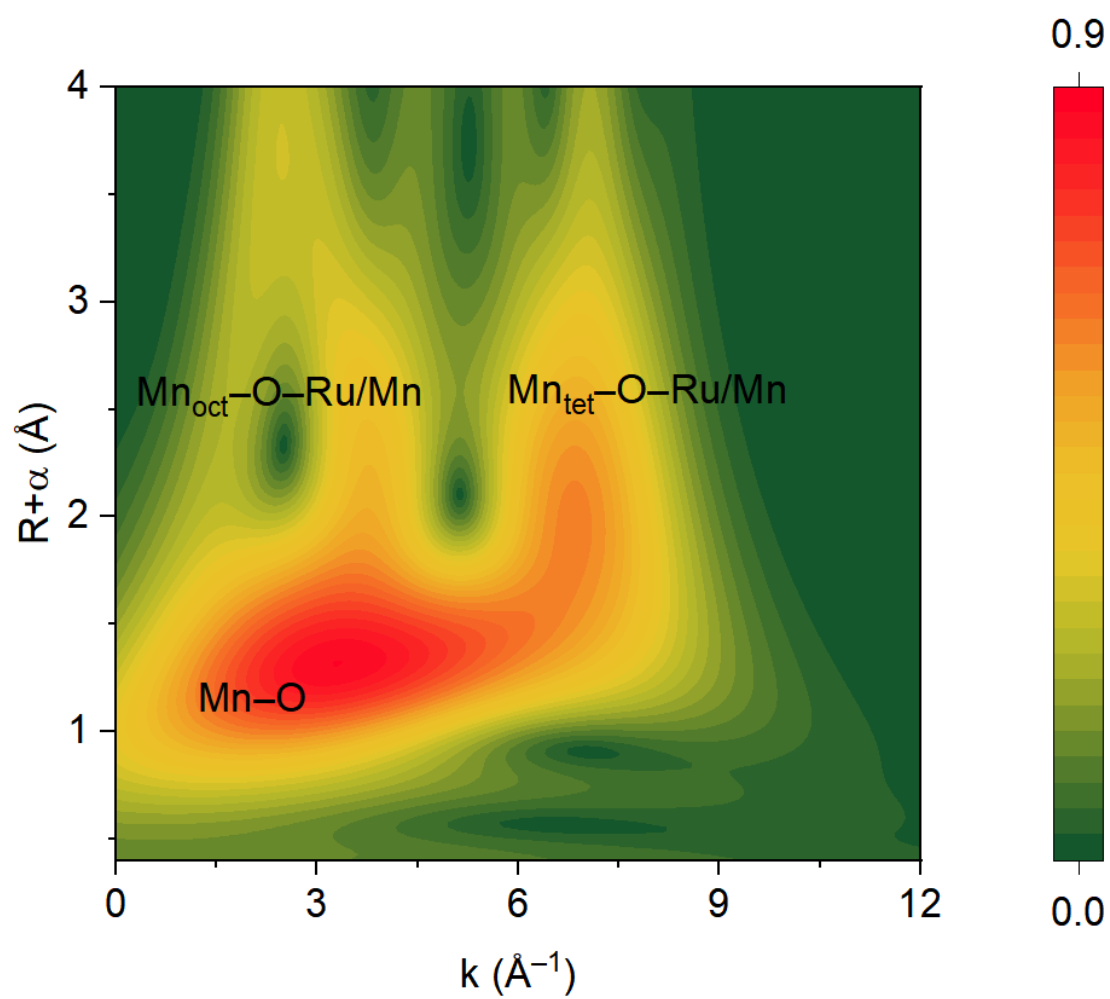


Figure S32. WT of Mn K-edge EXAFS spectra for DSC. $R+\alpha$ denotes the phase-uncorrected radial distance from the absorbing atom.

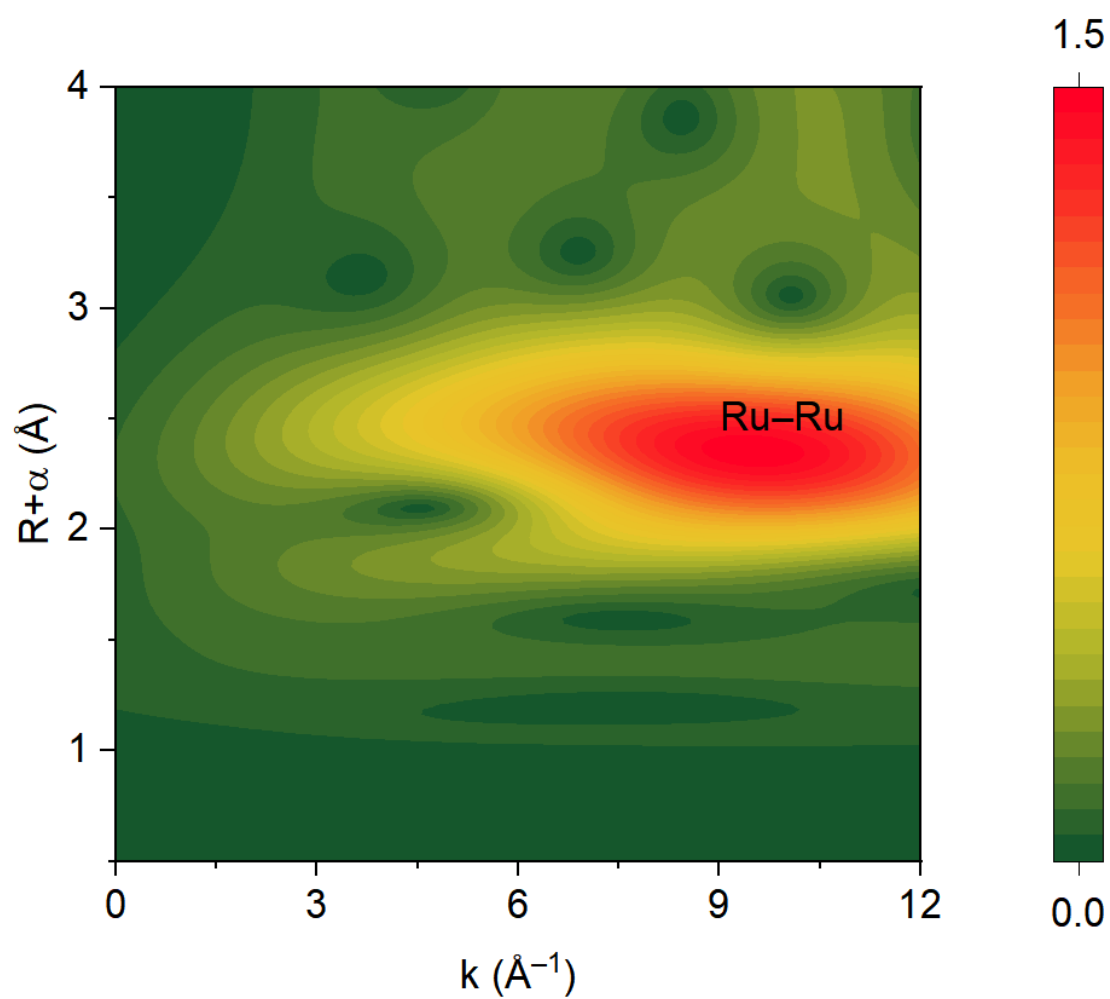


Figure S33. WT of Ru K-edge EXAFS spectra for Ru-foil. $R+\alpha$ denotes the phase-uncorrected radial distance from the absorbing atom.

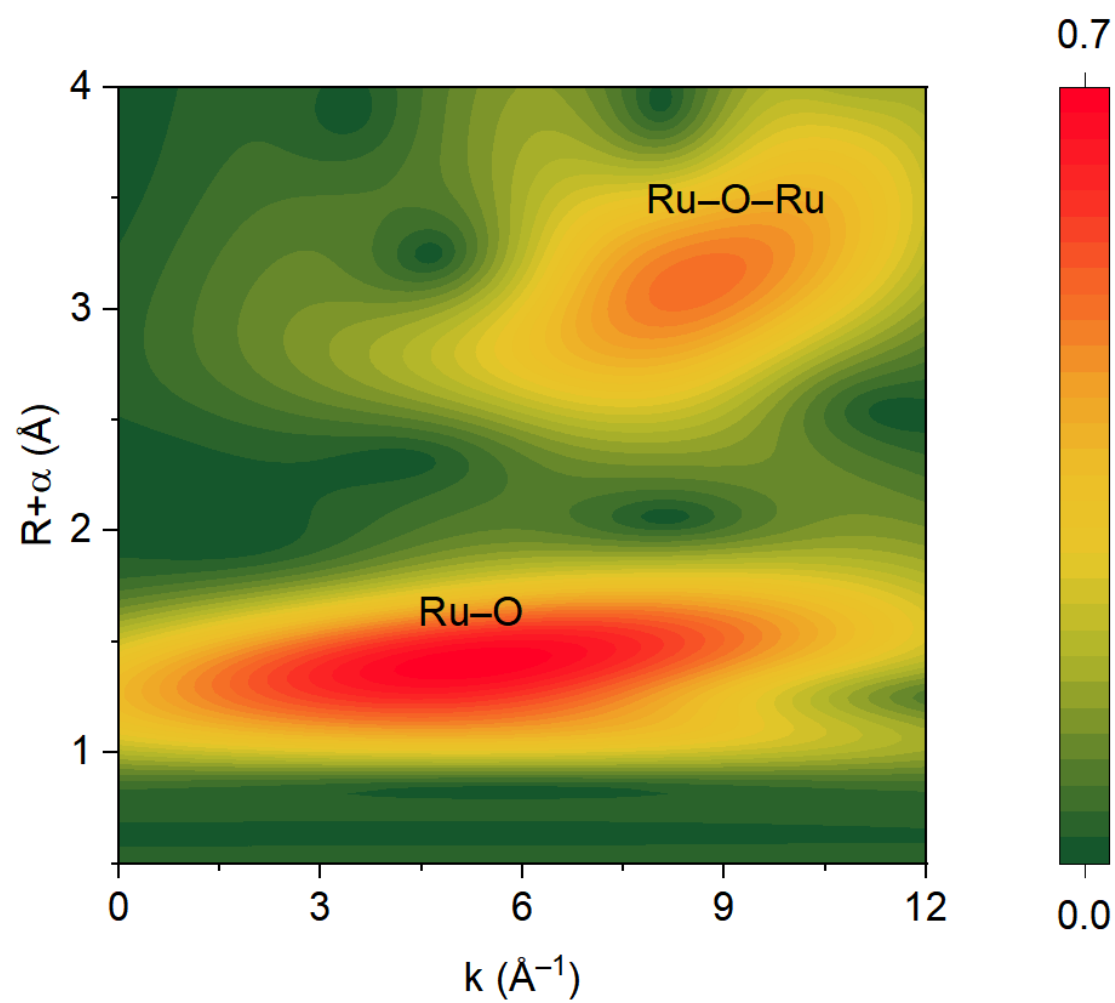


Figure S34. WT of Ru K-edge EXAFS spectra for RuO₂. $R+\alpha$ denotes the phase-uncorrected radial distance from the absorbing atom.

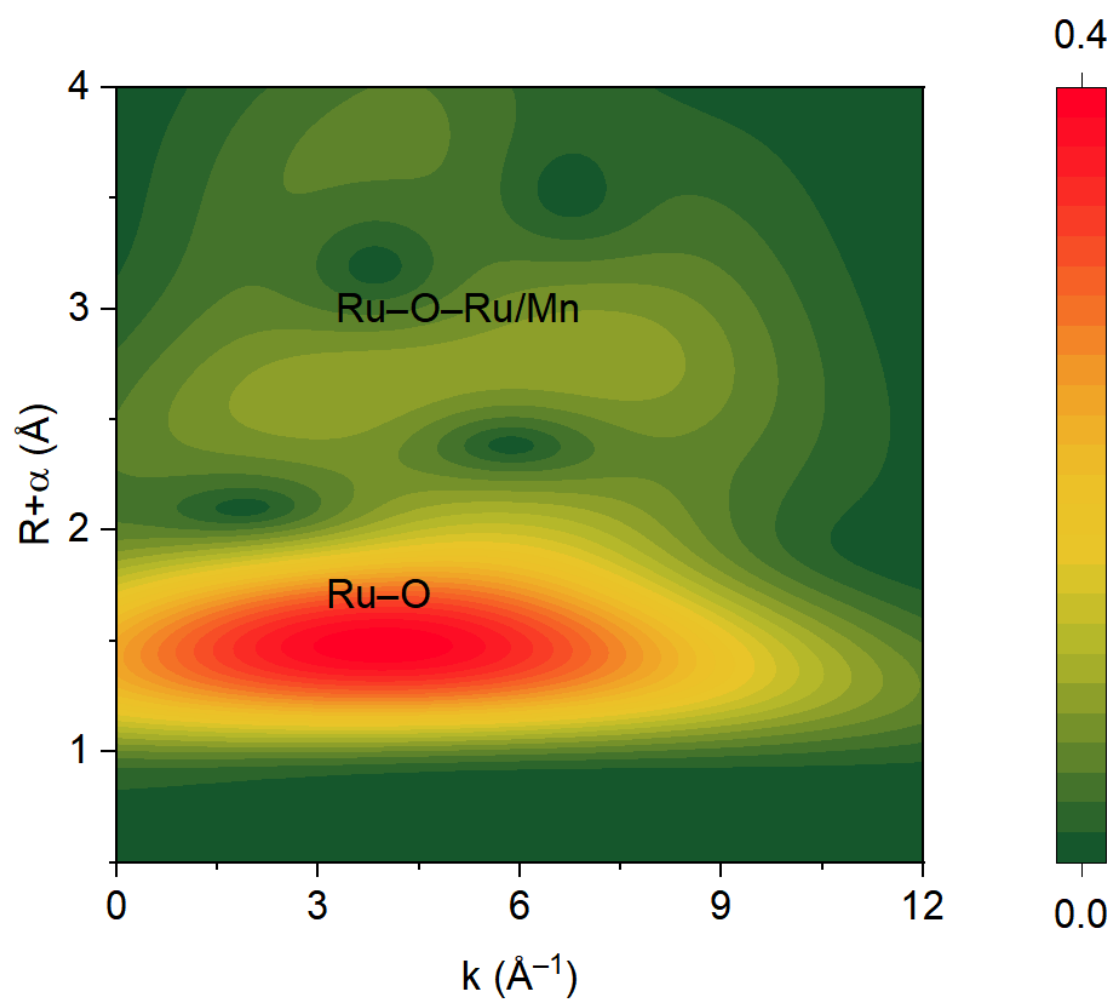


Figure S35. WT of Ru K-edge EXAFS spectra for DSC. $R+\alpha$ denotes the phase-uncorrected radial distance from the absorbing atom.

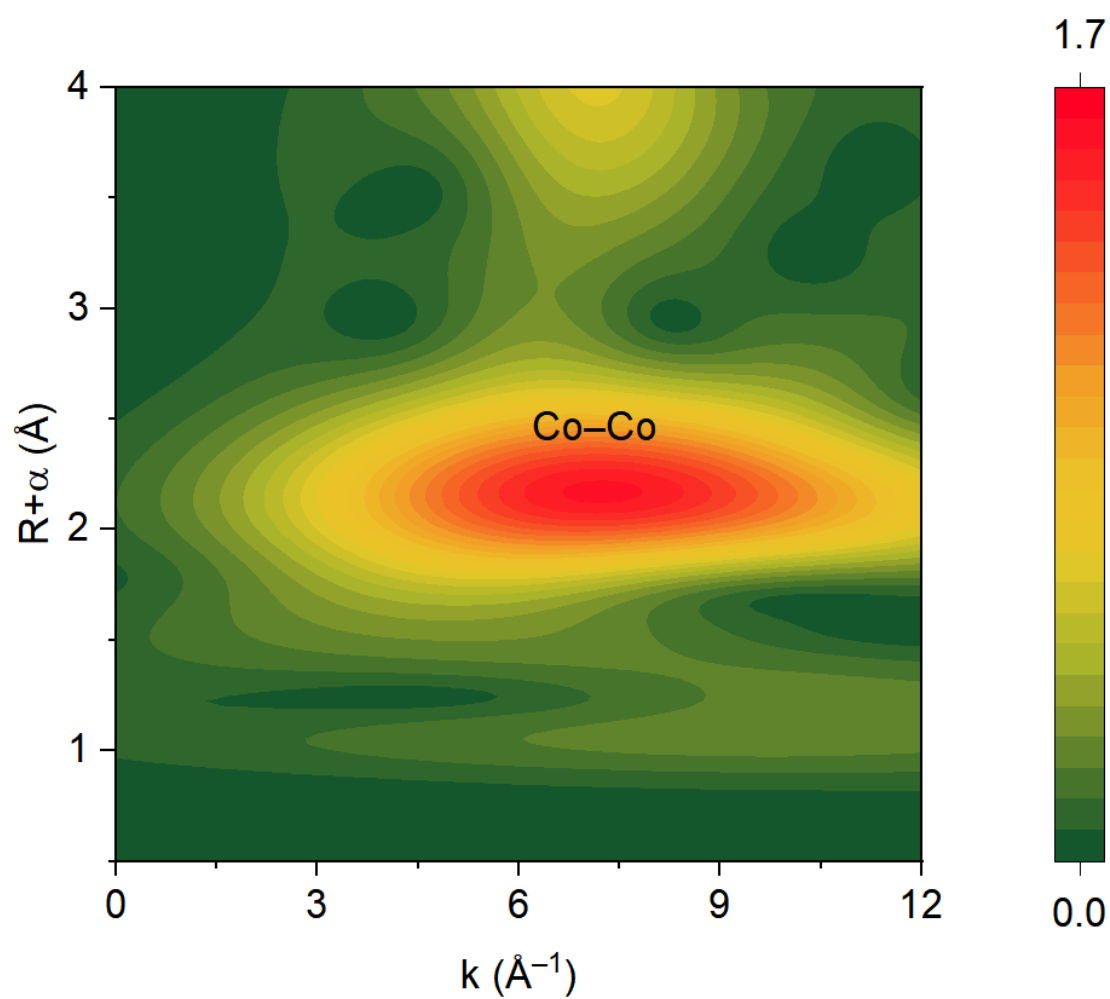


Figure S36. WT of Co K-edge EXAFS spectra for Co-foil. $R+\alpha$ denotes the phase-uncorrected radial distance from the absorbing atom.

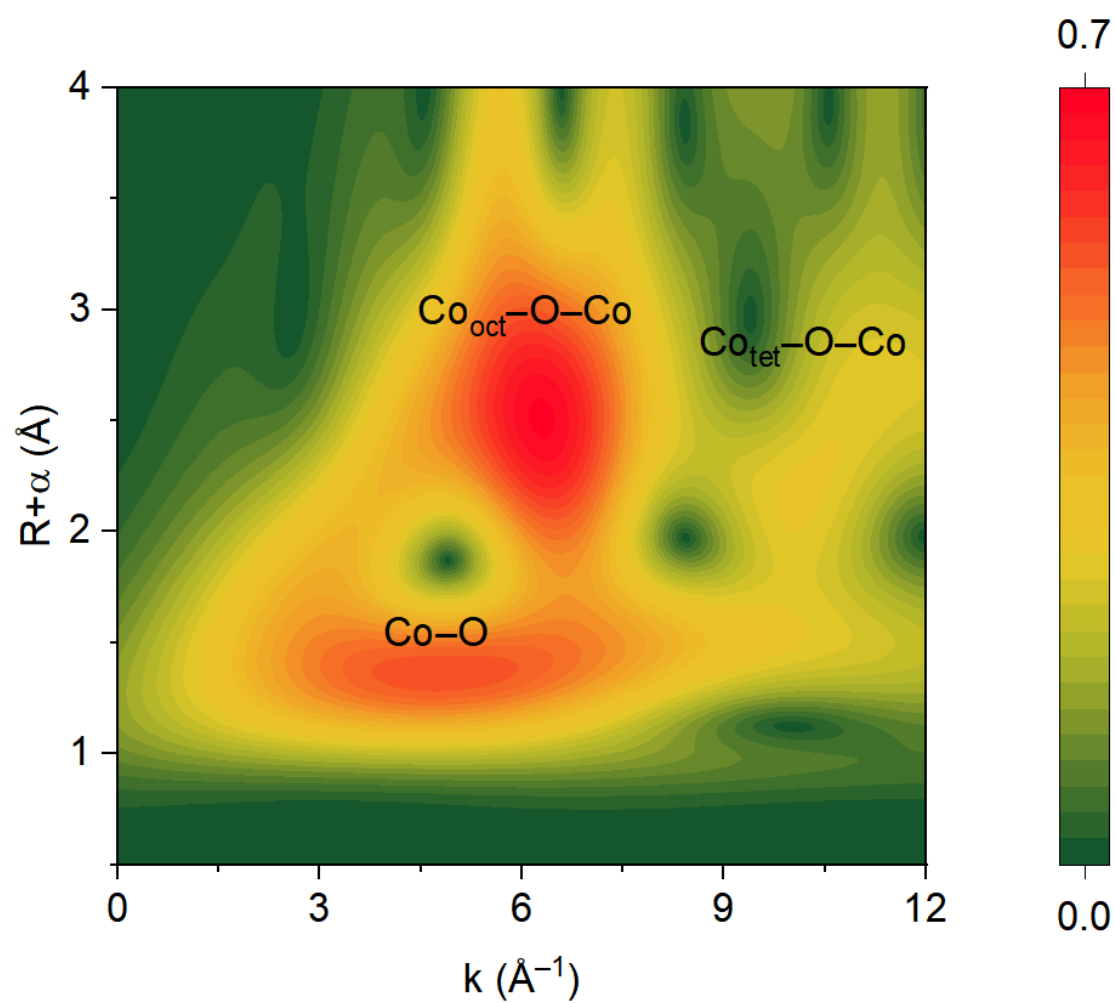


Figure S37. WT of Co K-edge EXAFS spectra for Co_3O_4 . $R+\alpha$ denotes the phase-uncorrected radial distance from the absorbing atom.

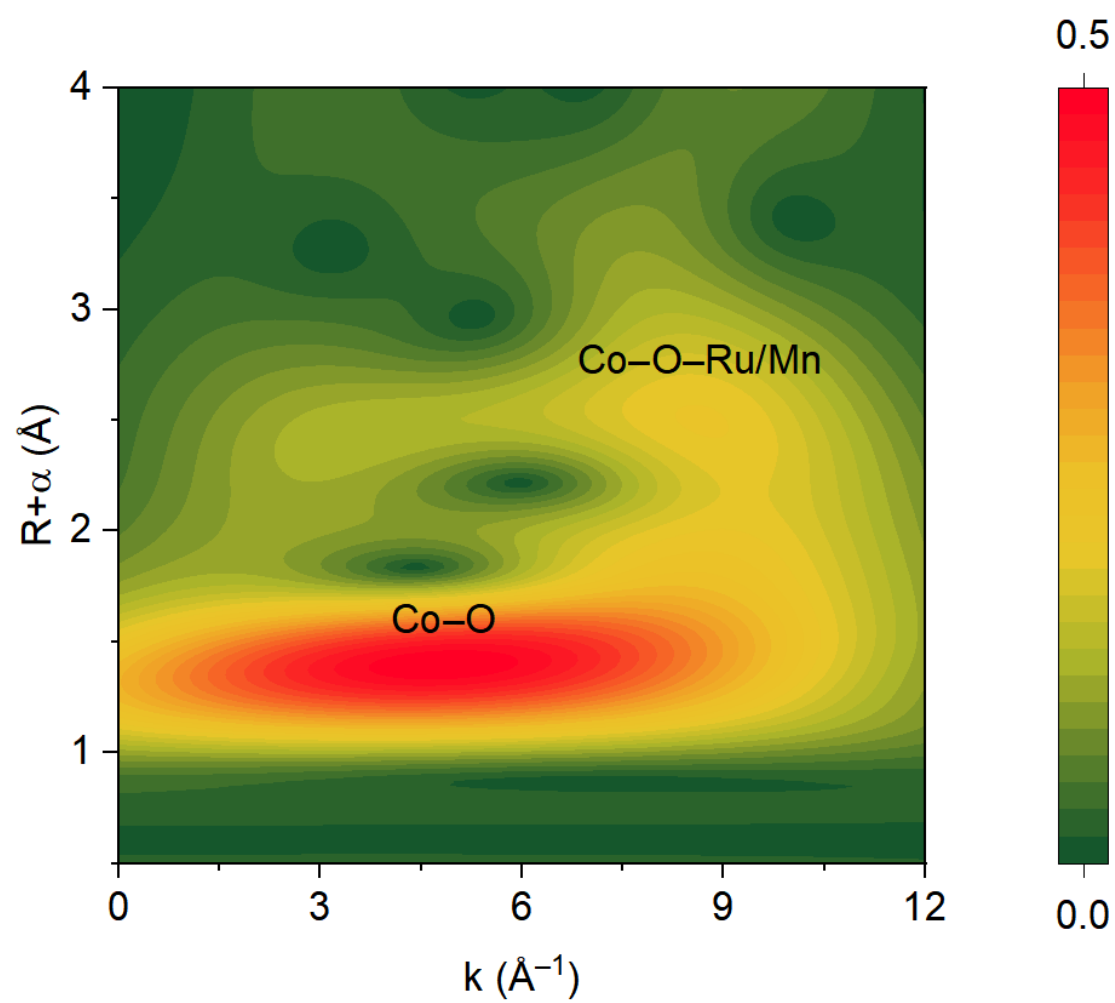


Figure S38. WT of Co K-edge EXAFS spectra for DSC. $R+\alpha$ denotes the phase-uncorrected radial distance from the absorbing atom.

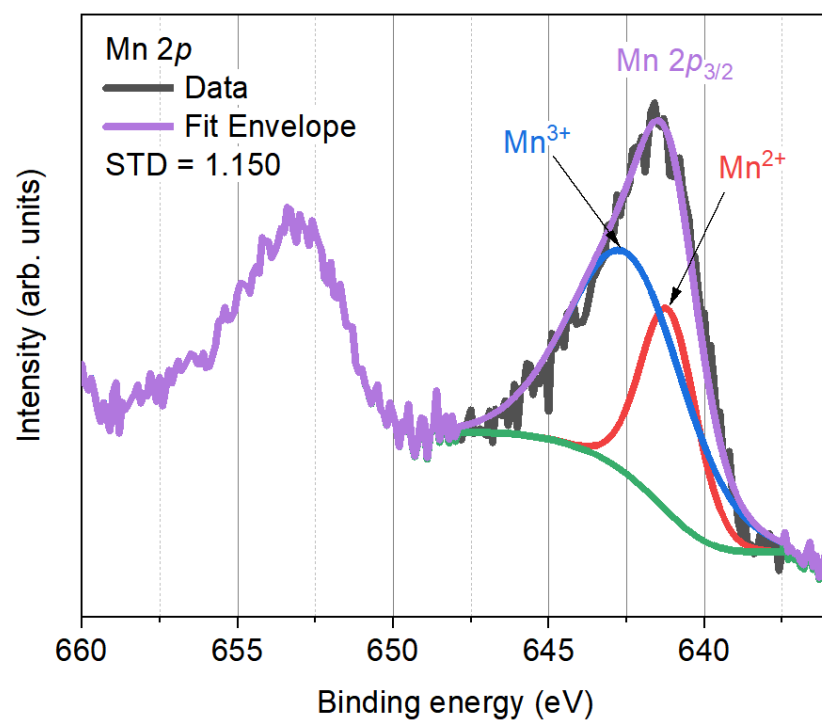


Figure S39. Mn 2p XPS spectra of DSC1.

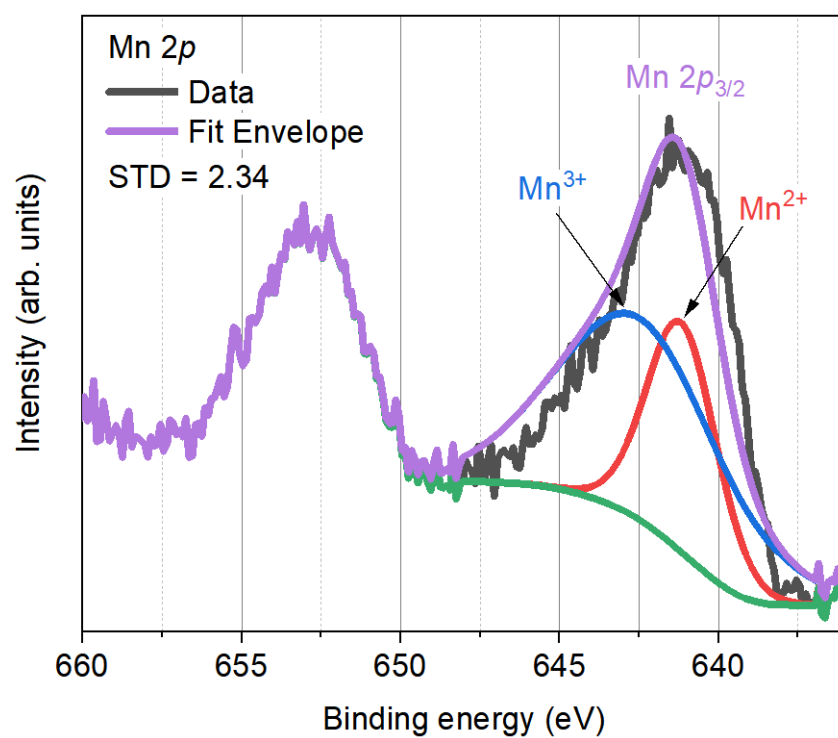


Figure S40. Mn 2p XPS spectra of DSC2.

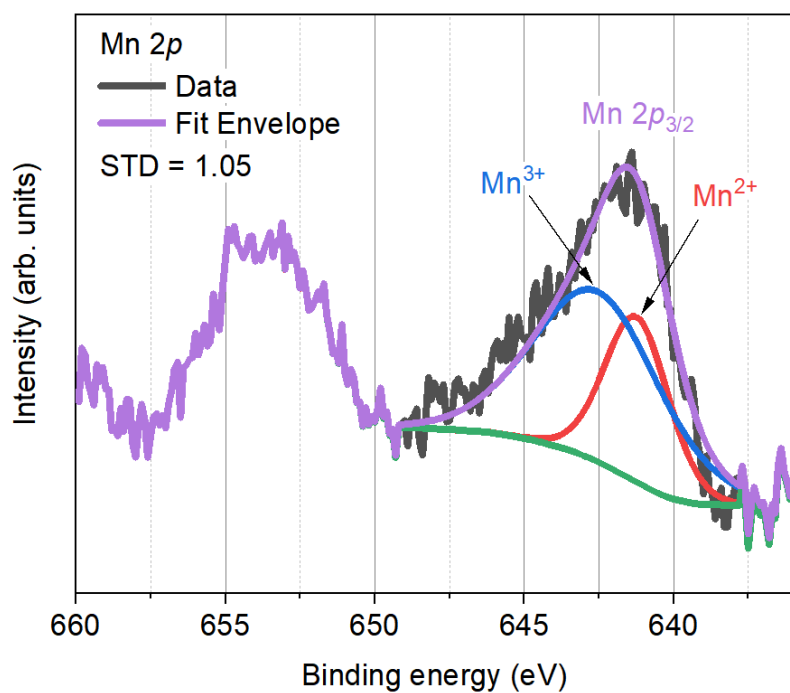


Figure S41. Mn 2p XPS spectra of DSC3.

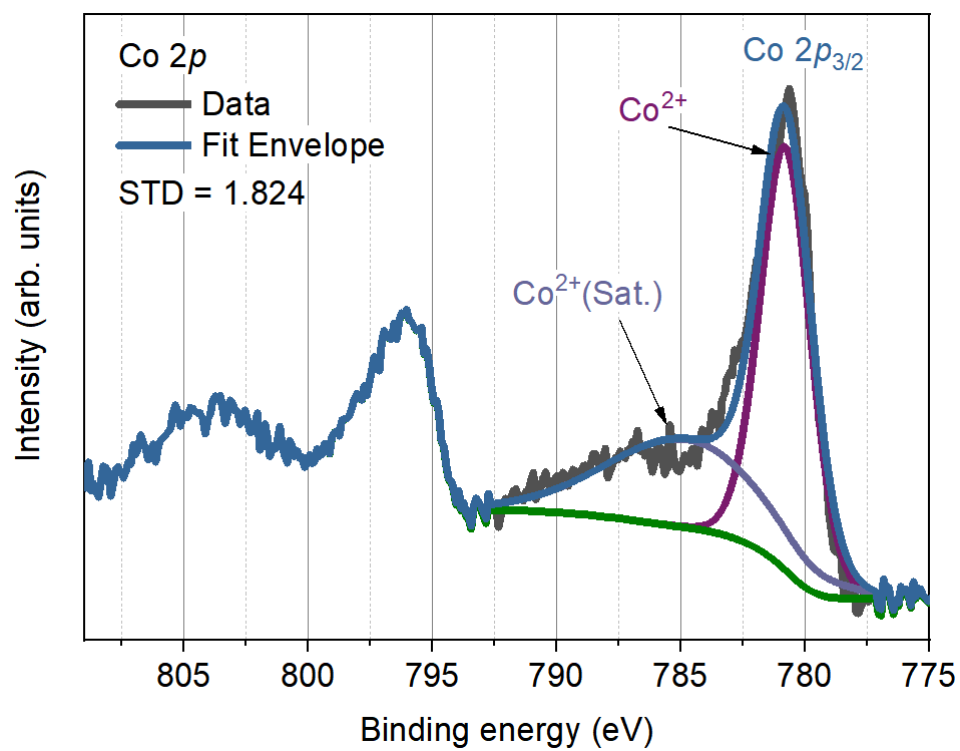


Figure S42. Co 2p XPS spectra of SSC.

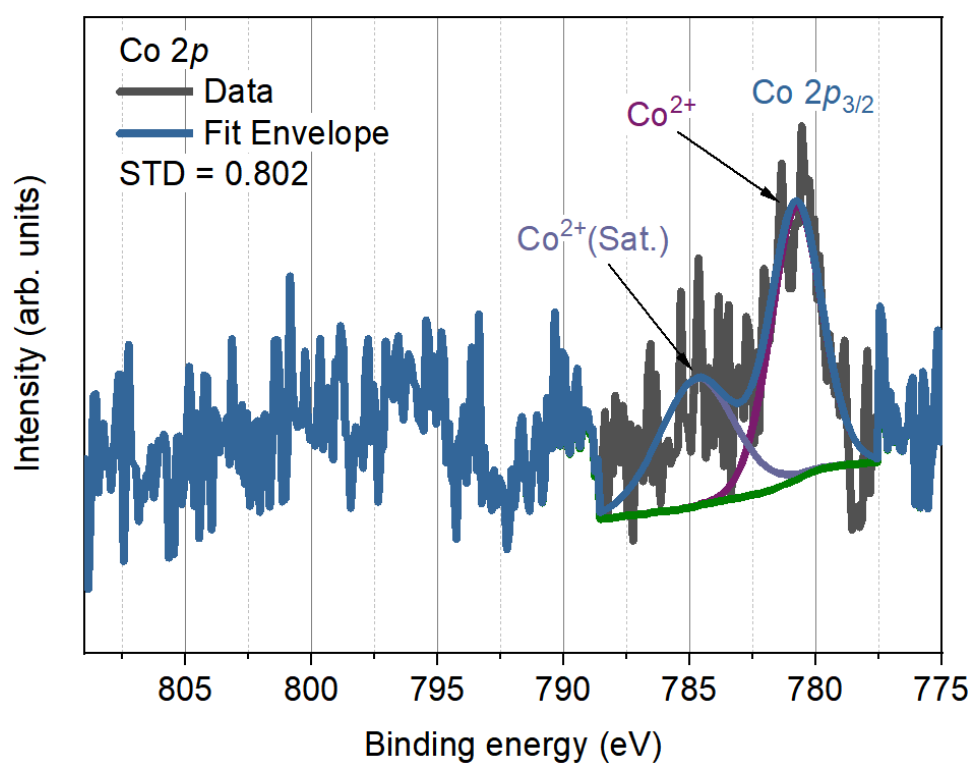


Figure S43. Co 2p XPS spectra of DSC1.

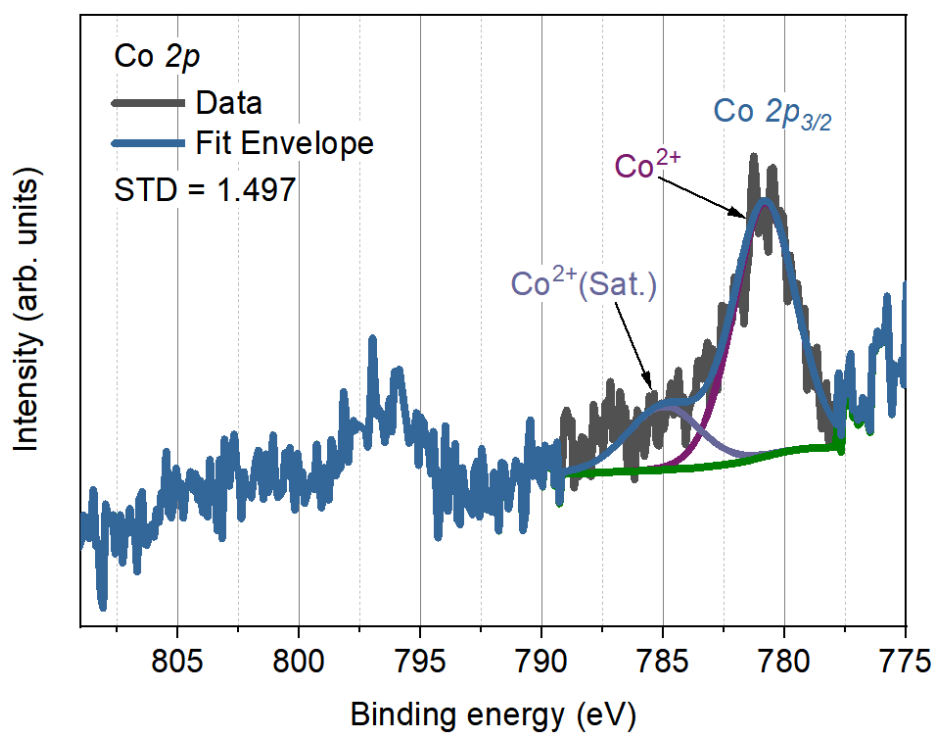


Figure S44. Co 2p XPS spectra of DSC2.

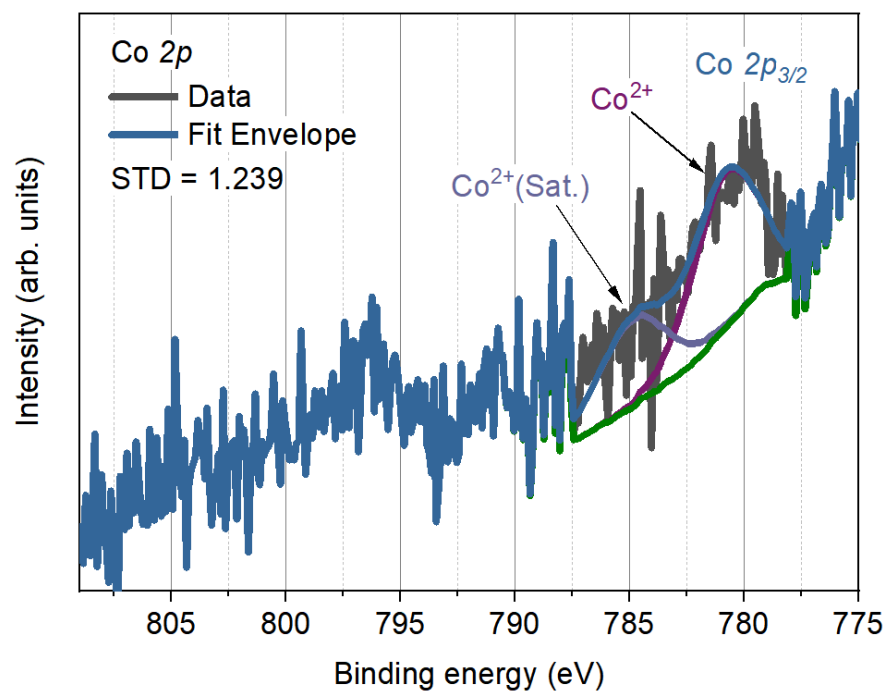


Figure S45. Co 2p XPS spectra of DSC3.

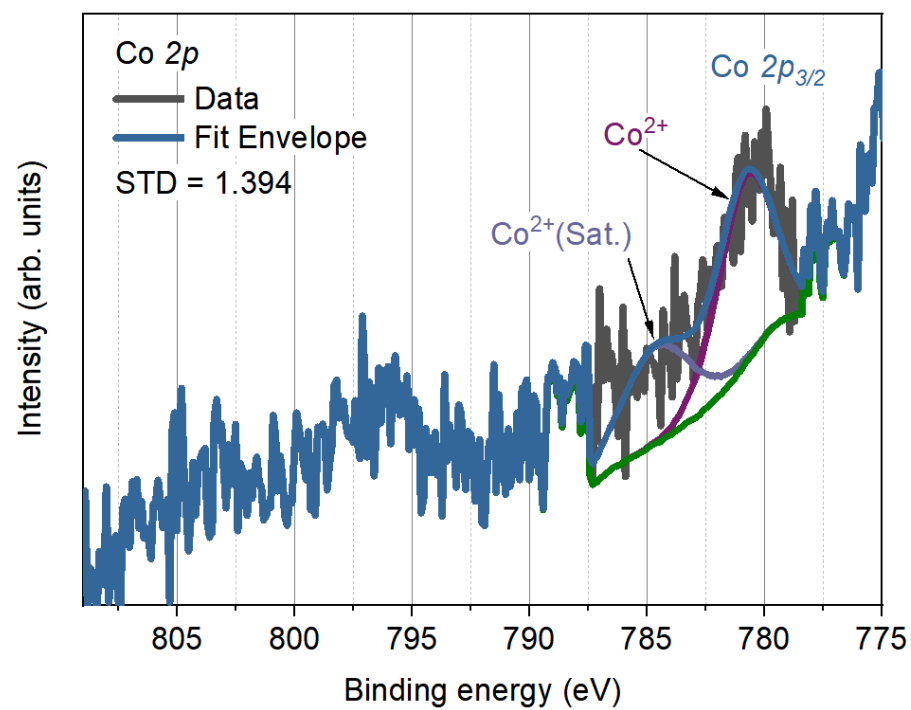


Figure S46. Co 2p XPS spectra of DSC.

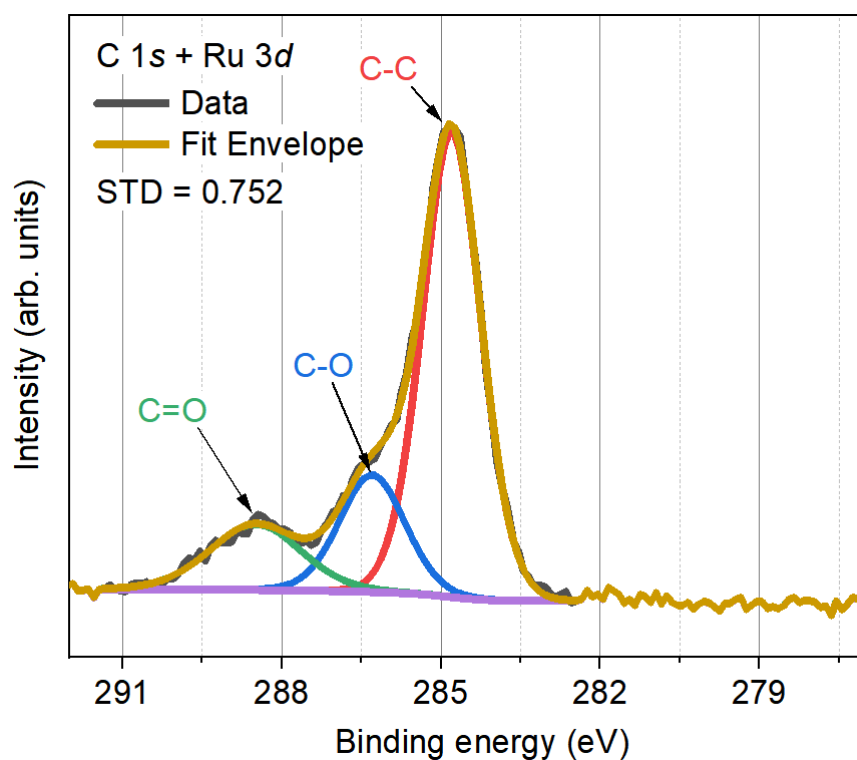


Figure S47. C 1s and Ru 3d XPS spectra of SSC.

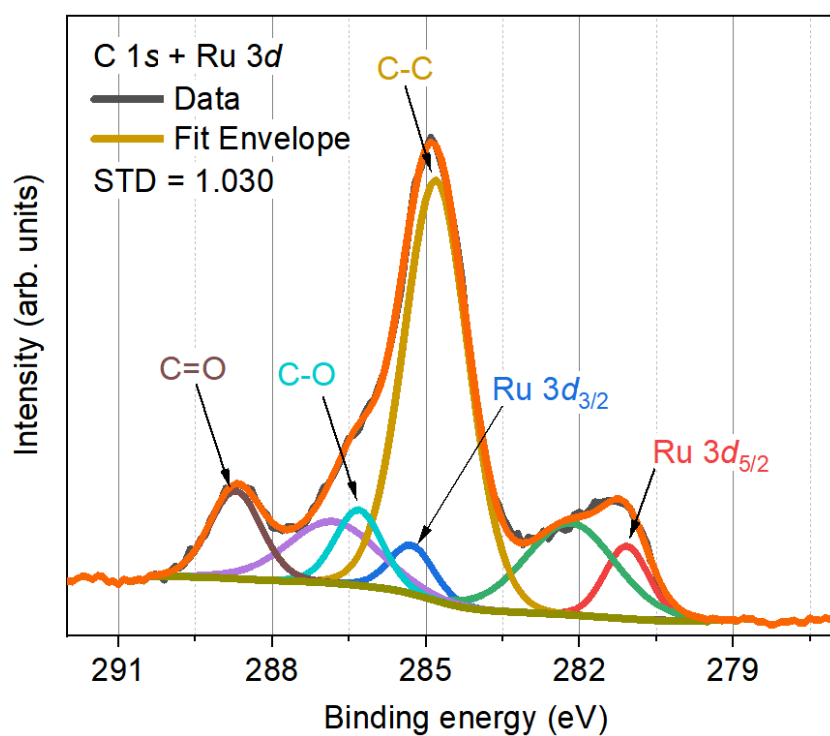


Figure S48. C 1s and Ru 3d XPS spectra of DSC1.

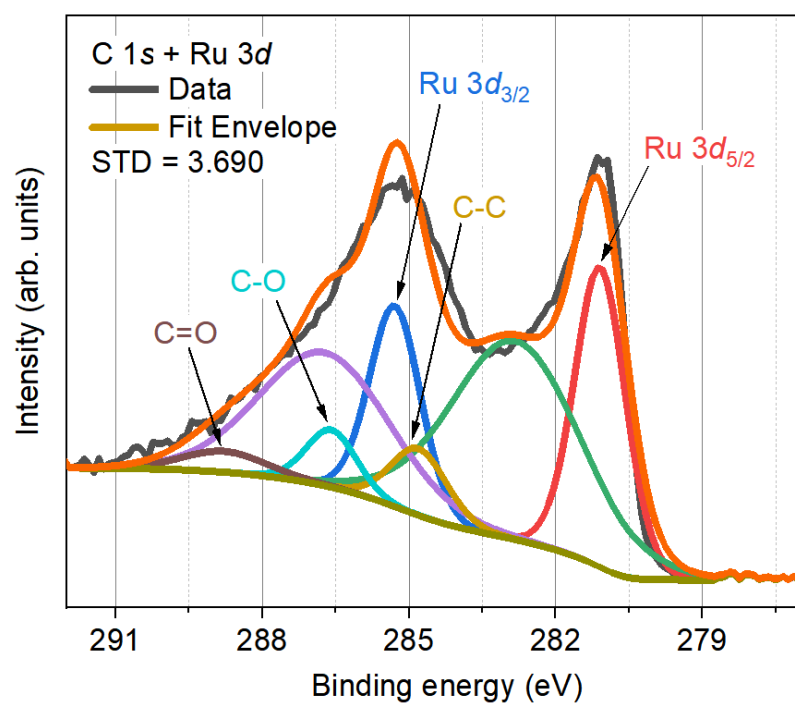


Figure S49. C 1s and Ru 3d XPS spectra of DSC2.

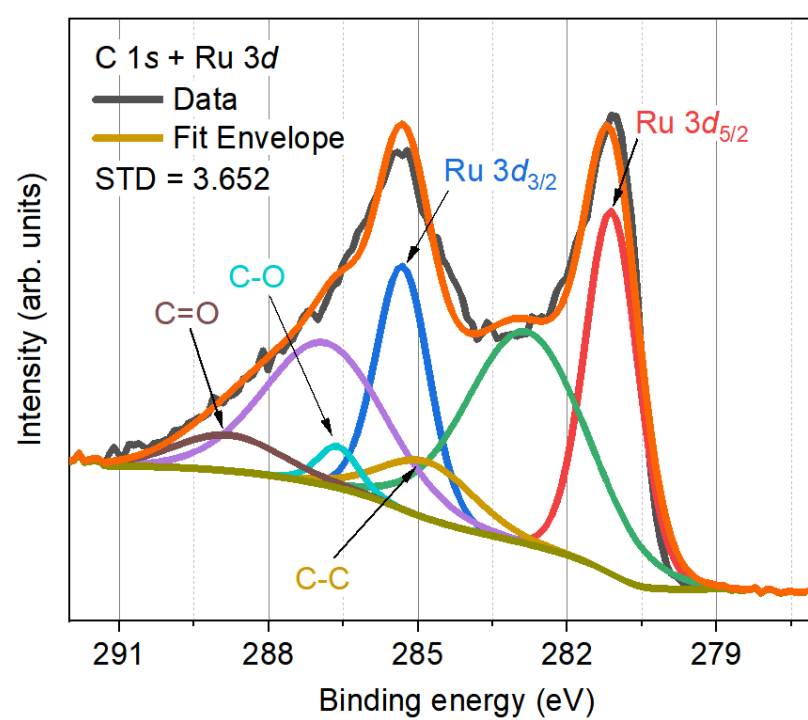


Figure S50. C 1s and Ru 3d XPS spectra of DSC3.

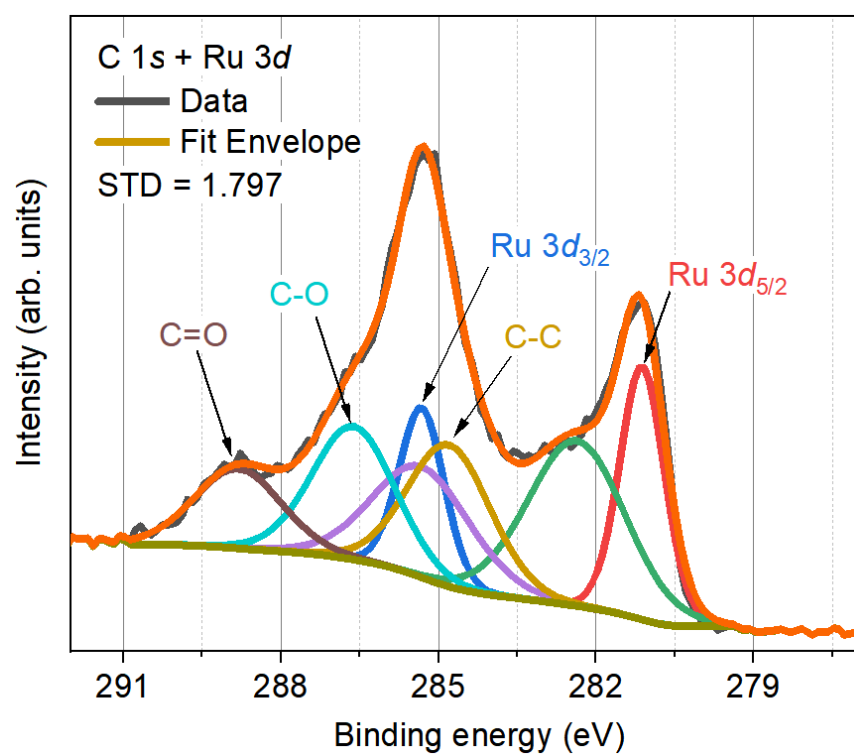


Figure S51. C 1s and Ru 3d XPS spectra of DSC.

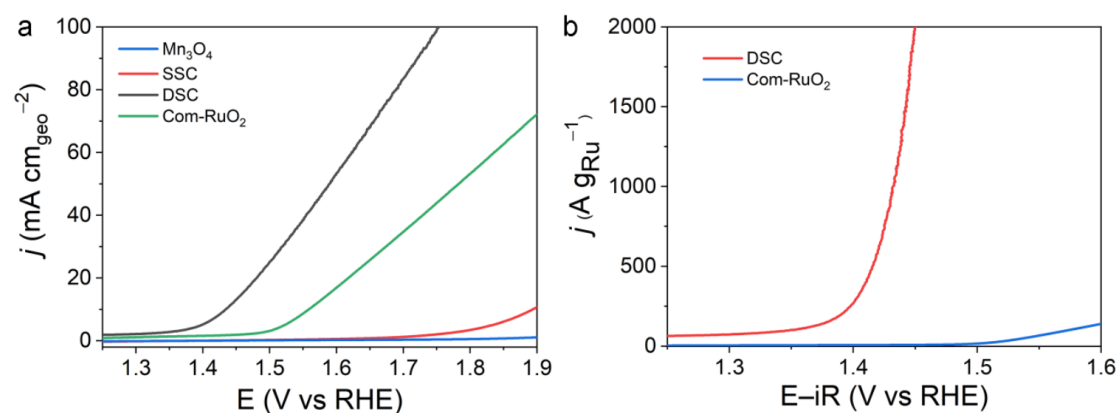


Figure S52. (a) Non-iR-corrected polarization curves of Mn₃O₄, SSC, DSC, and Com-RuO₂. The current densities were normalized to this area. (b) Polarization curves of DSC, and Com-RuO₂. The activity of each catalytic material was normalized to the mass of Ru.

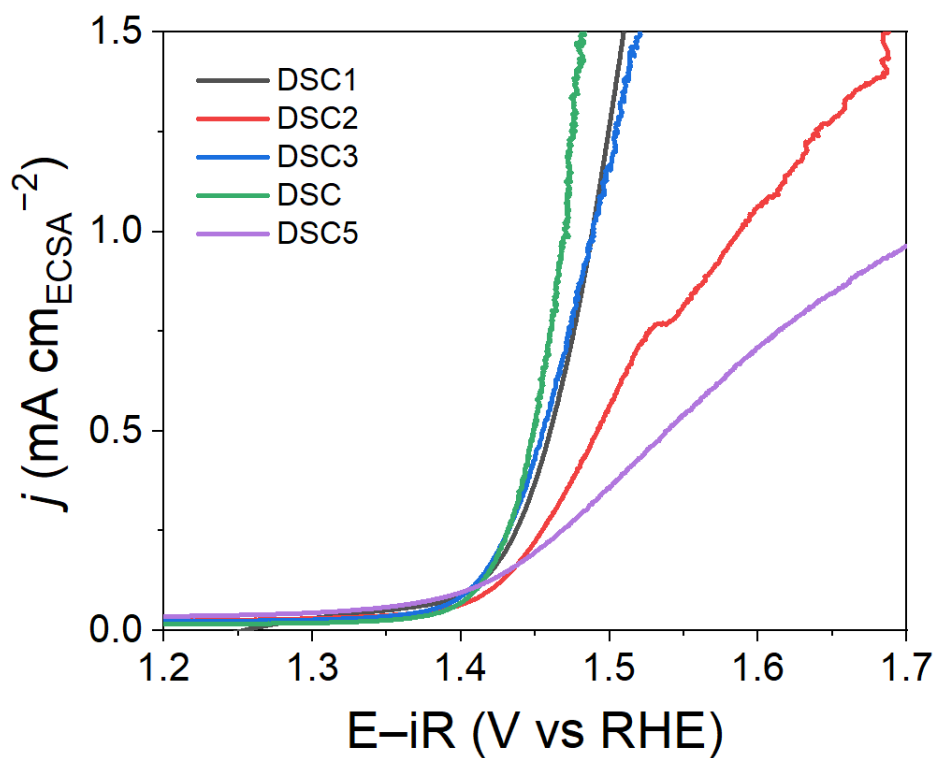


Figure S53. Polarization curves of DSC1, DSC2, DSC3, DSC, and DSC5. The activities of the catalytic materials were normalized to ECSA.

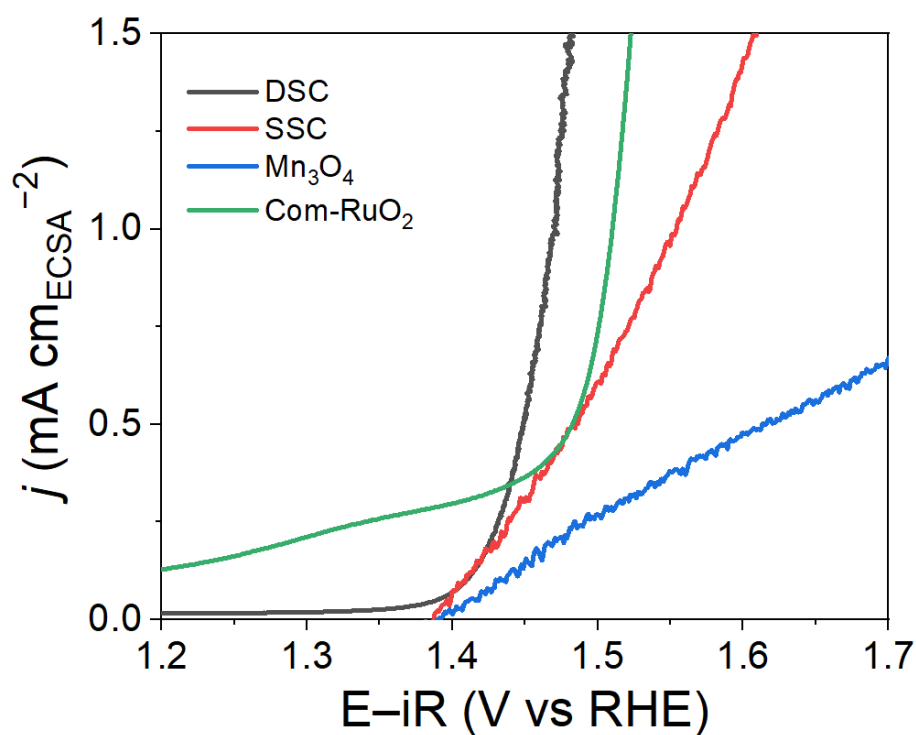


Figure S54. Polarization curves of Mn_3O_4 , SSC, DSC, and Com-RuO₂. The activities of the catalytic materials were normalized to ECSA.

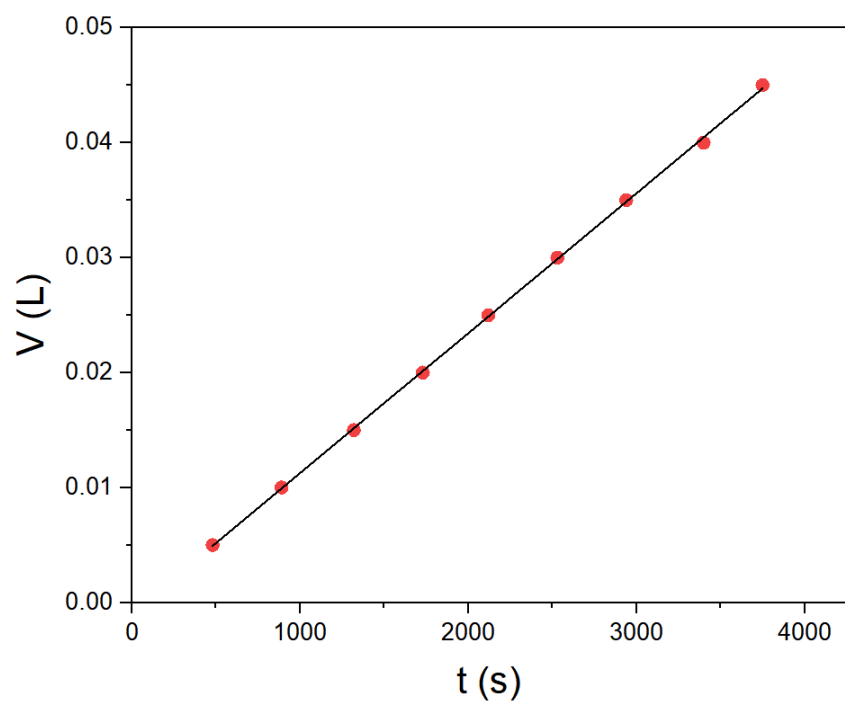


Figure S55. Linear relationship between the volume of evolved O_2 and electrolysis time during the OER process on the DSC catalyst, confirming nearly 100% Faradaic efficiency.

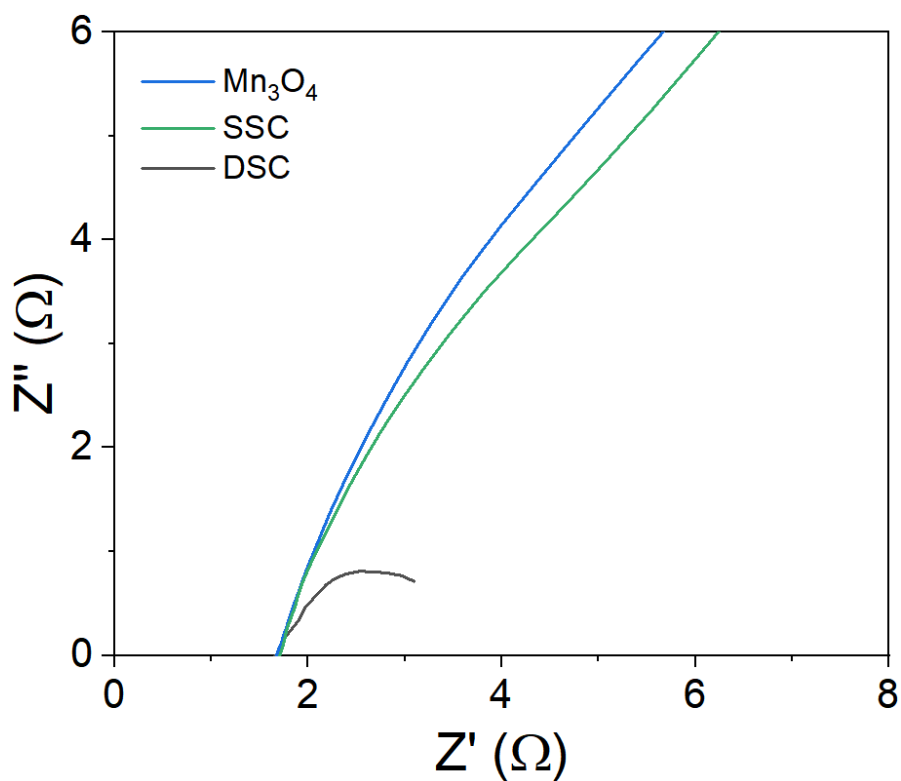


Figure S56. (a) EIS Nyquist plots of Mn_3O_4 , SSC, and DSC.

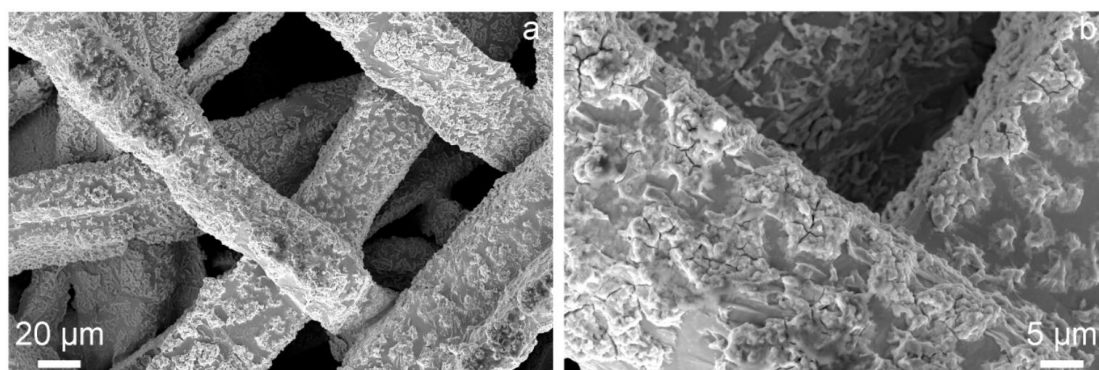


Figure S57. Low and high-resolution SEM images of the DSC after CP stability testing at $50 \text{ mA cm}_{\text{geo}}^{-2}$.

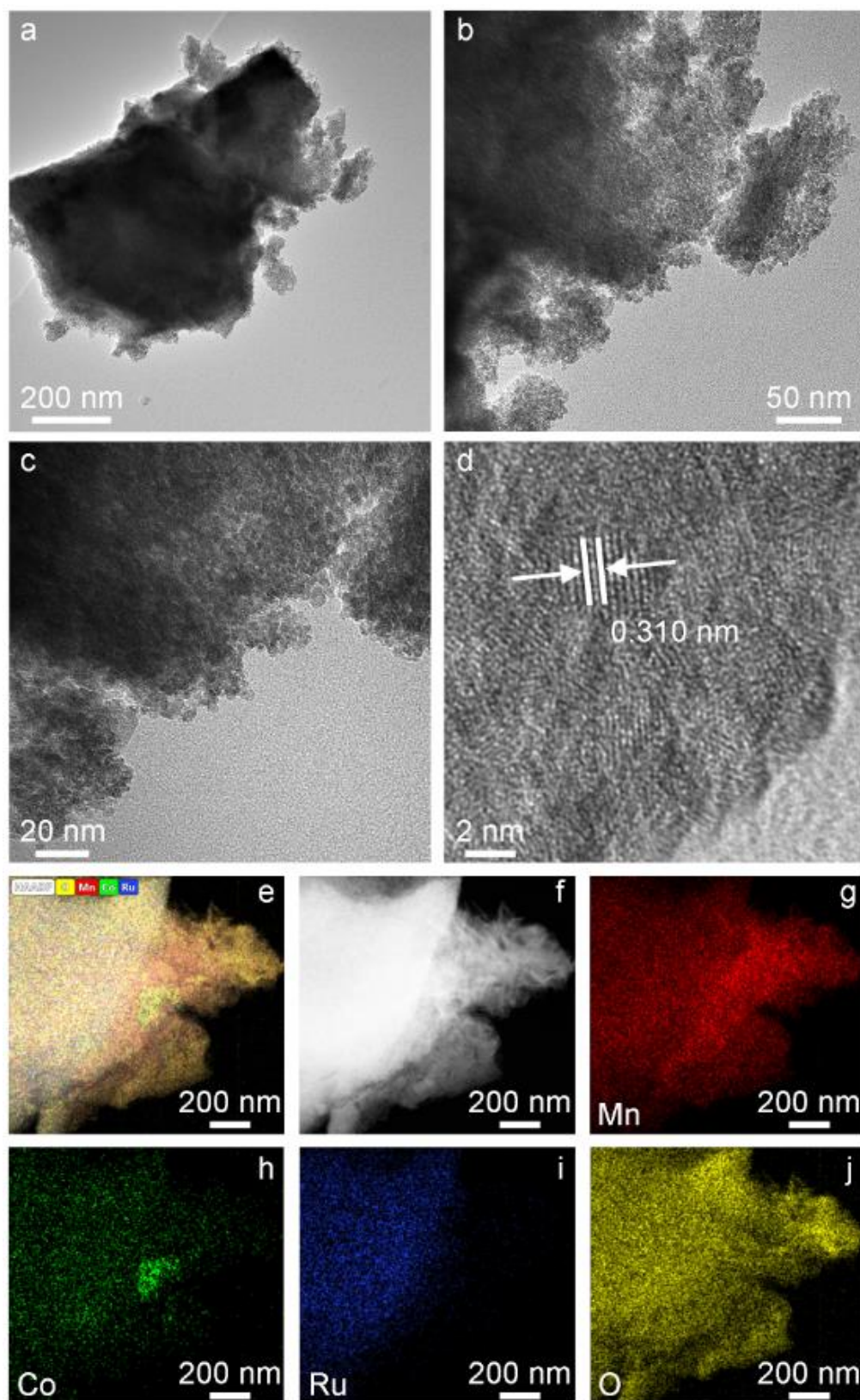


Figure S58. Low and high-resolution TEM and the corresponding elemental mapping images of the DSC after CP stability testing at $50 \text{ mA cm}_{\text{geo}}^{-2}$.

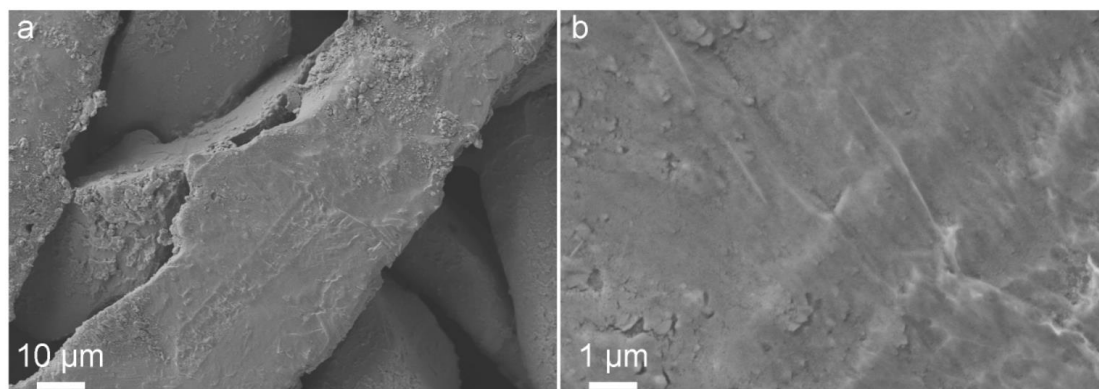


Figure S59. Low and high-resolution SEM images of the DSC after CP stability testing at $100 \text{ mA cm}_{\text{geo}}^{-2}$.

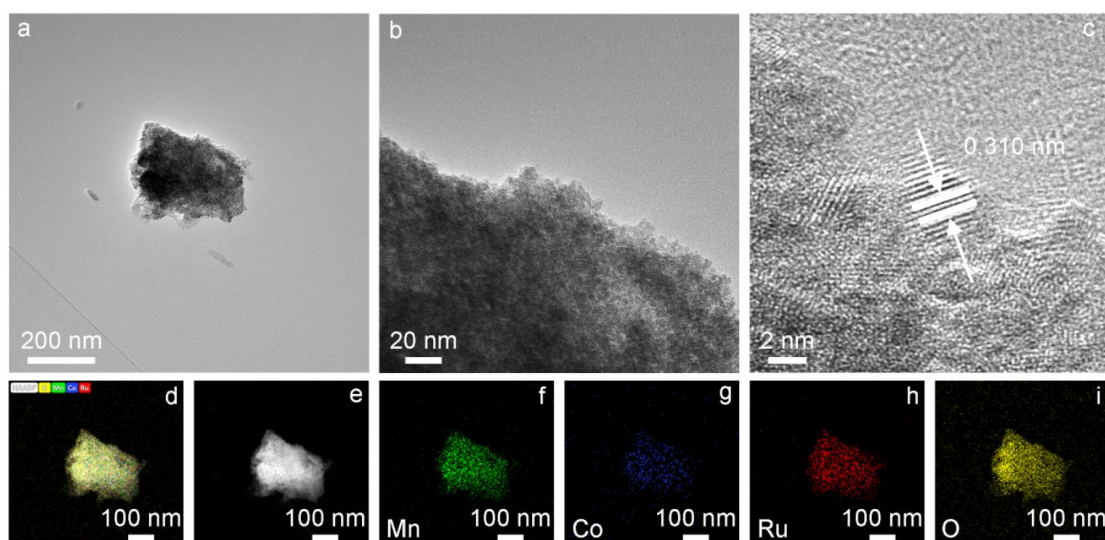


Figure S60. Low and high-resolution TEM and the corresponding elemental mapping images of the DSC after CP stability testing at $100 \text{ mA cm}_{\text{geo}}^{-2}$.

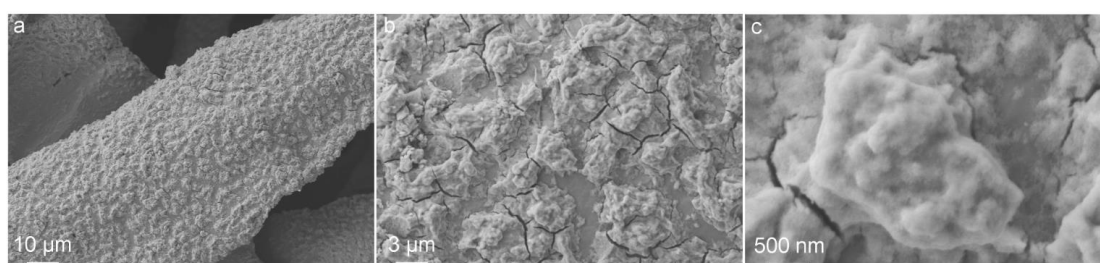


Figure S61. Low and high-resolution SEM images of the DSC after CP stability testing at $200 \text{ mA cm}_{\text{geo}}^{-2}$.

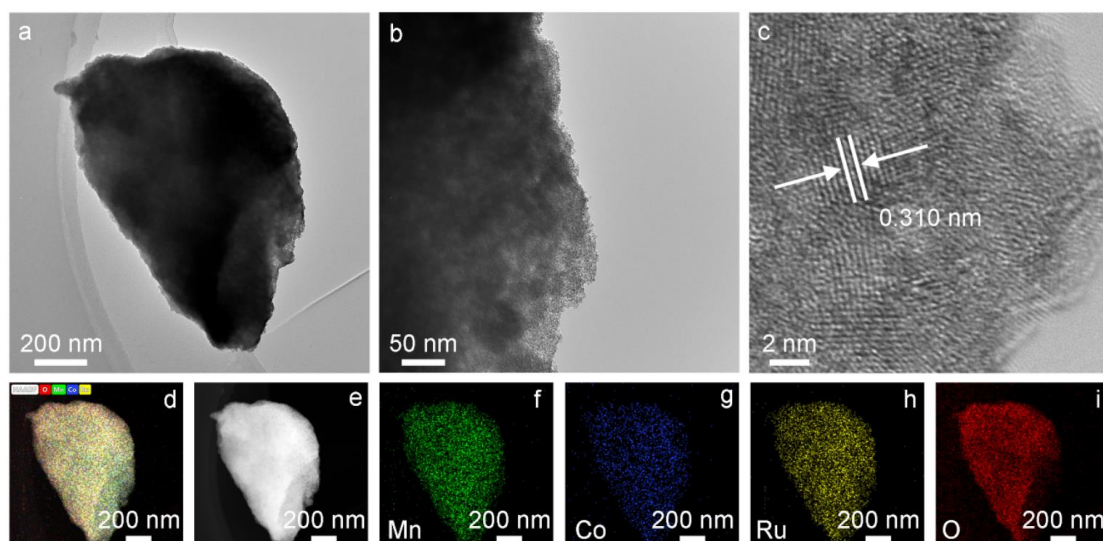


Figure S62. Low and high-resolution TEM and the corresponding elemental mapping images of the DSC after CP stability testing at $200 \text{ mA cm}_{\text{geo}}^{-2}$.

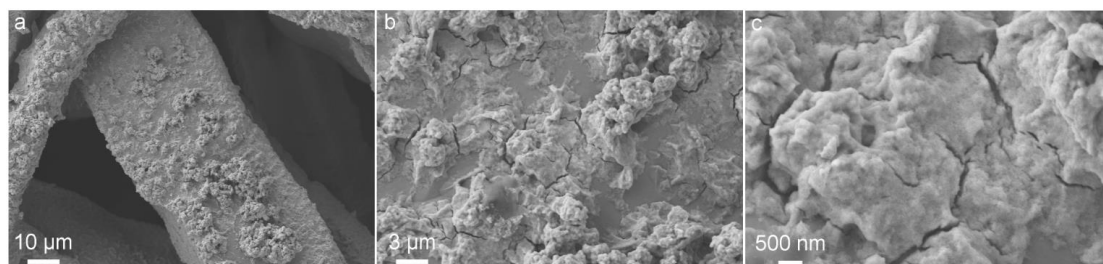


Figure S63. Low and high-resolution SEM images of the DSC after CP stability testing at $500 \text{ mA cm}_{\text{geo}}^{-2}$.

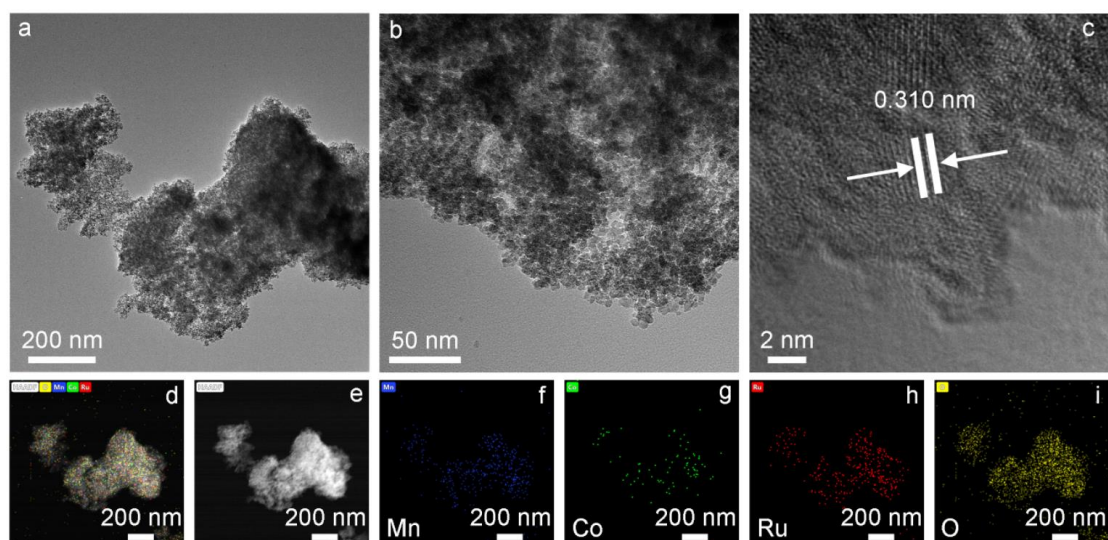


Figure S64. Low and high-resolution TEM and the corresponding elemental mapping images of the DSC after CP stability testing at $500 \text{ mA cm}_{\text{geo}}^{-2}$.

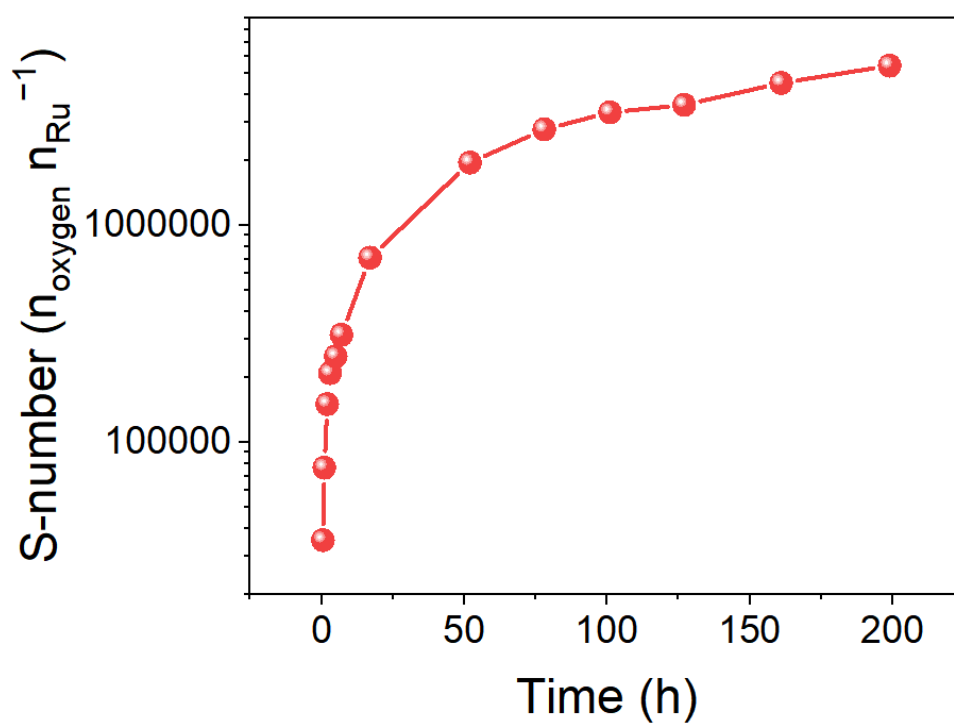


Figure S65. S-number of the DSC catalysts as a function of reaction time.

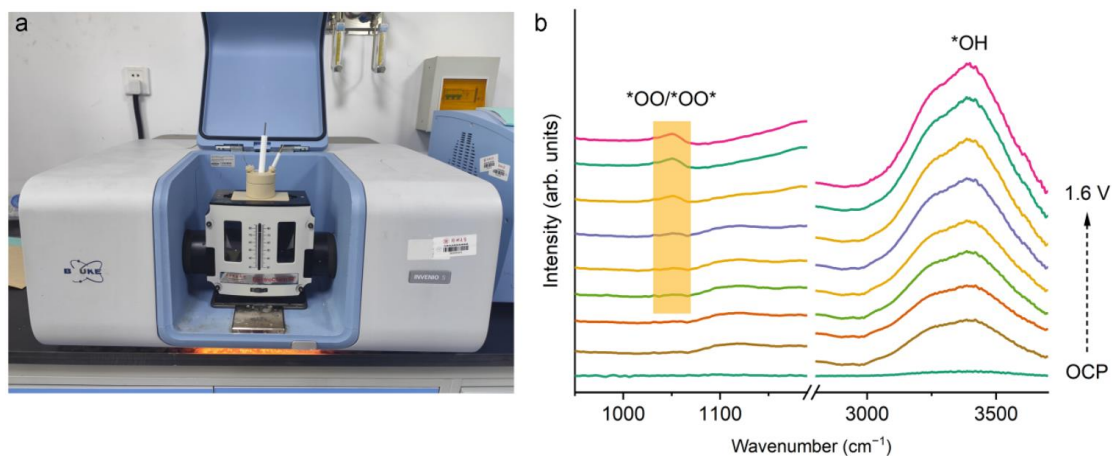


Figure S66. (a) Photograph of the custom-built in-situ ATR-SEIRAS setup. (b) In-situ ATR-SEIRAS spectra at various applied potentials for Mn_3O_4 . Catalytic sites are indicated by the symbol *.

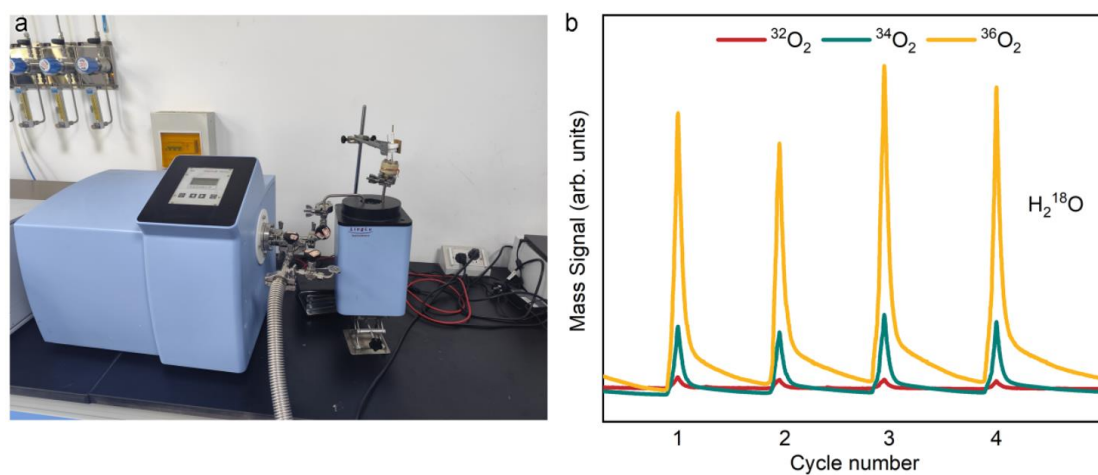


Figure S67. (a) Photograph of the custom-built in-situ DEMS setup. (b) DEMS signals of the O_2 product formed over Mn_3O_4 in 0.5 M H_2SO_4 electrolyte with H_2^{18}O as solvent.

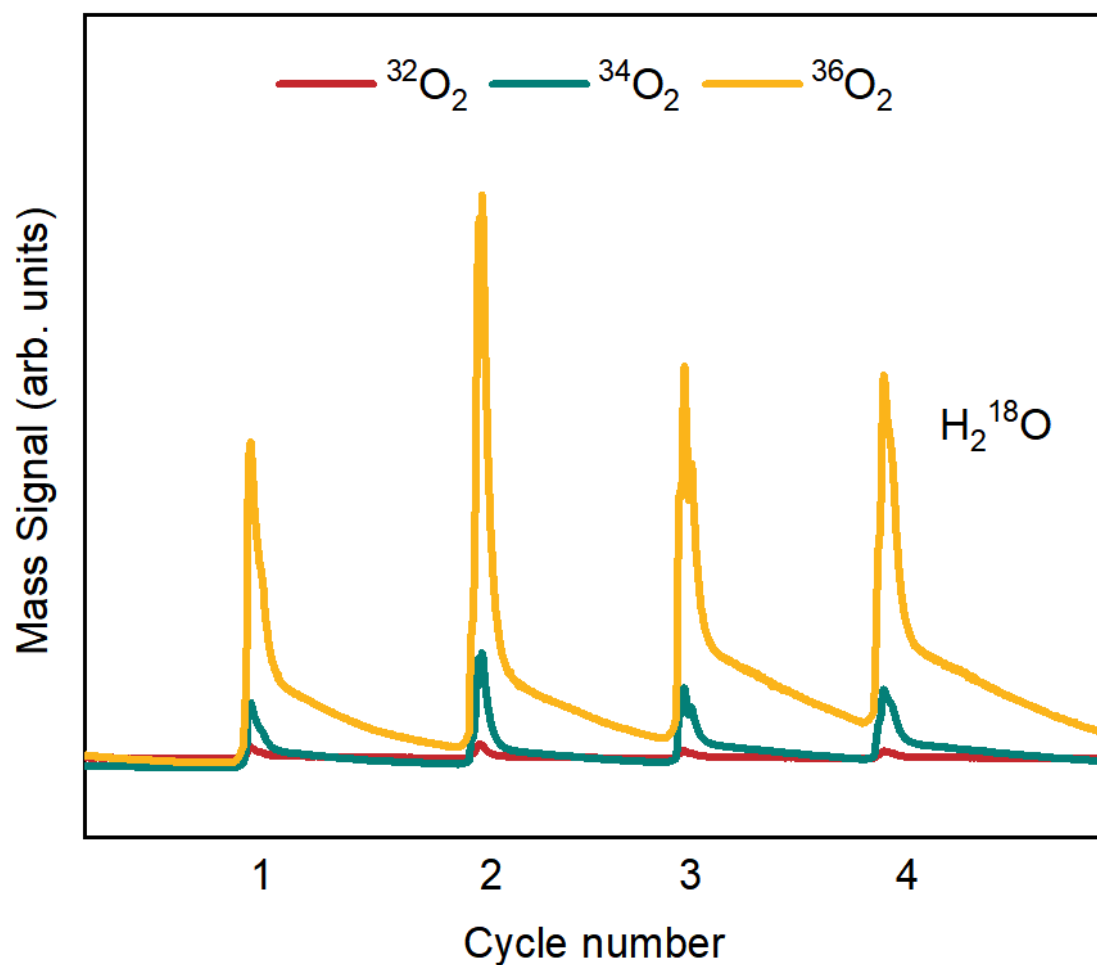


Figure S68. DEMS signals of the O_2 product formed over SSC in 0.5 M H_2SO_4 electrolyte with H_2^{18}O as solvent.

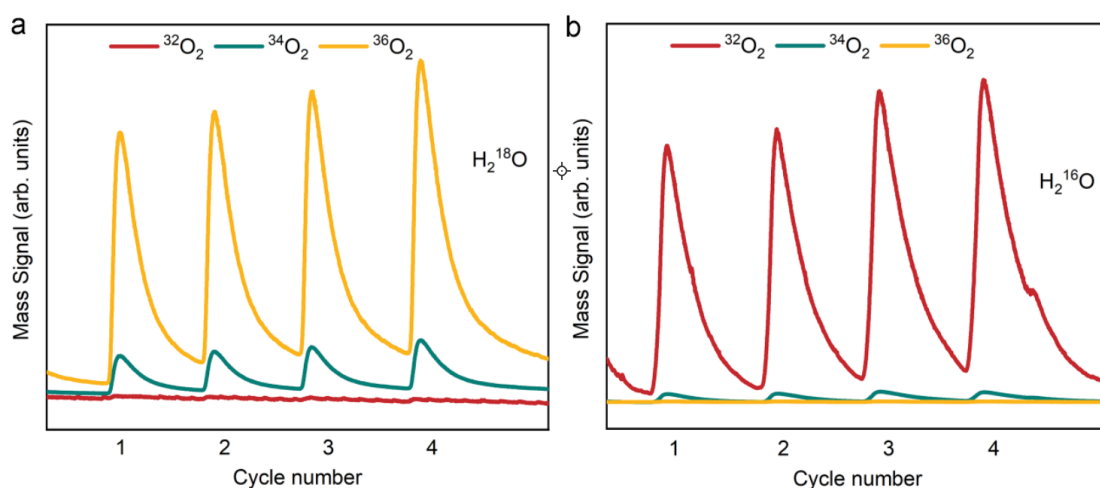


Figure S69. (a) DEMS signals of the O_2 product formed over DSC in 0.5 M H_2SO_4 electrolyte with H_2^{18}O as solvent. (b) DEMS signals of the O_2 product formed over Mn_3O_4 in 0.5 M H_2SO_4 electrolyte with H_2^{16}O as solvent.

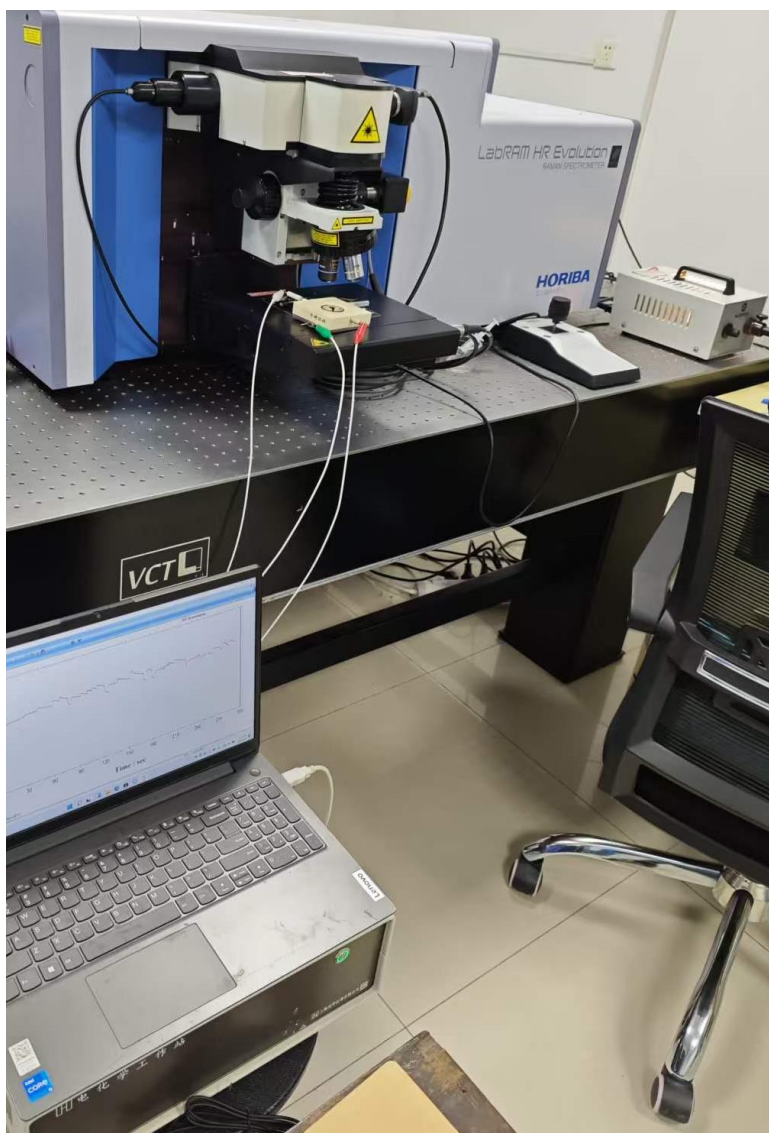


Figure S70. Photograph of the custom-built in situ electrochemical Raman spectroscopy setup.

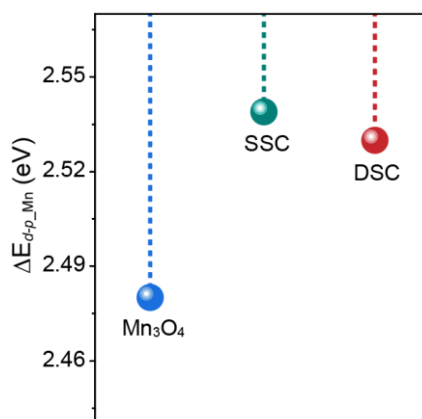


Figure S71. ΔE_{d-p} of Mn_3O_4 , SSC, and DSC. ΔE_{d-p_Mn} calculated from the ϵ_{d_Mn} and ϵ_p .

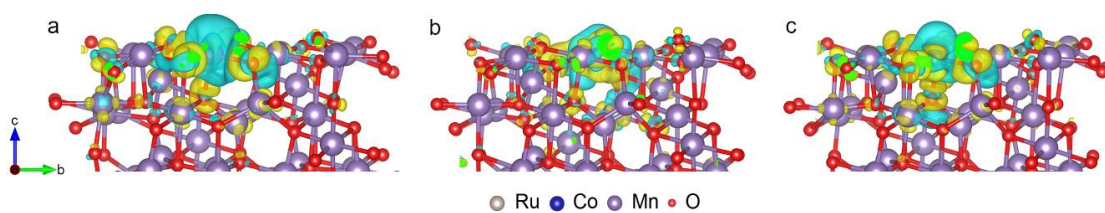


Figure S72. Charge density difference of Mn_3O_4 , SSC, and DSC. Yellow and blue iso-surfaces represent charge accumulation and depletion, respectively.

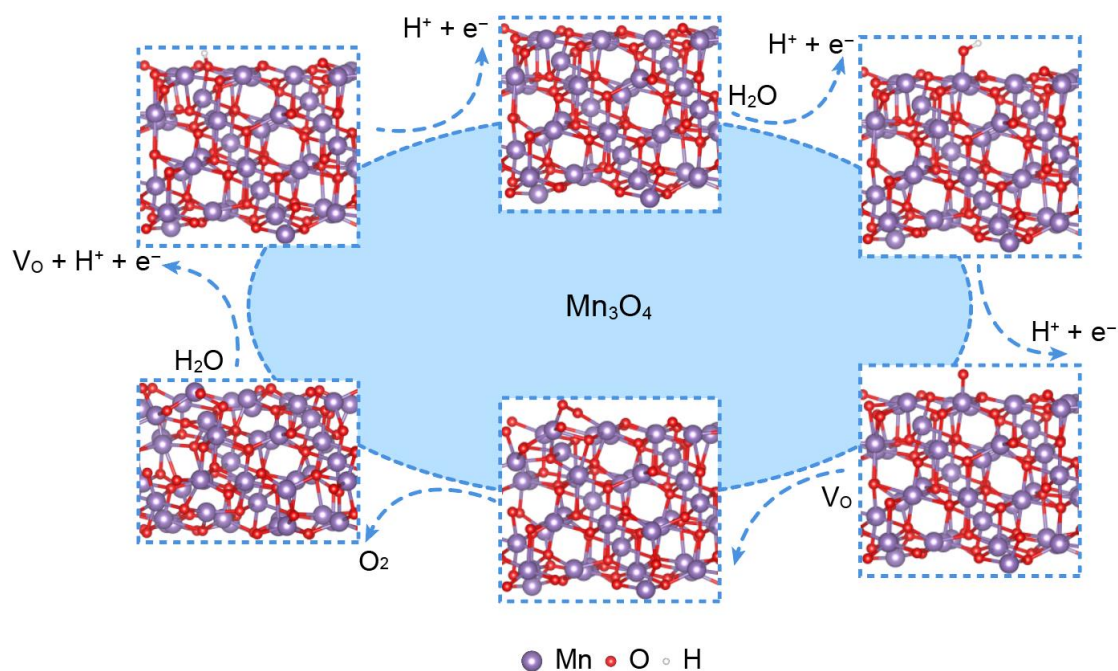


Figure S73. Schematic illustration of the LOM pathway for Mn_3O_4 .

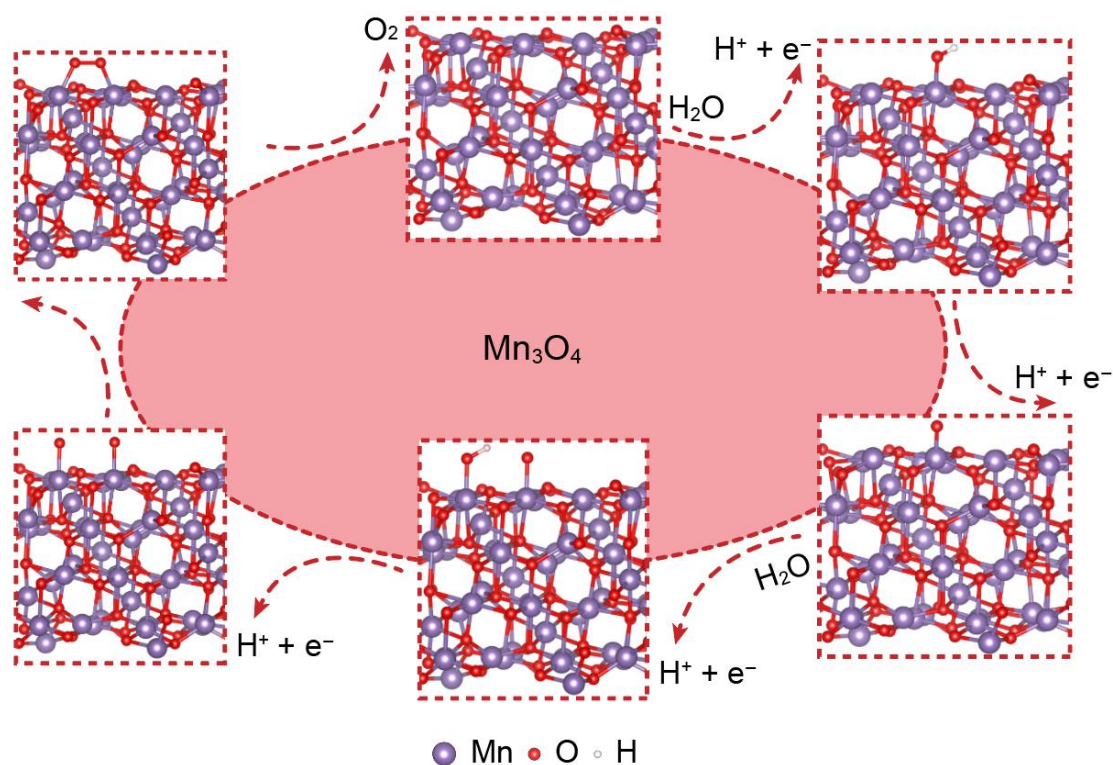


Figure S74. Schematic illustration of the DOM pathway for Mn_3O_4 .

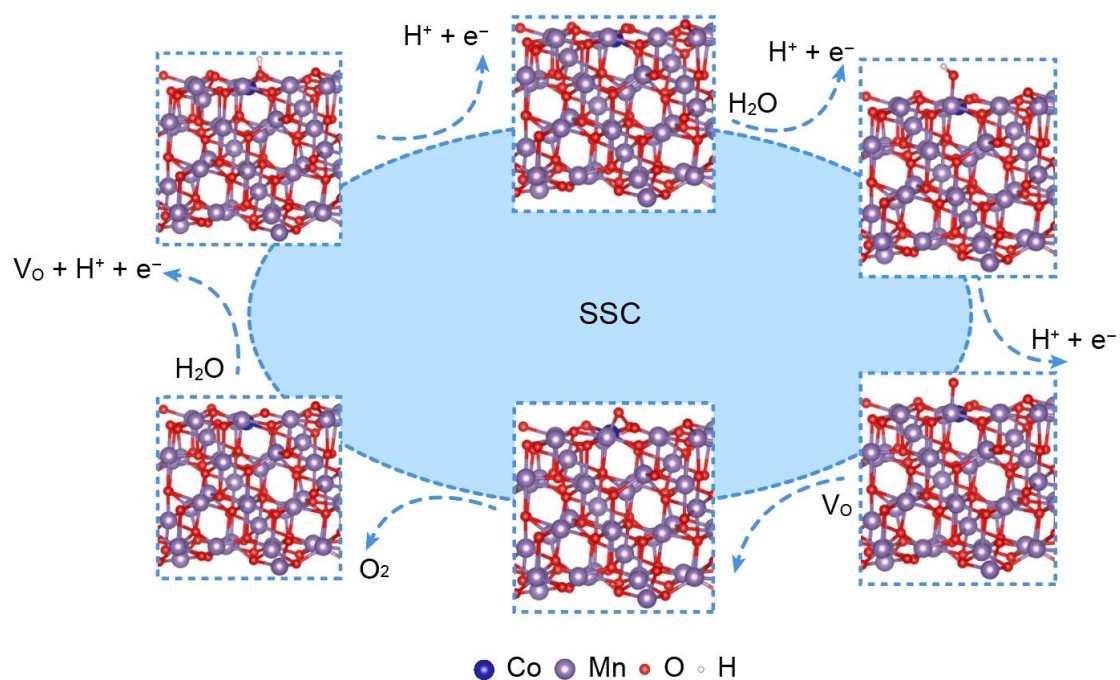


Figure S75. Schematic illustration of the LOM pathway for SSC.

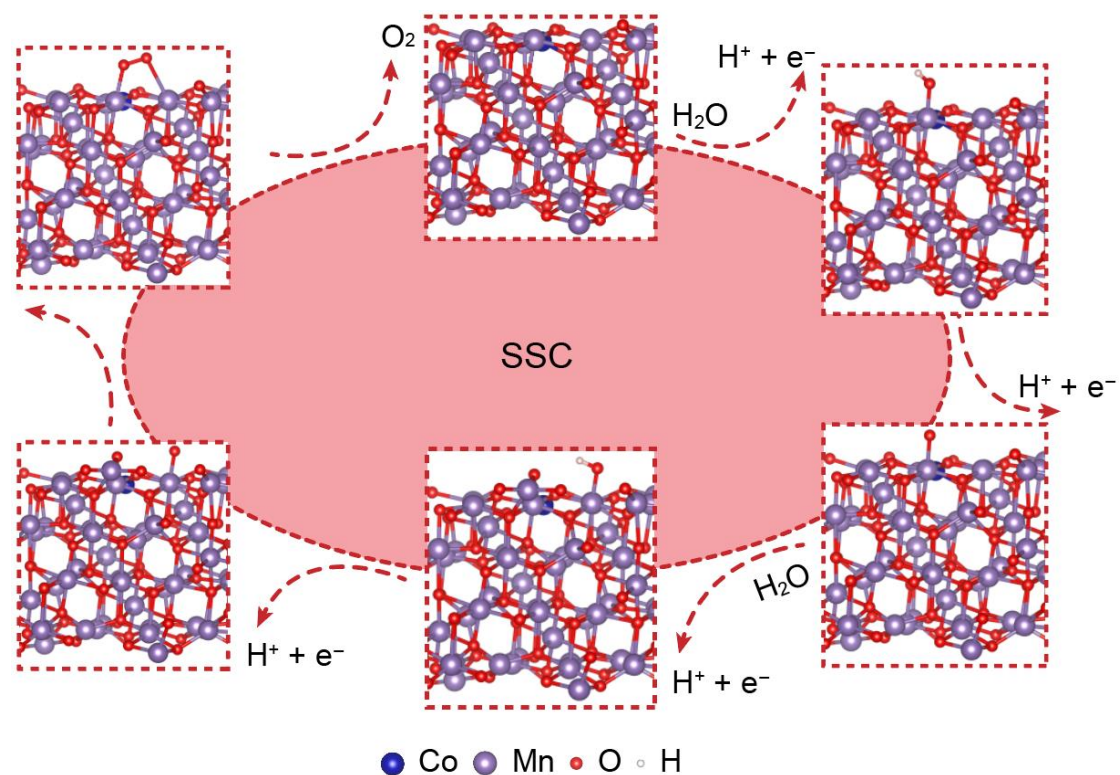


Figure S76. Schematic illustration of the DOM pathway for SSC.

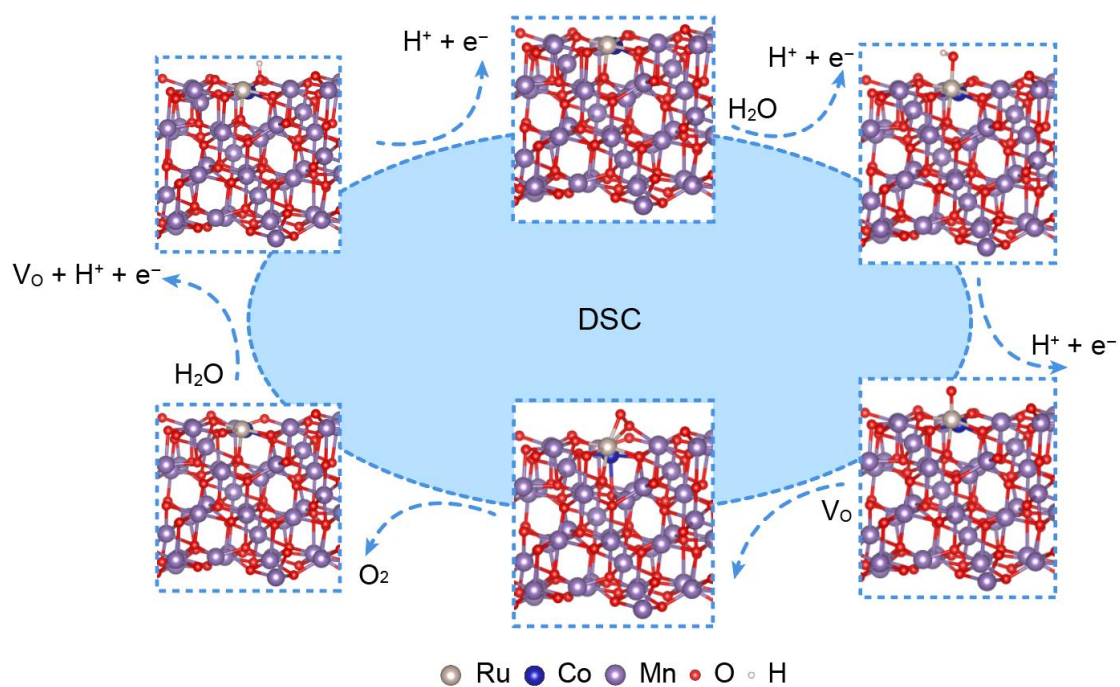


Figure S77. Schematic illustration of the LOM pathway for DSC.

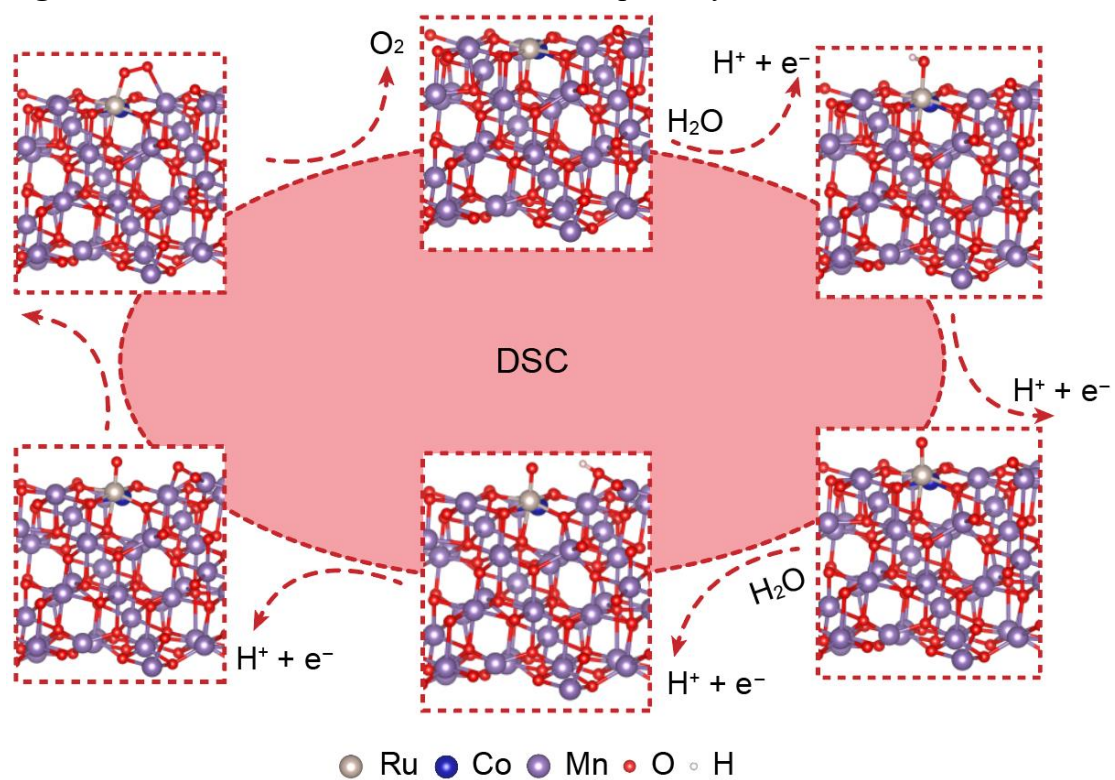


Figure S78. Schematic illustration of the DOM pathway for DSC.

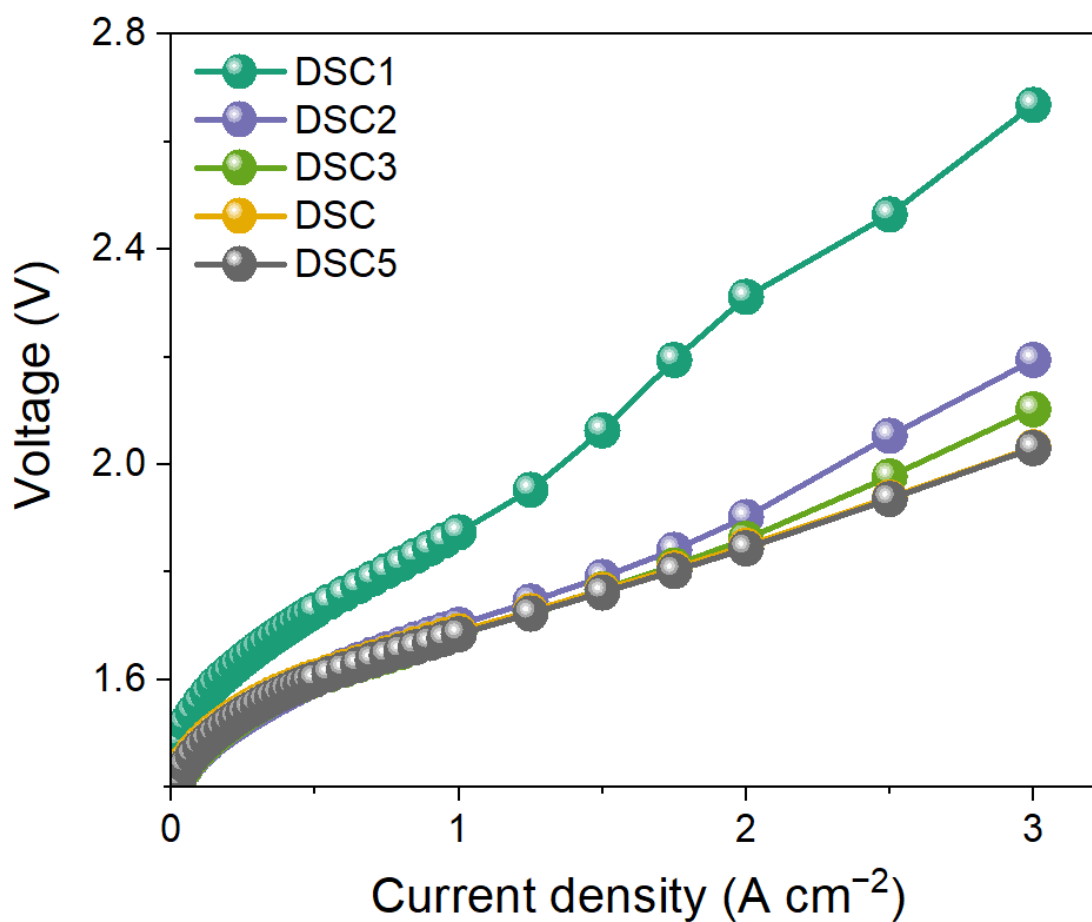


Figure S79. PEM-based polarization curves for DSC1, DSC2, DSC3, DSC, and DSC5 as anode materials, 40 wt% Pt/C as cathode material, and Nafion 115 as the membrane.

Table S1. The element contents (at.%) of Ru, Co, and Mn in catalysts that had undergone microwave-assisted alkali dissolution was detected by ICP-AES.

Sample	Ru ($\times 10^{-6}$ mol)	Co ($\times 10^{-6}$ mol)	Mn ($\times 10^{-6}$ mol)	ν ($n_{\text{Ru}}/n_{\text{Co}}$)
DSC1	0.500	1.187	6.133	0.42
DSC2	0.615	1.161	5.671	0.53
DSC3	0.843	1.155	4.711	0.73
DSC	1.186	1.151	4.309	1.03
DSC5	2.035	1.143	3.591	1.78

Table S2. EXAFS fitting parameters at the Co K-edge of DSC.

Shell	CN	R($\text{\AA}$)	$\sigma^2(10^{-3}\text{\AA}^2)$	R-factor
Co – O1	0.8	1.74	1.6	0.09
Co – O2	3.1	1.89	1.6	

CN, coordination number; R, the distance between absorber and backscatter atoms; σ^2 , Debye-Waller factor to account for both thermal and structural disorders; R-factor indicates the goodness of the fit; S_0^2 was fixed as 0.9.

Table S3. EXAFS fitting parameters at the Ru K-edge of DSC.

Shell	CN	R($\text{\AA}$)	$\sigma^2(10^{-3}\text{\AA}^2)$	R-factor
Ru – O	2.8	1.82	2.3	0.03

Ru – O 3.3 1.98 2.3

CN, coordination number; R, the distance between absorber and backscatter atoms; σ^2 , Debye-Waller factor to account for both thermal and structural disorders; R-factor indicates the goodness of the fit; S_0^2 was fixed as 1.0.

Table S4. Fitting parameters of XPS Mn 2p spectrum of Mn₃O₄.

No	Name	Position (eV)	FWHM (eV)	Lineshape	%At
1	Mn 2p _{3/2} -Peak	641.20	3.24	-	100%
2	Mn ²⁺ -Peak	641.21	2.02	GL(30)	33.34%
3	Mn ³⁺ -Peak	642.49	3.66	GL(30)	66.66%

Table S5. Fitting parameters of XPS Mn 2p spectrum of SSC.

No	Name	Position (eV)	FWHM (eV)	Lineshape	%At
1	Mn 2p _{3/2} -Peak	641.95	3.24	-	100%
2	Mn ²⁺ -Peak	641.24	2.14	GL(30)	28.58%
3	Mn ³⁺ -Peak	642.54	3.18	GL(30)	71.42%

Table S6. Fitting parameters of XPS Mn 2p spectrum of DSC.

No	Name	Position (eV)	FWHM (eV)	Lineshape	%At
1	Mn 2p _{3/2} -Peak	641.36	3.70	-	100%
2	Mn ²⁺ -Peak	641.16	3.08	GL(30)	34.49%
3	Mn ³⁺ -Peak	642.45	4.91	GL(30)	65.51%

Table S7. Fitting parameters of XPS Mn 2p spectrum of DSC1.

No	Name	Position (eV)	FWHM (eV)	Lineshape	%At
1	Mn 2p _{3/2} -Peak	641.61	3.32	-	100%
2	Mn ²⁺ -Peak	641.16	2.09	GL(30)	30.31%
3	Mn ³⁺ -Peak	642.45	4.42	GL(30)	69.69%

Table S8. Fitting parameters of XPS Mn 2p spectrum of DSC2.

No	Name	Position (eV)	FWHM (eV)	Lineshape	%At
1	Mn 2p _{3/2} -Peak	641.55	3.77	-	100%
2	Mn ²⁺ -Peak	641.17	2.56	GL(30)	32.26%
3	Mn ³⁺ -Peak	642.47	6.13	GL(30)	67.74%

Table S9. Fitting parameters of XPS Mn 2p spectrum of DSC3.

No	Name	Position (eV)	FWHM (eV)	Lineshape	%At
1	Mn 2p _{3/2} -Peak	641.41	3.48	-	100%
2	Mn ²⁺ -Peak	641.24	2.53	GL(30)	32.79%
3	Mn ³⁺ -Peak	642.54	5.03	GL(30)	67.21%

Table S10. Fitting parameters of XPS Co 2p spectrum of SSC.

No	Name	Position (eV)	FWHM (eV)	Lineshape	%At
1	Co ²⁺ -Peak	780.79	2.40	GL(30)	60.74%
2	Co ₂ ⁺ (Sat.)-Peak	784.79	7.45	GL(30)	39.26%

Table S11. Fitting parameters of XPS Co 2p spectrum of DSC1.

No	Name	Position (eV)	FWHM (eV)	Lineshape	%At
1	Co ²⁺ -Peak	780.79	2.39	GL(30)	59.46%
2	Co ₂ ⁺ (Sat.)-Peak	784.73	3.50	GL(30)	40.54%

Table S12. Fitting parameters of XPS Co 2p spectrum of DSC2.

No	Name	Position (eV)	FWHM (eV)	Lineshape	%At
1	Co ²⁺ -Peak	780.84	3.03	GL(30)	77.62%
2	Co ₂ ⁺ (Sat.)-Peak	784.99	3.50	GL(30)	22.38%

Table S13. Fitting parameters of XPS Co 2p spectrum of DSC3.

No	Name	Position (eV)	FWHM (eV)	Lineshape	%At
1	Co ²⁺ -Peak	780.80	3.37	GL(30)	63.23%
2	Co ₂ ⁺ (Sat.)-Peak	784.87	2.50	GL(30)	36.77%

Table S14. Fitting parameters of XPS Co 2p spectrum of DSC.

No	Name	Position (eV)	FWHM (eV)	Lineshape	%At
1	Co ²⁺ -Peak	780.82	2.78	GL(30)	60.58%
2	Co ₂ ⁺ (Sat.)-Peak	784.66	3.50	GL(30)	39.42%

Table S15. Fitting parameters of XPS C 1s and Ru 3d spectrum of SSC.

No	Name	Position (eV)	FWHM (eV)	Lineshape	%At
1	C-C-Peak	284.80	1.31	GL(30)	66.85%

Table S16. Fitting parameters of XPS C 1s and Ru 3d spectra of DSC1.

No	Name	Position (eV)	FWHM (eV)	Lineshape	%At
1	C-C-Peak	284.80	1.49	GL(30)	50.03%
2	Ru 3d _{5/2} -Peak	281.08	1.01	GL(30)	5.9%
3	Ru 3d _{3/2} -Peak	285.28	1.01	GL(30)	3.95%

Table S17. Fitting parameters of XPS C 1s and Ru 3d spectra of DSC2.

No	Name	Position (eV)	FWHM (eV)	Lineshape	%At
1	C-C-Peak	284.80	1.35	GL(30)	4.85%
2	Ru 3d _{5/2} -Peak	281.09	1.23	GL(30)	19.78%
3	Ru 3d _{3/2} -Peak	285.29	1.23	GL(30)	13.26%

Table S18. Fitting parameters of XPS C 1s and Ru 3d spectra of DSC3.

No	Name	Position (eV)	FWHM (eV)	Lineshape	%At
1	C-C-Peak	284.80	2.21	GL(30)	6.17%
2	Ru 3d _{5/2} -Peak	281.11	1.26	GL(30)	22.46%
3	Ru 3d _{3/2} -Peak	285.31	1.26	GL(30)	15.05%

Table S19. Fitting parameters of XPS C 1s and Ru 3d spectra of DSC.

No	Name	Position (eV)	FWHM (eV)	Lineshape	%At
1	C-C-Peak	284.80	1.86	GL(30)	15.54%
2	Ru 3d _{5/2} -Peak	281.12	1.00	GL(30)	15.03%
3	Ru 3d _{3/2} -Peak	285.32	1.00	GL(30)	10.07%

Table S20. Comparison of acidic OER performance in a three-electrode system with previously reported Ru-based electrocatalysts.

Sample	Electrolyte	η (10 mA cm _{geo} ⁻²)	Stability (h)@10mA cm _{geo} ⁻²	Catalyst loading amounts (mg cm _{geo} ⁻²)	Ref.
DSC	0.5 M H ₂ SO ₄	175	2100@50	0.06	This work
NiRuCoO _x	0.1M HClO ₄	166	105	0.038	1
IrO _x /9R-BaIrO ₃	0.5 M H ₂ SO ₄	230	50	0.28	2
Ru-N-C	0.5 M H ₂ SO ₄	267	30	0.28	3
Ir _{0.06} Co _{2.94} O ₄	0.1M HClO ₄	300	45	-	4
Ni-RuO ₂	0.1 M HClO ₄	214	200	0.40	5
Re _{0.06} Ru _{0.94} O ₂	0.1 M HClO ₄	190	200	0.20	6

W _{0.2} Er _{0.1} Ru _{0.7} O _{2-δ}	0.5 M H ₂ SO ₄	168	500	0.33	7
Co-Ru@RuO ₂	0.5 M H ₂ SO ₄	203	420	0.51	8
Ru-Mn ₃ O ₄	0.5 M H ₂ SO ₄	176	8800@50	0.07	9
Ru-Co ₂ MnO _{4.5}	0.5 M H ₂ SO ₄	176	600	-	10

Table S21. Comparison of the S-number with previously reported electrocatalysts.

Sample	S-number	Ref.
DSC	5456184	This work
IrO ₂ @TaB ₂	52000	11
IrO ₂	29000	11
RuCoO _x	31774	12
Si-RuO ₂ -0.1	34200	13
py-RuO ₂ :Zn	60000	14

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