**Supporting Information**

**Metal Doped Layered MgB2 Nanoparticles as Novel Electrocatalysts for Water Splitting**

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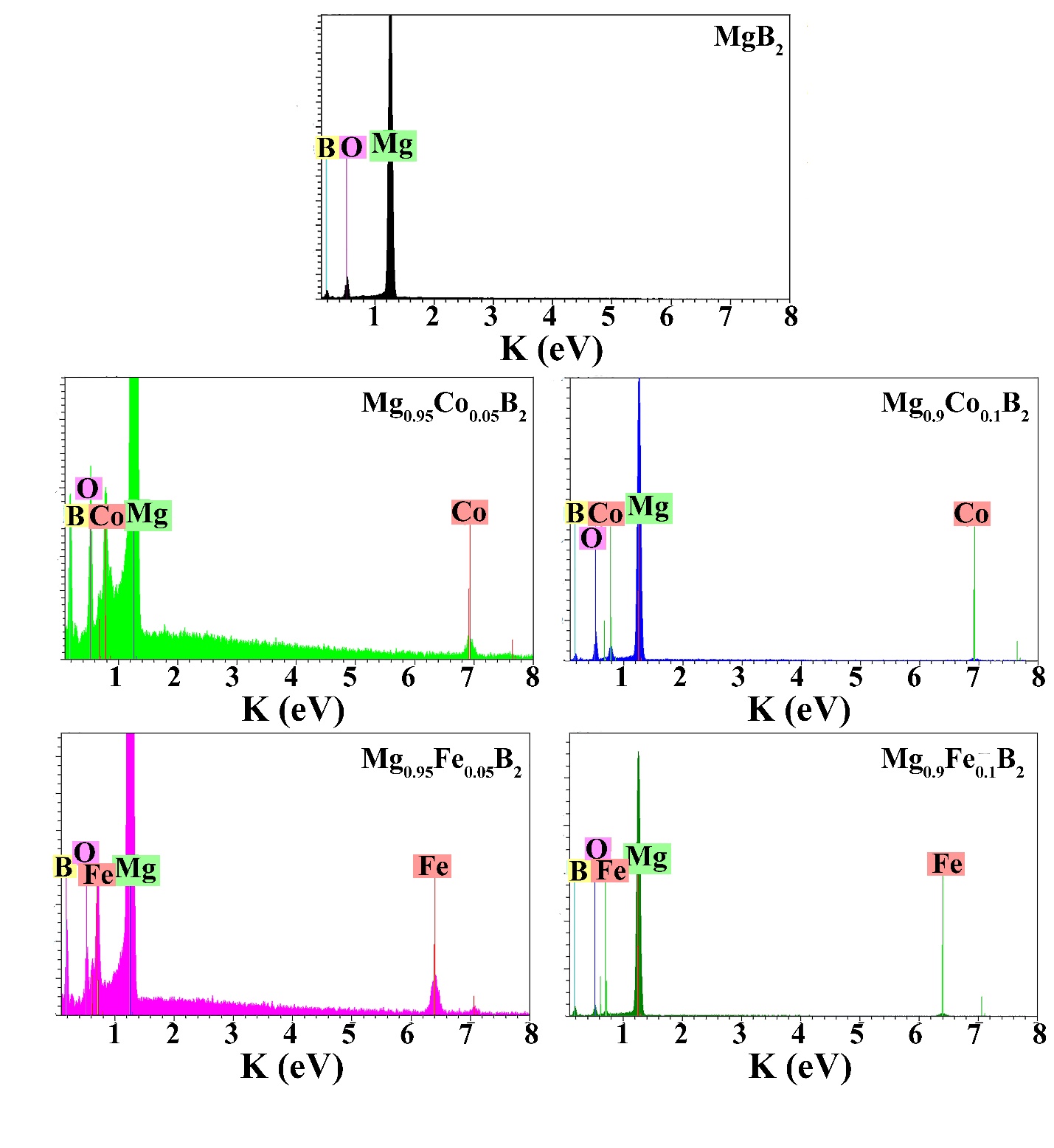
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***SEM EDX Analysis Data Before HER/OER***

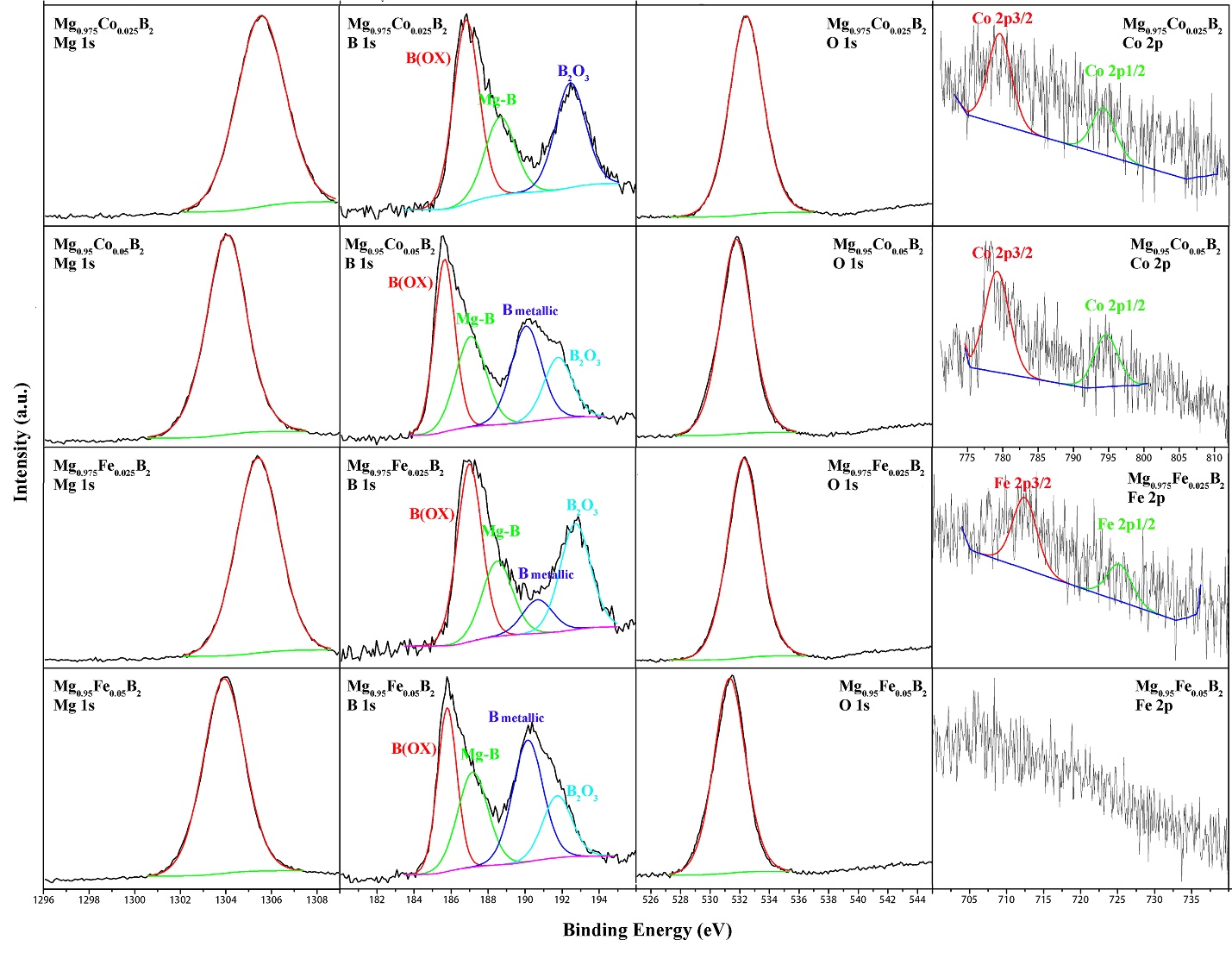


**Figure S1.** EDX analysis graph of pure MgB2 and Mg1-*xTmx*B2 (*x* = 0.05, and 0.1; *Tm* = Fe and Co) before electrochemical reactions.

**Table S1.** Energy-dispersive X-ray spectroscopy (EDX) analysis of samples before HER and OER.

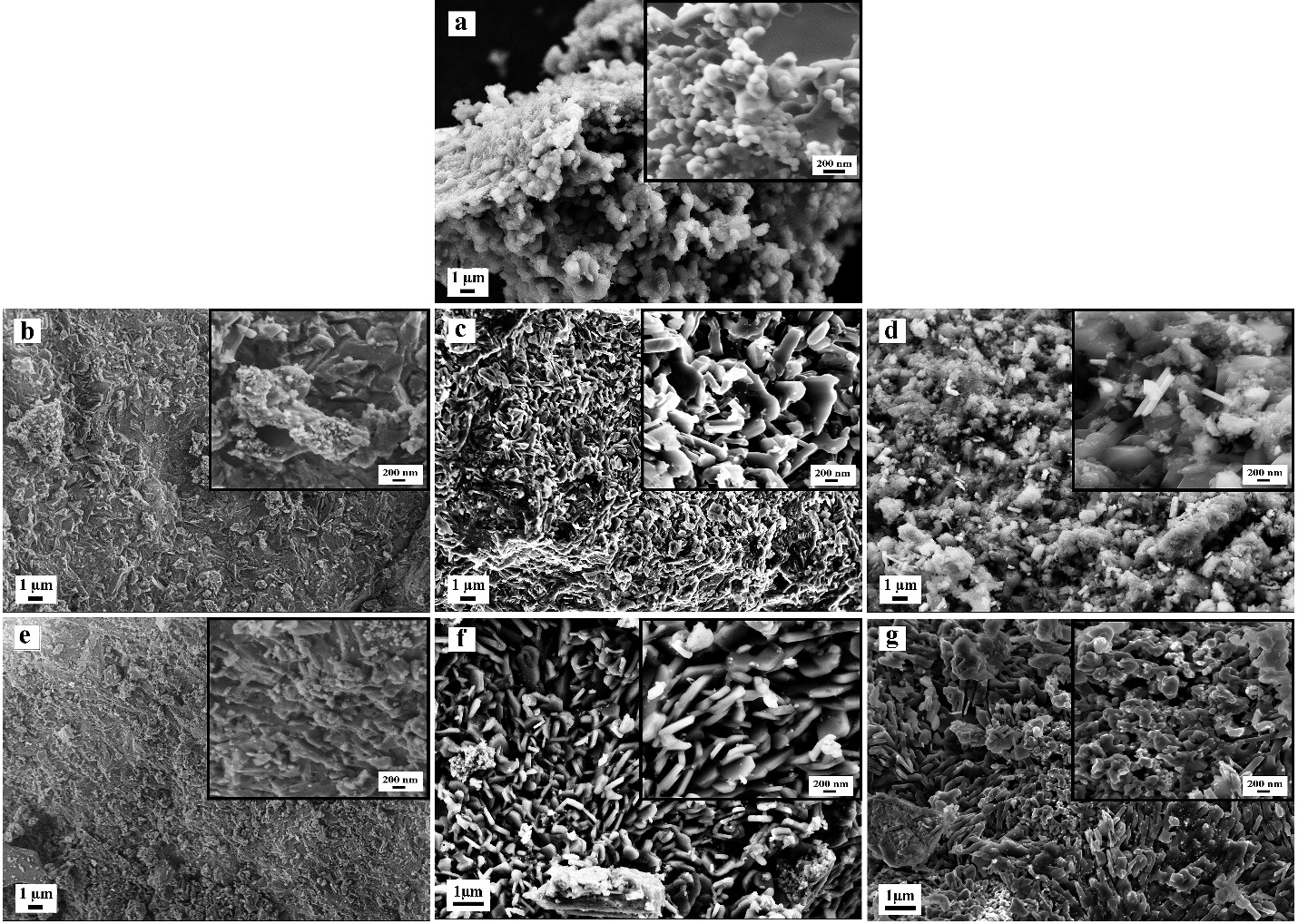
|  |  |  |  |
| --- | --- | --- | --- |
| Sample | Element | atomic % | atomic % |
| MgB2 | Mg  B  O | 30.51  69.49  - | 29.28  63.26  7.46 |
| Mg0.95Fe0.05B2 | Mg  Fe  B  O | 26.65  1.32  72.03  - | 26.12  1.29  69.58  3.01 |
| Mg0.95Co0.05B2 | Mg  Co  B  O | 26.22  1.59  72.19  - | 25.62  1.55  69.20  3.63 |
| Mg0.9Fe0.1B2 | Mg  Fe  B  O | 27.20  2.31  70.49  - | 26.63  2.26  67.99  3.12 |
| Mg0.9Co0.1B2 | Mg  Co  B  O | 31.45  3.23  65.32  - | 29.54  3.00  58.40  9.06 |

***XPS Spectra***



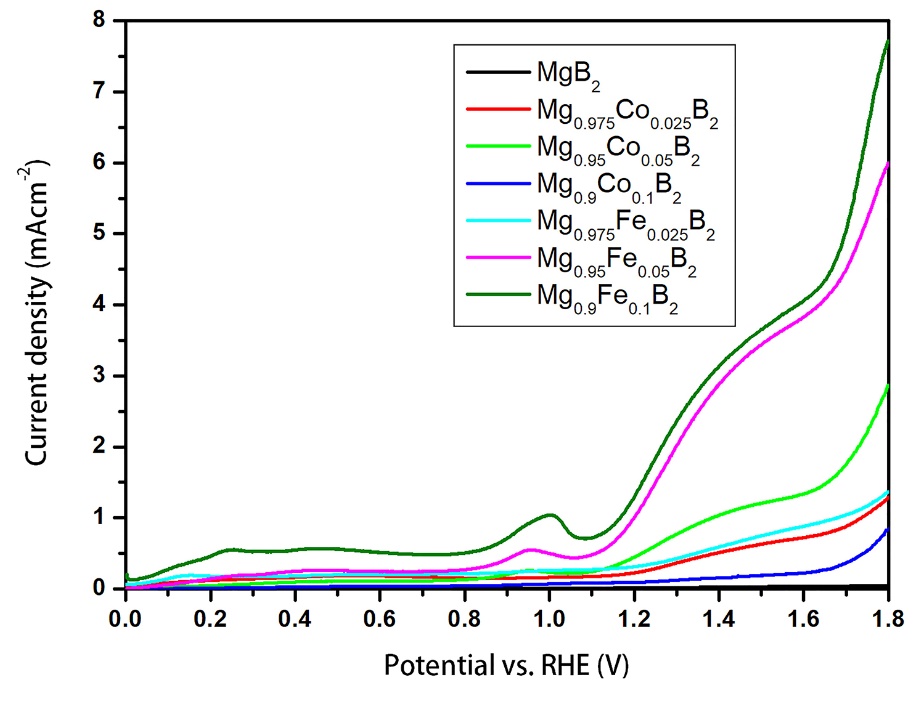
**Figure S2.** XPS spectra of Mg 1s, B 1s, O 1s, Co 2p, and F e 2p in Mg0.975Co0.025B2, Mg0.95Co0.05B2, Mg0.975Fe0.025B2, and Mg0.95Fe0.05B2.

***SEM Images***



**Figure S3.** SEM images of a) pure MgB2, b) Mg0.975Co0.025B2, c) Mg0.95Co0.05B2, d) Mg0.9Co0.1B2, e) Mg0.975Fe0.025B2, f) ‎Mg0.95Fe0.05B2, and g) Mg0.9Fe0.1B2‎

***OER Plots***



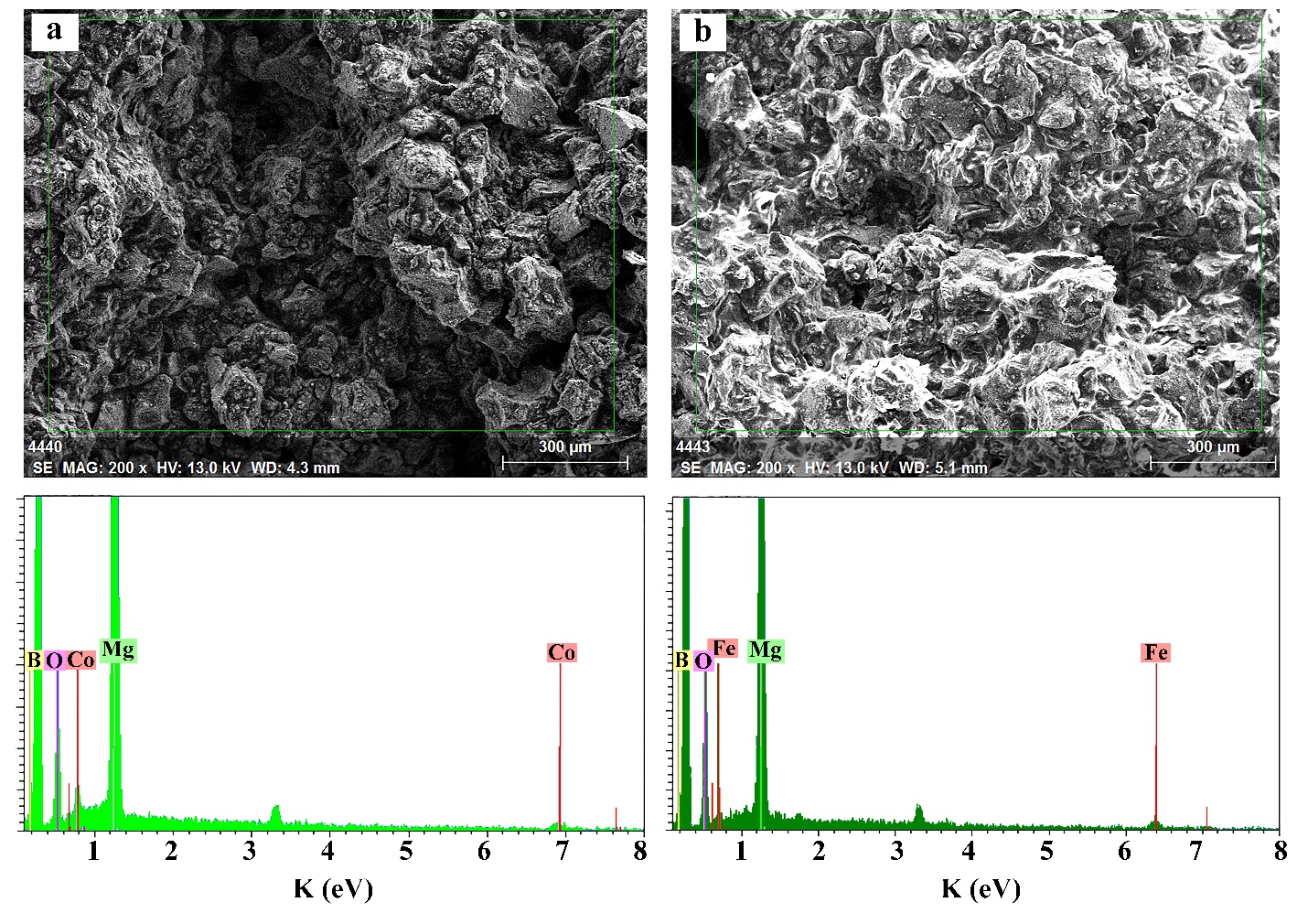
**Figure S4.** Polarization curves of the OER reaction of pure and doped MgB2.

***EIS Data***

**Table S2.** Equivalent-circuit element values for EIS data in the 1 M KOH electrolyte.

|  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- |
|  | **Re (Ωcm-2)** | **R1**  **(kΩ cm-2)** | **CPE1-T**  **(Fsp-1**  **cm-2)** | **CPE1-P** | **R2**  **(****kΩ cm-2)** | **CPE2-T**  **(Fsp-1**  **cm-2)** | **CPE2-P** |
| MgB2 | 130.5 | 10.993 | 2.5E-05 | 0.43 | 127.114 | 5.1E-05 | 0.56 |
| Mg0.975Co0.025B2 | 12.2 | 1.436 | 4.5E-04 | 0.47 | 18.331 | 2.4E-04 | 0.72 |
| Mg0.95Co0.05B2 | 13.6 | 0.409 | 1.3E-03 | 0.45 | 10.409 | 5.1E-04 | 0.66 |
| Mg0.9Co0.1B2 | 13.2 | 3.539 | 3.4E-04 | 0.45 | 13.712 | 2.7E-04 | 0.71 |
| Mg0.975Fe0.025B2 | 7.52 | 0.454 | 2.7E-04 | 0.36 | 710 | 1.5E-05 | 0.56 |
| Mg0.95Fe0.05B2 | 13.5 | 0.277 | 3.4E-03 | 0.40 | 14.325 | 6.2E-04 | 0.67 |
| Mg0.9Fe0.1B2 | 11.9 | 0.332 | 1.6E-03 | 0.45 | 7.980 | 1.2E-03 | 0.62 |

***SEM EDX Analysis Data After HER/OER***



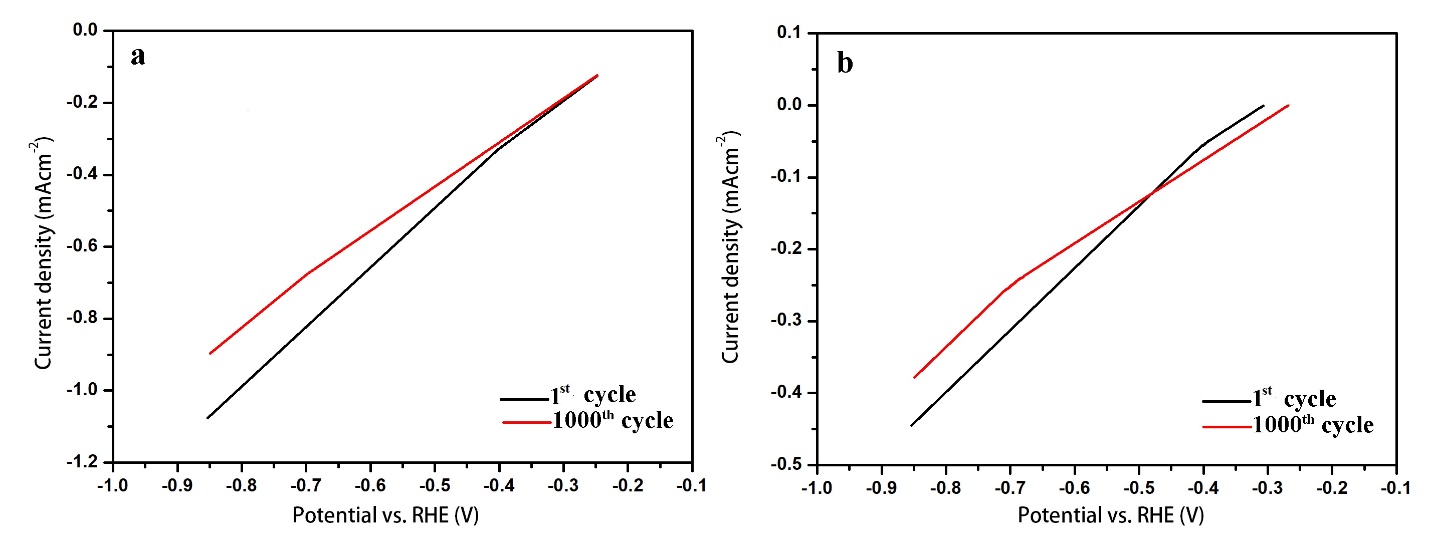
**Figure S5.** EDX results of the (a) Mg0.95Co0.05B‎2 and (b) Mg0.9Fe0.1B2 after electrochemical reactions.

**Table S3.** Energy-dispersive X-ray spectroscopy (EDX) analysis of the best-performing samples after HER and OER.

|  |  |  |  |
| --- | --- | --- | --- |
| Sample | Element | atomic % | atomic % |
| Mg0.95Co0.05B2 | Mg  Co  B  O | 25.29  1.19  73.52  - | 23.65  1.09  64.68  10.59 |
| Mg0.9Fe0.1B2 | Mg  Fe  B  O | 21.71  1.64  76.65  - | 19.59  1.48  64.00  14.93 |

***CV Stability Tests***

Aside from catalytic activity, stability is another influential criterion. To appraise the cycling durability of the Mg0.95Co0.05B2 and Mg0.9Fe0.1B2 electrodes, the cyclic voltammetry (CV) plots were measured within a potential range of -1 and -0.2 V for 1000 cycles at a scan rate of 50 mV s-1. Fig. S6a and b demonstrate the reordered CV curves of Mg0.95Co0.05B2 and Mg0.9Fe0.1B2 electrodes for the 1st and 1000th cycles in 1 M KOH electrolyte. As shown in the figure, it is obvious that after 1000 cycles the current density diminished for both electrodes. Even though the current density did not decrease appreciably, yet further investigations are needed to excel in the stability of these very new electrocatalysts.



**Figure S6.** Electrochemical cycling stability measurement. a) and b) CV curves of Mg0.95Co0.05B2 and Mg0.9Fe0.1B2, respectively, for 1st and 1000th cycles in a 1 M KOH electrolyte at a scan rate of 50 mV s−1.