Electronic Supplementary Material (ESI) for Journal of Materials Chemistry A.

Controlled synthesis of $Ni(OH)_2/Ni_3S_2$ hybrid Nanosheet Arrays as Highly Active and stable Electrocatalysts for Water Splitting

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Characterization technique

The same volume of gas sample in the headspace of the electrolytic cell was withdrawn by a SGE gas-tight syringe and analyzed by gas chromatography (GC). The H_2 and O_2 in the sampled gas was separated by passing through a 2 m × 3 mm packed molecular sieve 5A column with an Ar carrier gas and quantified by a Thermal Conductivity Detector (TCD)(Shimadzu GC-9A).

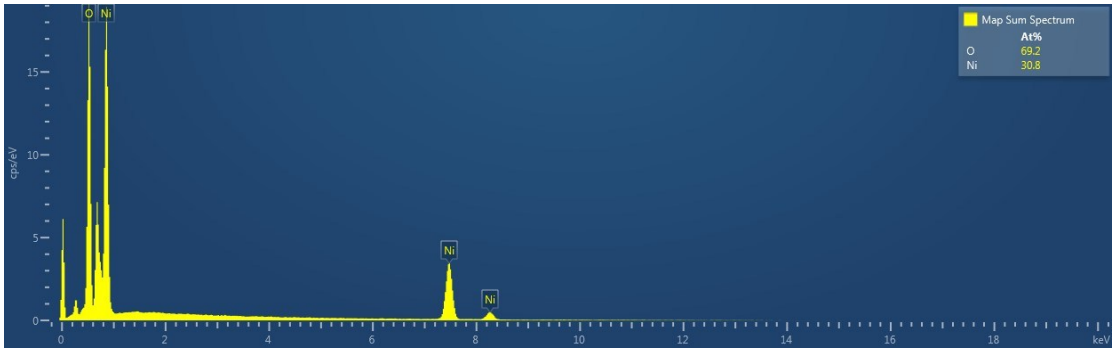

Fig.S1 EDS of $Ni(OH)_2$.

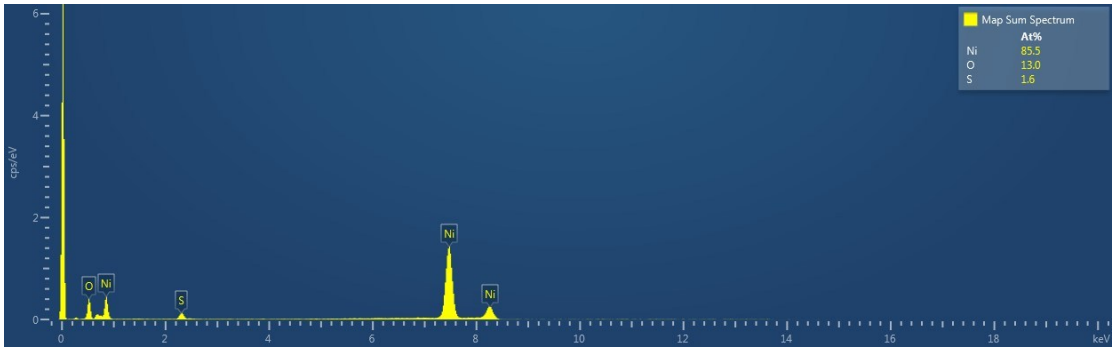

Fig.S2 EDS of $Ni(OH)_2/Ni_3S_2$-9h.

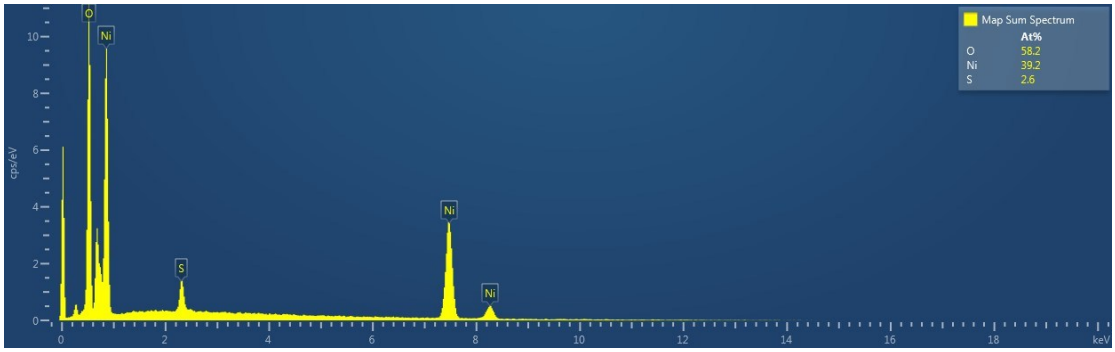

Fig.S3 EDS of $Ni(OH)_2/Ni_3S_2$-12h.

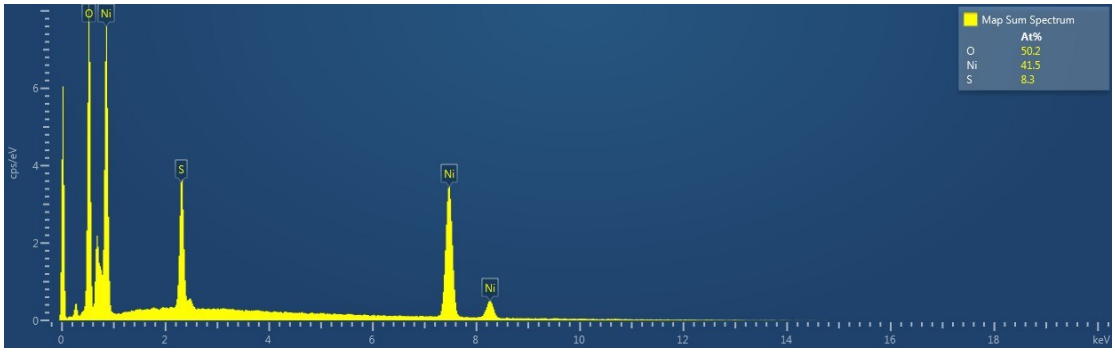

Fig.S4 EDS of $Ni(OH)_2/Ni_3S_2$-15h.

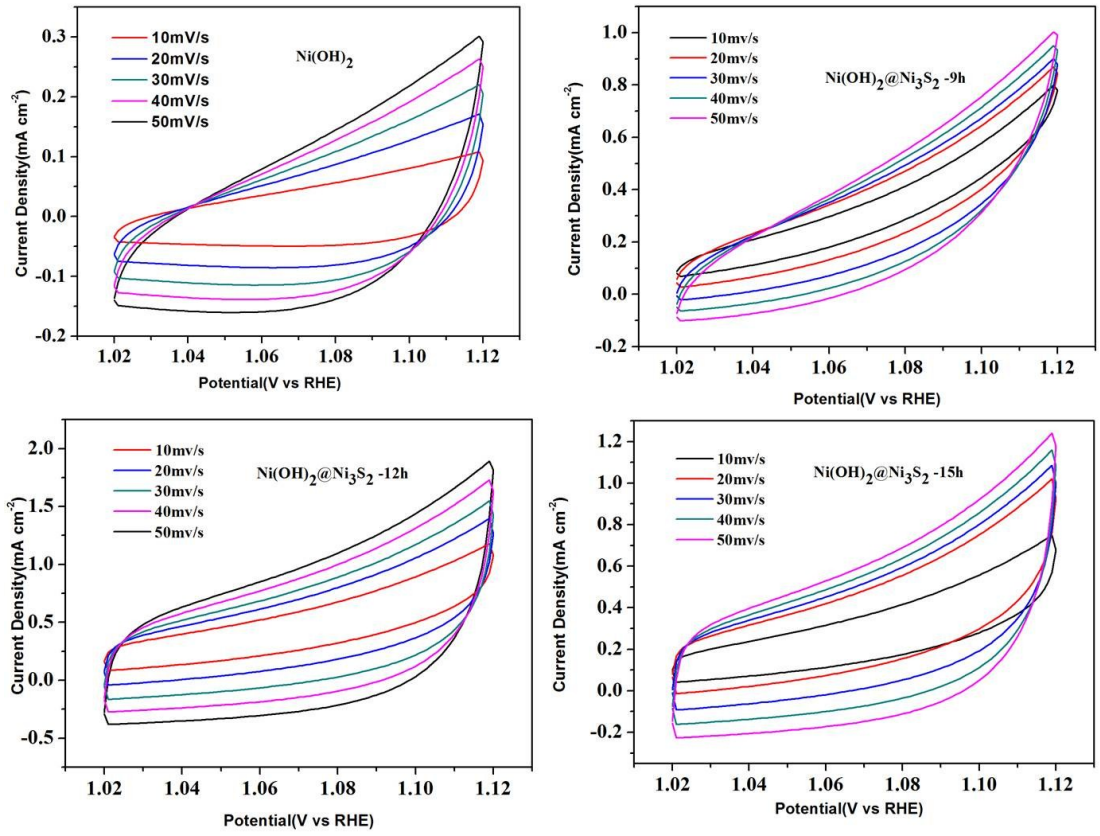

Fig. S5 CV_S of $Ni(OH)_2/Ni_3S_2$ with different scan rates (10-50 mV s^{-1}) in the region of 1.02-1.12V *vs* RHE.

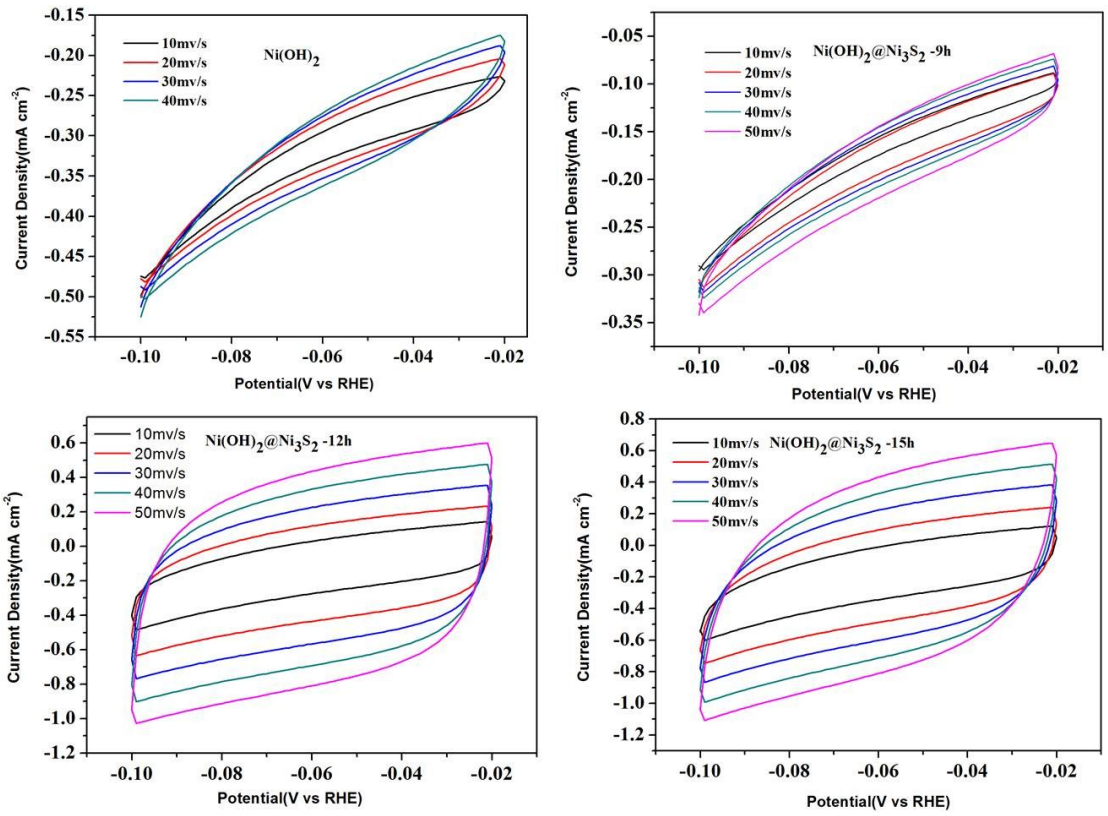

Fig. S6 CV_S of $Ni(OH)_2/Ni_3S_2$ with different scan rates (10-50 mV s^{-1}) in the region of -0.1—0.02V *vs* RHE.

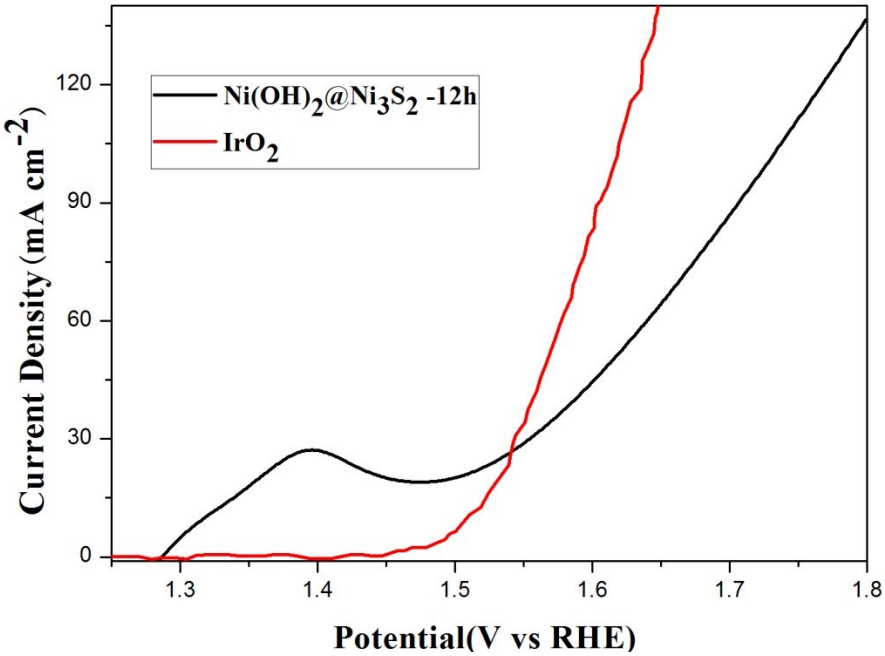

Fig. S7 (a) Polarization curves of $Ni(OH)_2/Ni_3S_2$-12h and IrO_2 in 1.0 M KOH at a potential sweep rate of 5 mV s^{-1}.

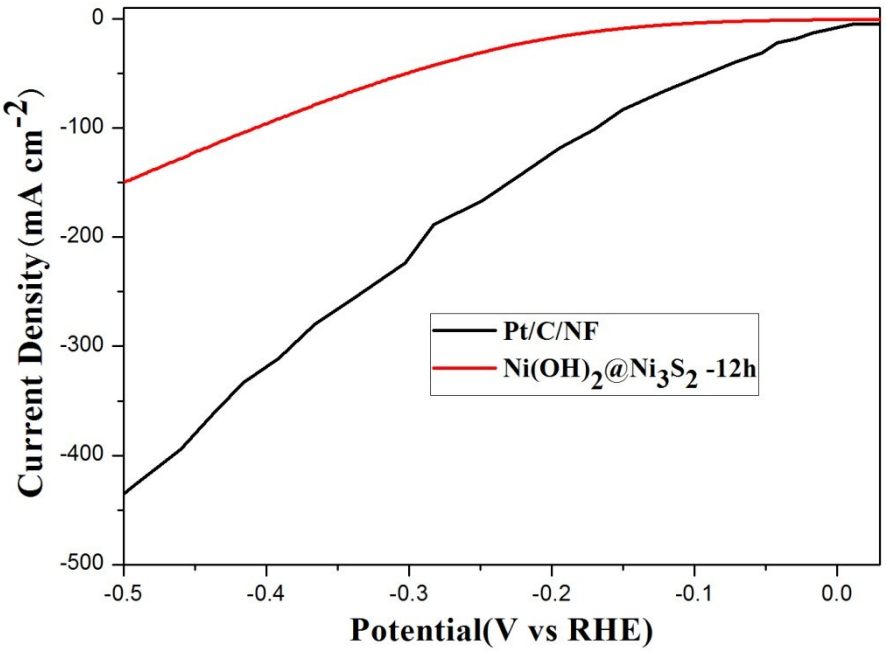

Fig. S8 (a) Polarization curves of $Ni(OH)_2/Ni_3S_2$-12h and Pt/C/NF in 1.0 M KOH at a potential sweep rate of 5 mV s^{-1}.

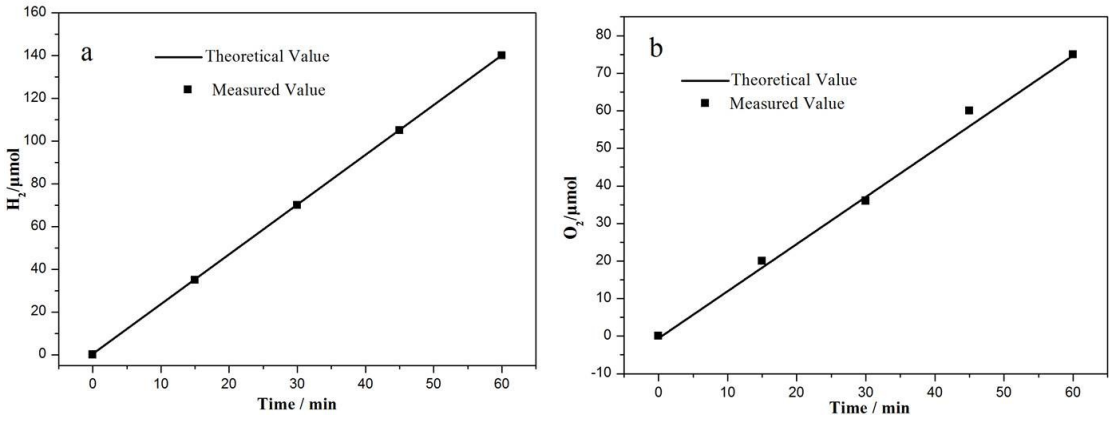

Fig. S9(a) Electrocatalytic efficiency of H_2 production over $Ni(OH)_2/Ni_3S_2$-12h at a potential of ca. -0.2V, measured for 60 min.(b) Electrocatalytic efficiency of O_2 production over $Ni(OH)_2/Ni_3S_2$-12h at a potential of ca. 1.50 V, measured for 60 min.

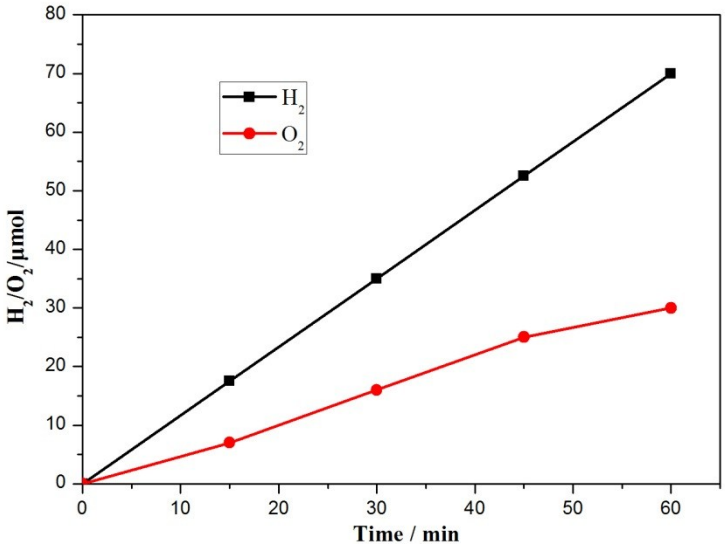

Fig. S10 H_2 and O_2 production over $Ni(OH)_2/Ni_3S_2$-12h at a potential of ca. 1.60 V, measured for 60 min.

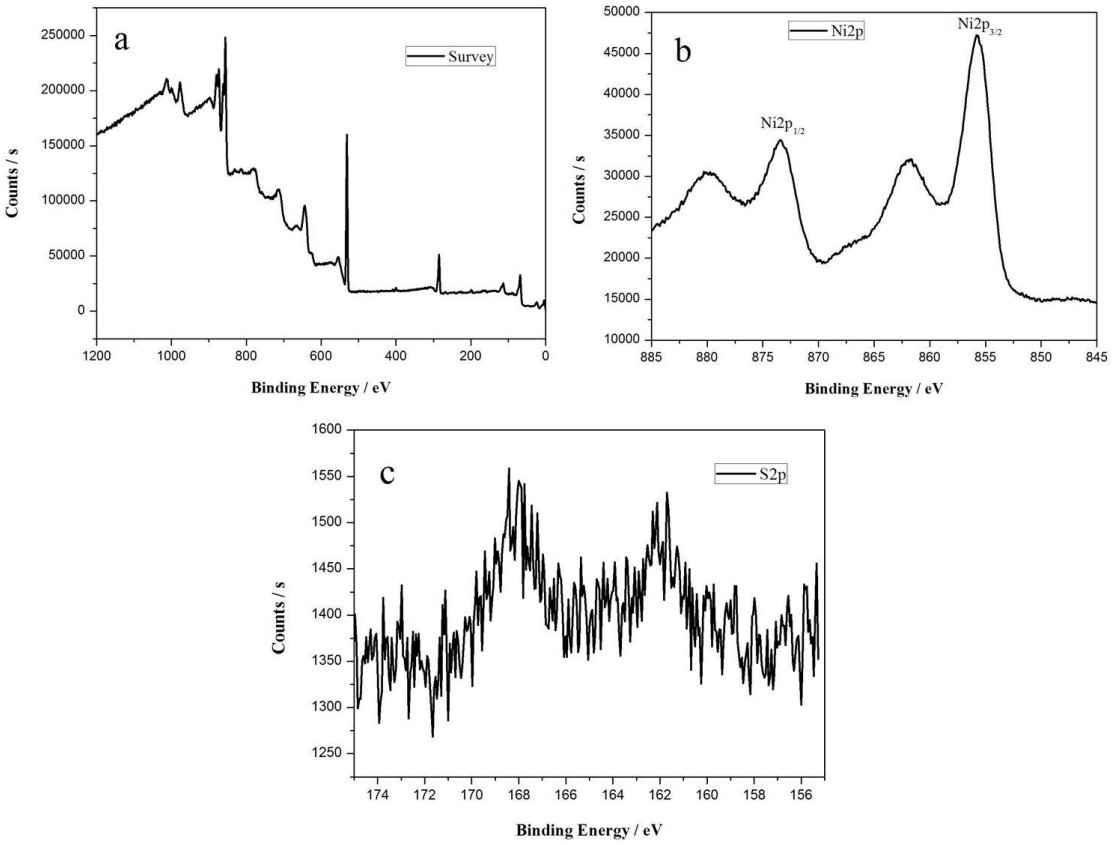

Fig. S11 XPS spectra of the (a) Survey, (b) Ni 2p and (c) S 2p peaks of Ni_3S_2/NF. The binding energy of each element was corrected by the C1s peak (284.8 eV).

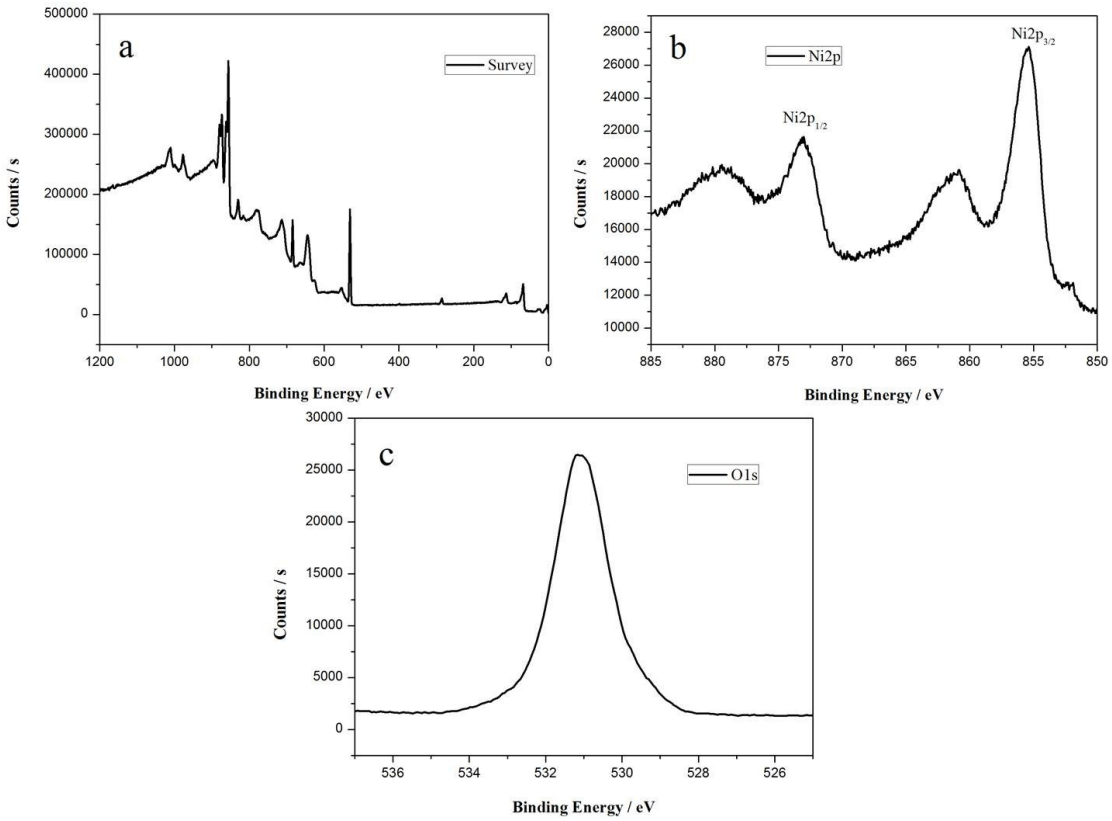

Fig. S12 XPS spectra of the (a) Survey, (b) Ni 2p and (c) O 1s peaks of $Ni(OH)_2$/NF. The binding energy of each element was corrected by the C1s peak (284.8 eV).