Supporting Information for

Post Cobalt Doping and Defect Engineering of NbSSe for Efficient Hydrogen Evolution Reaction

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Fig. S15 XRD patterns of NbSSe-Co_{0.1} before and after 5000 cycles.



Fig. S1 (a) Optical image of the single crystal flakes of NbSSe (b) Schematic crystal

structure of NbSSe.



Fig. S2 (a) XRD patterns of NbSSe with added standard sample (Si) and NbSSe-Co_{0.1} with added standard sample (Si) (b-c) the partially enlarged XRD patterns of (a).



Fig. S3 (a) SEM image of NbSSe. (b-d) element mapping images of Nb, S and Se, respectively. (e) TEM image of NbSSe. (f) The HRTEM image of NbSSe. Inset shows the selected area electron diffraction image of NbSSe.



Fig. S4 SEM-EDAX results of the NbSSe-Co_{0.1}



Fig. S5 SEM-EDAX results of the NbSSe.



Fig. S6 SEM-EDAX results of the NbSSe-Co_{0.02.}



Fig. S7 SEM-EDAX results of the NbSSe-Co_{0.05.}



Fig. S8 SEM-EDAX results of the NbSSe-Co_{0.15.}



Fig. S9 XPS spectra of NbSSe (a) XPS survey spectra. (b-d) high-resolution spectra

of Nb 3d, S 2P and Se 3d, respectively



Fig. S10 EPR spectra of NbSSe and NbSSe-Co_{0.1.}



Fig. S11 The HER polarization curves for Grounded NbSSe and Ex-NbSSe.



Fig. S12 XRD patterns of NbSSe doped with 15% Co.



Fig. S13 Overpotentials at 10 mAcm⁻² and Tafel slope of NbSSe bulks, Ex-NbSSe flakes, and Co-doped NbSSe.



Fig. S14 Cyclic voltammograms in the region of 0.25–0.45 V vs. RHE for the (a) NbSSe bulks (b) Ex-NbSSe flakes and (c) NbSSe-Co_{0.1}.



Fig. S15 XRD patterns of NbSSe-Co $_{0.1}$ before and after 5000 cycles. (The broad

peak labeled as # belongs to the sample holder).