

Supporting Information for:
**Electrodeposition of Ni-doped FeOOH Oxygen Evolution Reaction Catalyst
for Photoelectrochemical Water Splitting**

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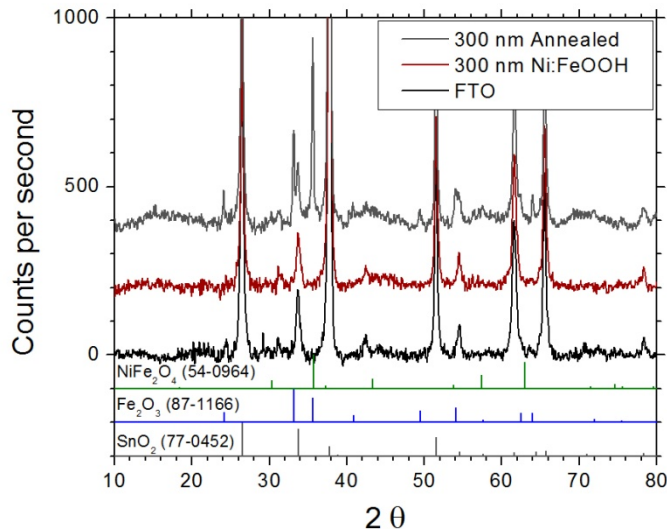


Figure S1. XRD pattern comparison of the substrate, freshly prepared Ni:FeOOH, and annealed Ni:FeOOH film. No significant differences from the substrate are notable for the freshly prepared film. There is perhaps a slight difference around 31.3° , however, there is a similar, albeit narrower, peak for the substrate trace. Furthermore, a peak at 31.3° is not consistent with any peaks of any Ni, Fe, and/or O separate or combined compounds, the peak also appears nearly unchanged after annealing. Therefore, it seems likely this peak is actually due to the substrate.

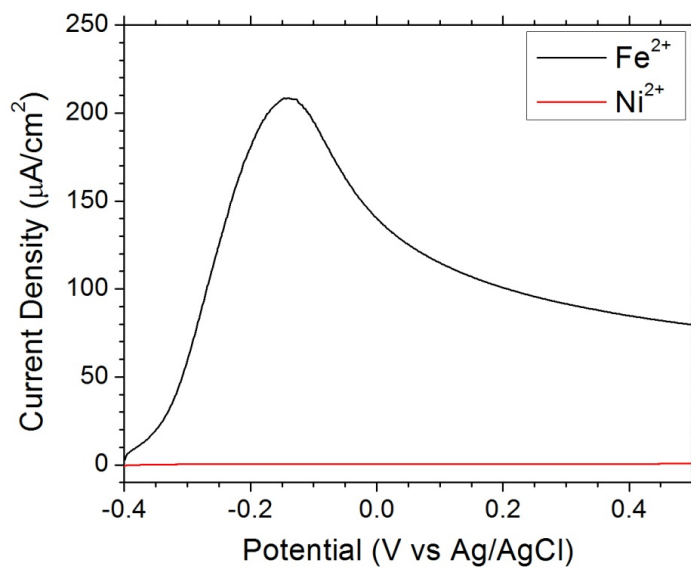


Figure S2. Linear voltage sweeps at 50 mV/s of FTO in 0.4 M NaCl + 0.1 M NMI + 5 mM of either FeCl_2 (black) or NiCl_2 (red). From this it is very clear that Ni^{2+} species are not oxidized at the deposition potentials used – between -0.2 and -0.125 V – whereas the Fe^{2+} species are oxidized.

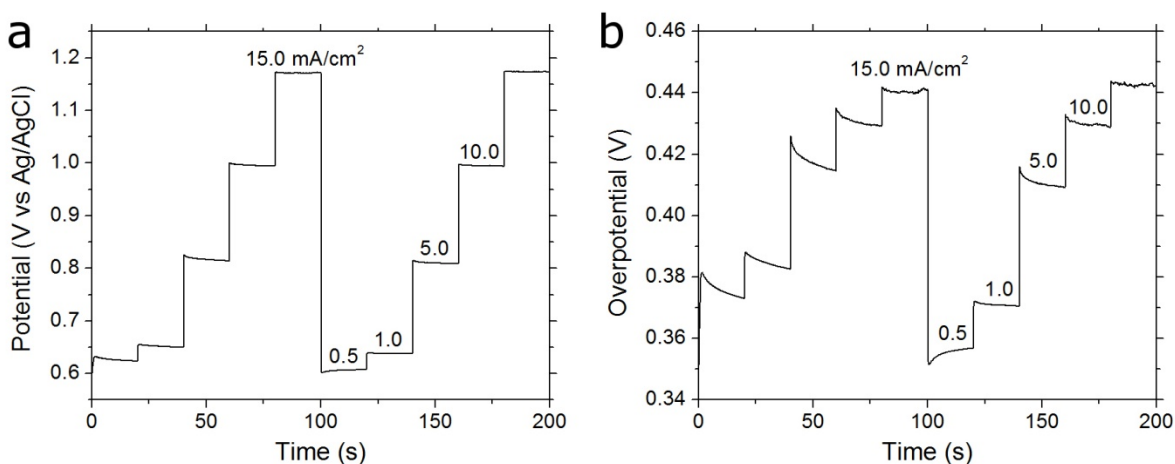


Figure S3. (a) Raw multi-current step data, text above each step is indicating the current density being passed for each step. (b) Data after adjustment for the series resistance and converted to difference from the thermodynamic OER potential (234 mV vs Ag/AgCl). Series resistance (which includes FTO resistance) is measured by the built-in step potentiometry method of the CHI potentiostat – for the data shown here the resistance was 38.5 Ω .

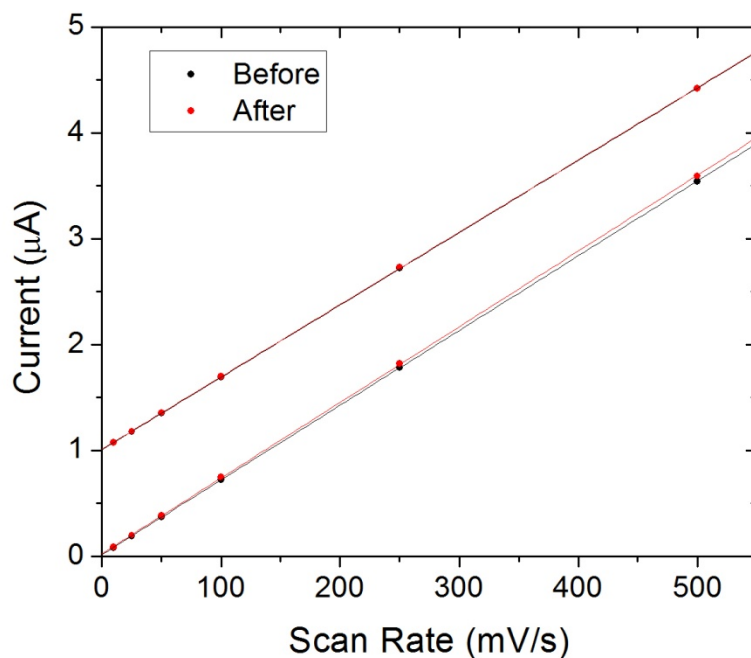


Figure S4. Comparison of the surface area measurement before and after 1 hour of testing at 10 mA/cm² for two 9/1 Ni:FeOOH films. The second set of data is shifted up by 1 μA for plotting purposes. The potential window was 0 to -0.1 V vs Ag/AgCl in a 0.1 M NaOH electrolyte, each CV scan was run until stable traces were obtained – usually no more than 3 cycles were necessary. One set of data shows no difference in slope (and thus area) while the other shows a difference of 1.3%, not enough to cause any significant change in the measured overpotential. All fits have an R^2 greater than 0.9999. Another interesting note is that the implied double layer

capacitance for these films is at most $7.4 \mu\text{F}/\text{cm}^2$, rather a bit lower than commonly reported values. The reason for this is not currently known, though the result is highly repeatable.

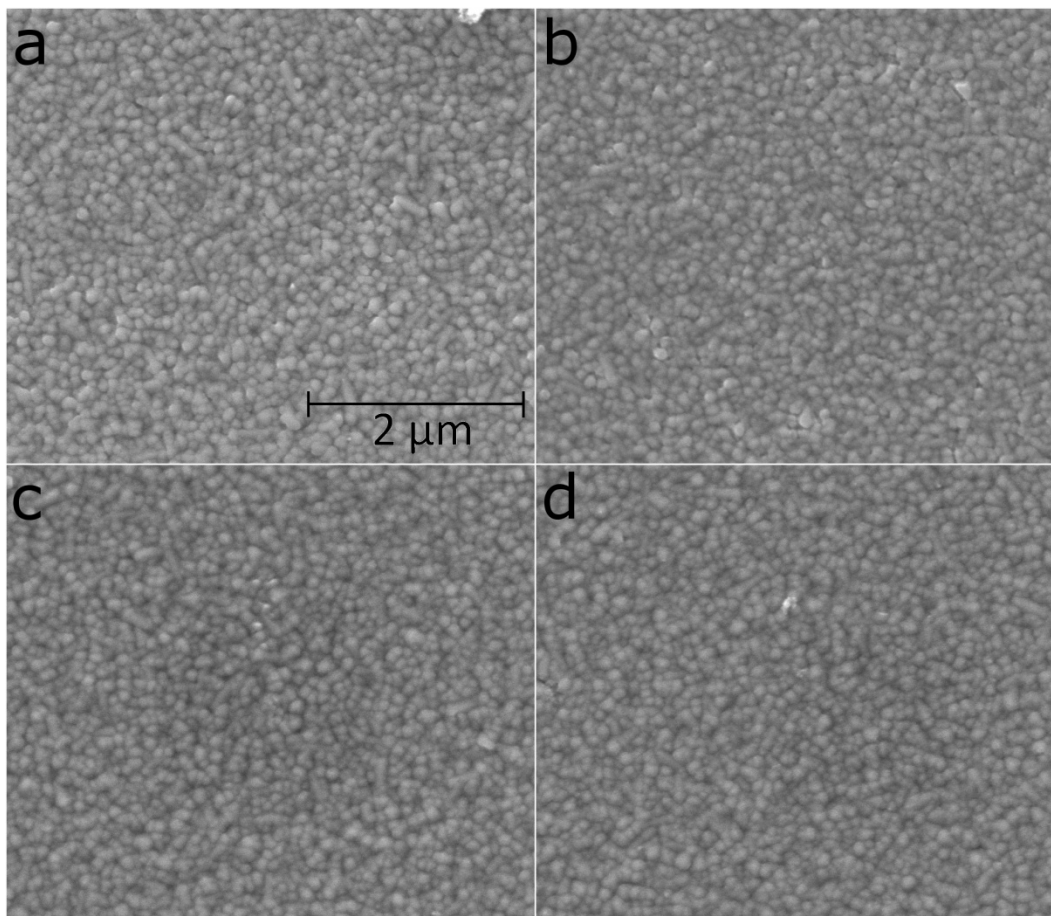


Figure S5. SEM images of pure FeOOH (a & b) and 15/1 Ni:FeOOH (c & d) before and after 1 hour of testing at $10 \text{ mA}/\text{cm}^2$ (see Figure 3b). There might be slight change to the pure FeOOH after testing (there are a number of what appear to be small pinholes) but this only affects a small portion of the film area and is unlikely to explain most of the drop in activity during the stability testing.

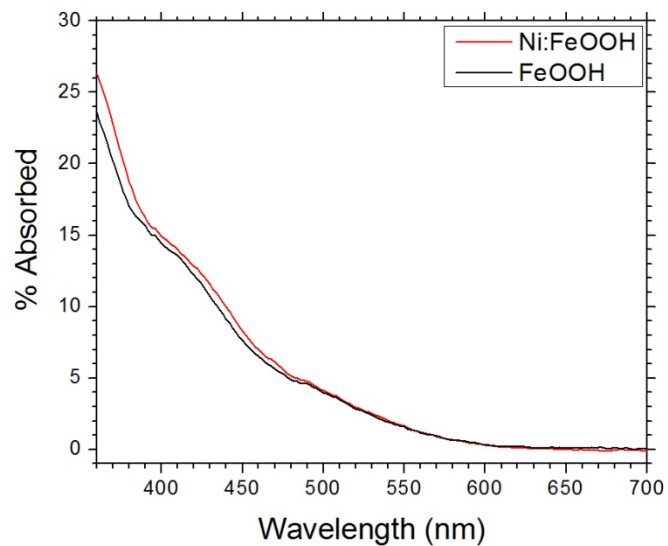


Figure S6. Absorption of 10 mC/cm² thick pure FeOOH and 9/1 Ni:FeOOH. Each trace is the average of 2 films; the film the film variability is quite low. Data taken with a Cary 500 spectrometer with a Labsphere integrating sphere attachment. The overall difference is quite small, integrating over the AM1.5G spectrum it is found that the pure FeOOH film absorbs a photon flux equivalent to 0.72 mA/cm² while the Ni-doped film absorbs 0.75 mA/cm², only a 30 μ A/cm² difference.