Supporting Information

# ALD of NiO and Pt on $TiO_2$ Nanotube Arrays Integrated into Titanium Porous Transport Layers for Dispersion Controlled Electrocatalysts

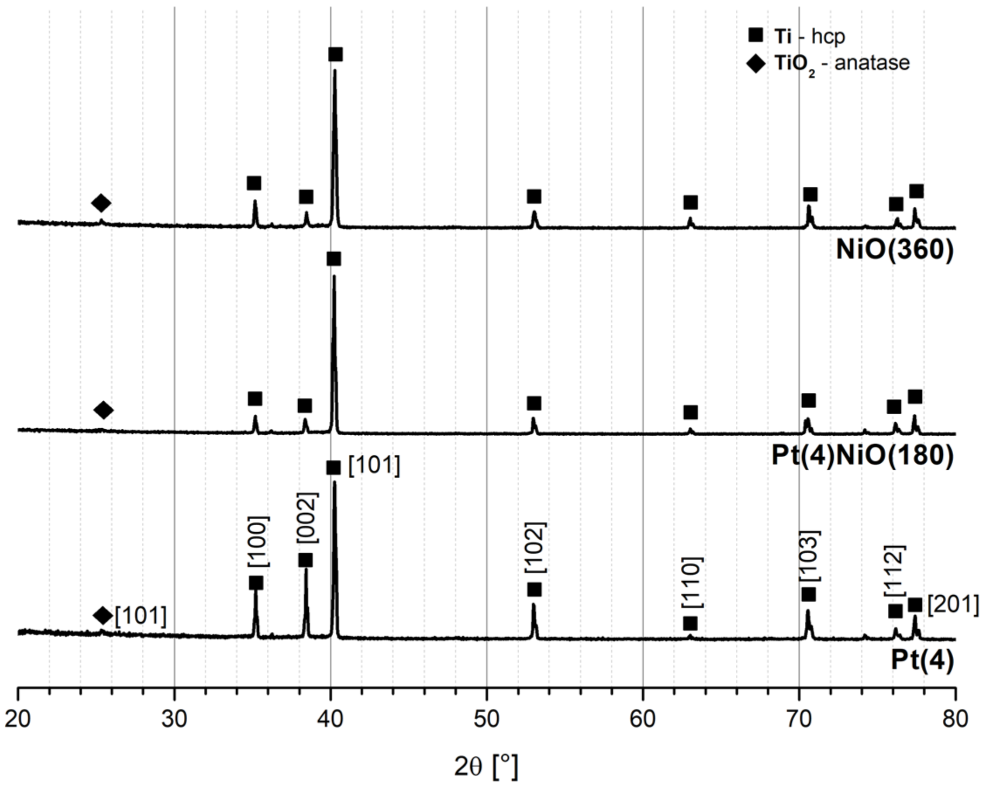
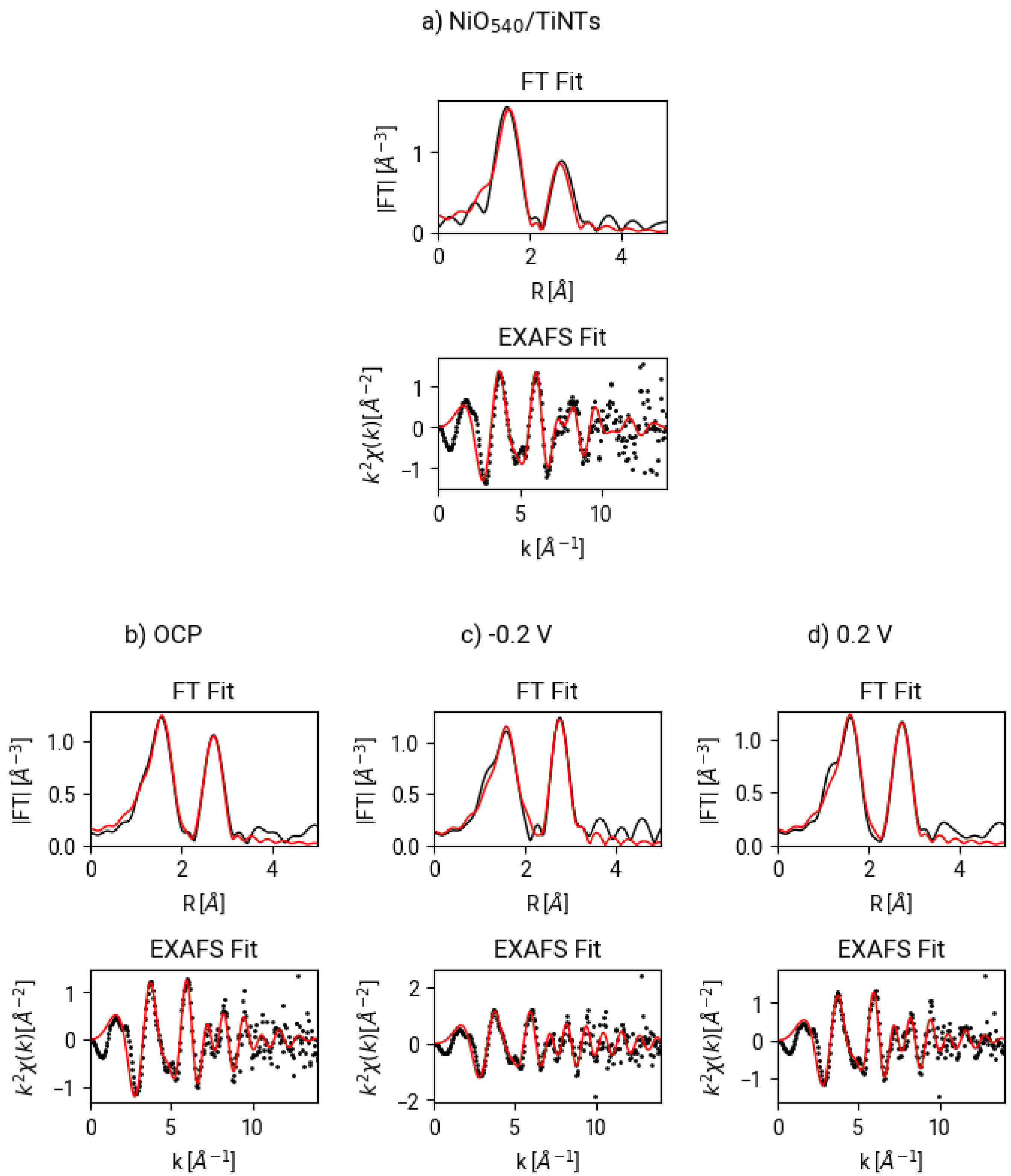



**Figure S1.** XRD diffractograms of a) and b) Pt(4)Ni(360)/TiNT; c) and d) Pt(4)/TiNT; e) and f) Ni(360)/TiNT. Each replica is a side of the sample

**Table S1.** Results of the EXAFS fit performed with an hanning window in a R range of 1-3.5Å

| **Sample** | **Ni-O** | | | **Ni-Ni** | | |
|---|---|---|---|---|---|---|
| | **$S_0$** | **R [Å]** | **$\Delta\sigma^2$ [×10³Å²]** | **$S_0$** | **R [Å]** | **$\Delta\sigma^2$ [×10³Å²]** |
| *$NiNT_{540}$* | 0.8 | 2.08 | 4.74 | 0.4 | 3.07 | 9.83 |
| *OCP* | 0.8 | 2.06 | 7.24 | 0.4 | 3.10 | 8.17 |
| *-0.2V* | 0.8 | 2.07 | 9.51 | 0.2 | 3.12 | 3.01 |
| *0.2V* | 0.8 | 2.07 | 7.26 | 0.4 | 3.11 | 6.89 |
| *0.5V* | 0.8 | 2.07 | 7.74 | 0.3 | 3.12 | 5.67 |
| *0.7V* | 0.8 | 2.07 | 8.34 | 0.2 | 3.12 | 3.01 |
| *OCP post* | 0.7 | 2.07 | 6.40 | 0.3 | 3.13 | 3.45 |


a) NiO540/TiNTs
FT Fit
|FT| [Å^-3]
R [Å]
EXAFS Fit
k^2χ(k) [Å^-2]
k [Å^-1]
b) OCP
c) -0.2 V
d) 0.2 V

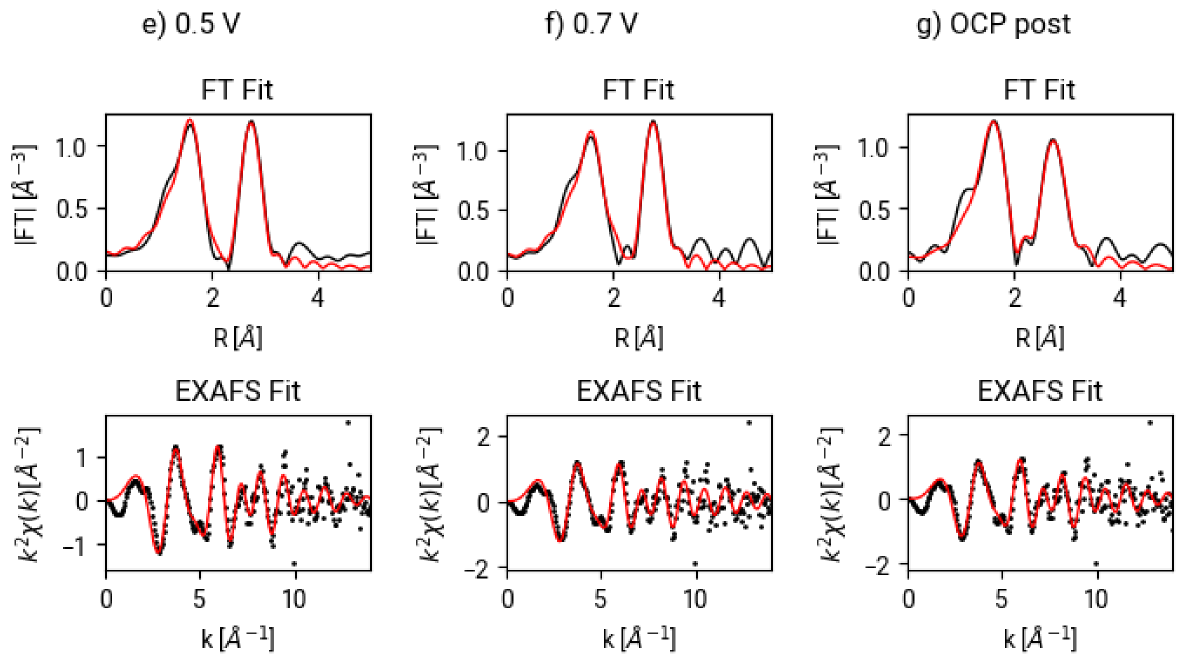



**Figure S2.** Fit results of the EXAFS spectra: a) Fresh sample, b) OCP before the in-situ, c-f) operando, g) OCP after the voltammetry.

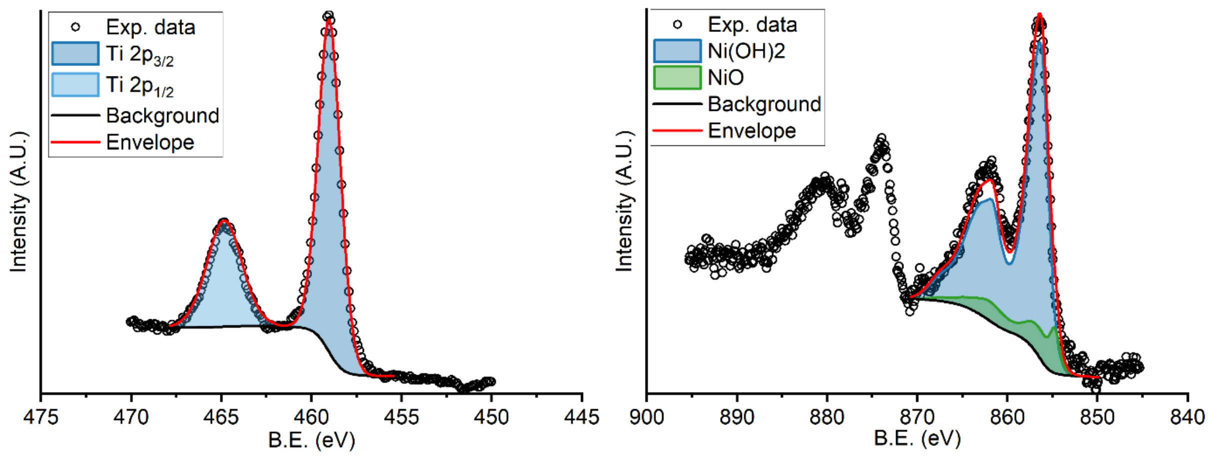



**Figure S3.** XPS fitting of NiO(360)/TiNTs

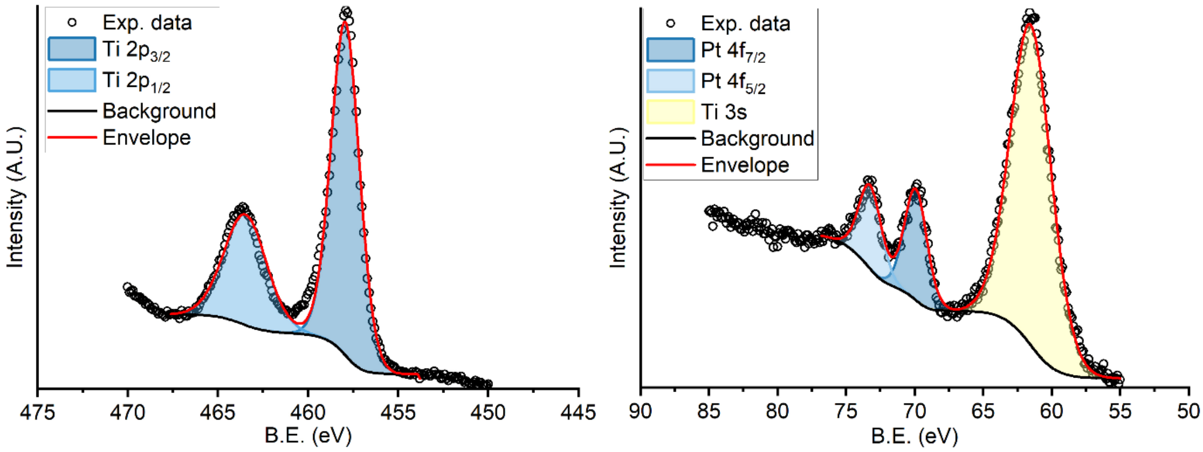



**Figure S4.** XPS fitting of Pt(4)/TiNTs

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| | | | | **Pt(4)** | | | |
| **Ti** | 2p | 457,9 | 1,9 | 19939 | 0,1069 | 186523 | 98,8% |
| **Pt** | 4f | 69,9 | 1,9 | 501 | 0,227 | 2206 | 1,2% |
| **Ni tot.** | 2p | - | - | 0 | 0,2998 | 0 | 0,0% |
| | | | | **NiO(360)** | | | |
| **Ti** | 2p | 459,0 | 1,5 | 2933 | 0,1069 | 27432 | 58,8% |
| **Pt** | 4f | N.A. | N.A. | 0 | 0,227 | 0 | 0,0% |
| **Ni tot.** | 2p | - | - | 5754 | 0,2998 | 19192 | 41,2% |
| | | | | **Pt(4)NiO(180)** | | | |
| **Ti** | 2p | 458,9 | 2,6 | 2918 | 0,1069 | 27293 | 77,5% |
| **Pt** | 4f | 70,9 | 2,5 | 58 | 0,227 | 256 | 0,7% |
| **Ni tot.** | 2p | - | - | 2292 | 0,2998 | 7646 | 21,7% |

**Table S2.** XPS Quantification for Pt, Ni and Ti

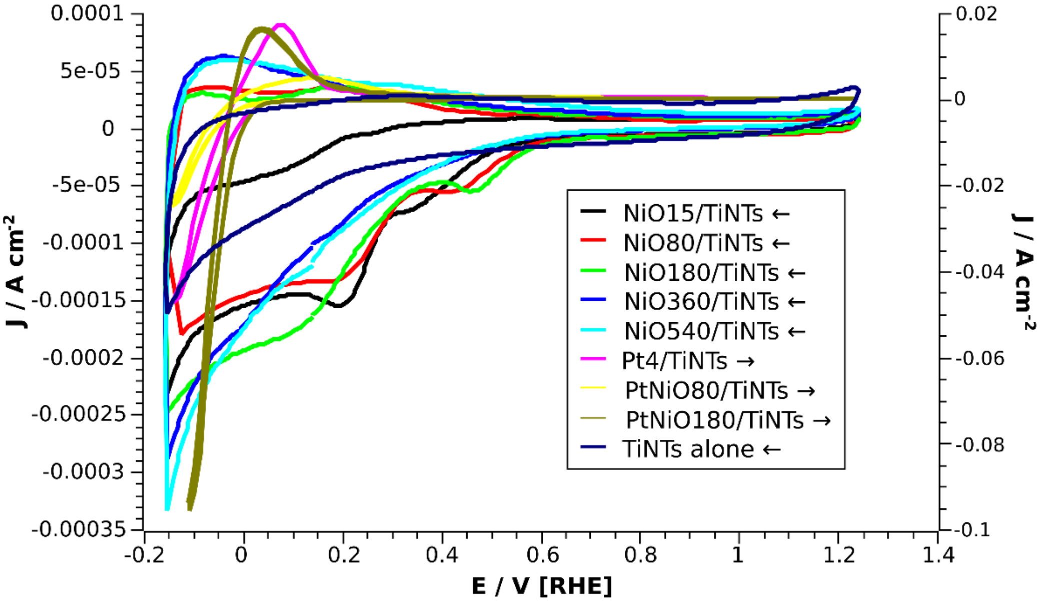



**Figure S5.** Cyclic voltammetry experiments of the ALD materials

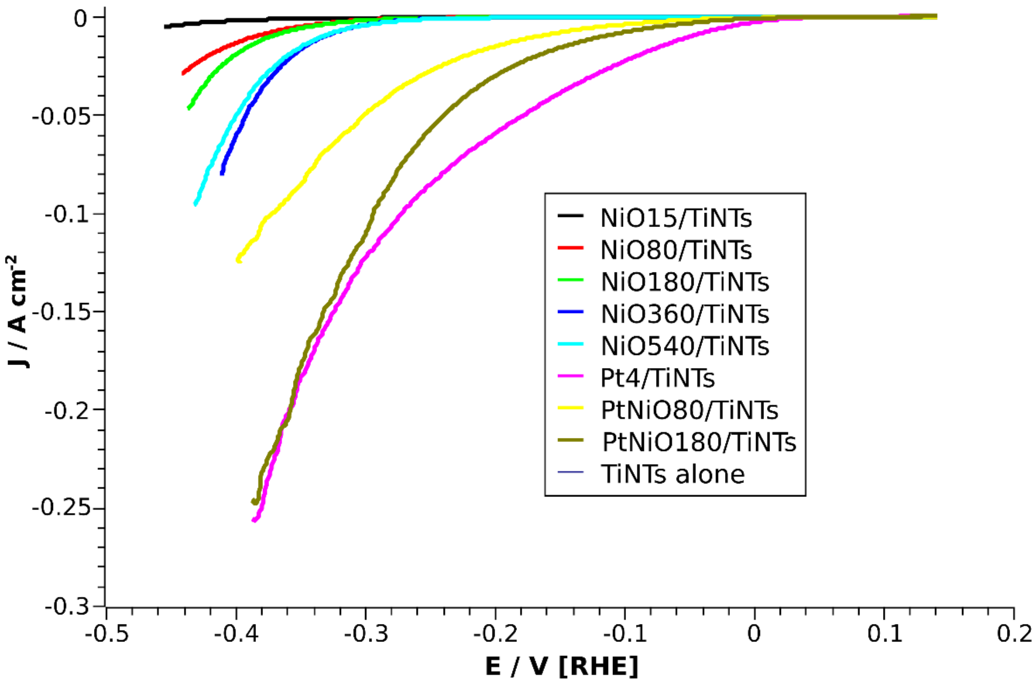



**Figure S6.** Hydrogen evolution polarization curves of ALD materials

Pt4: a) b) c) PtNiO80: d) e) f) PtNiO180 g) h) i)

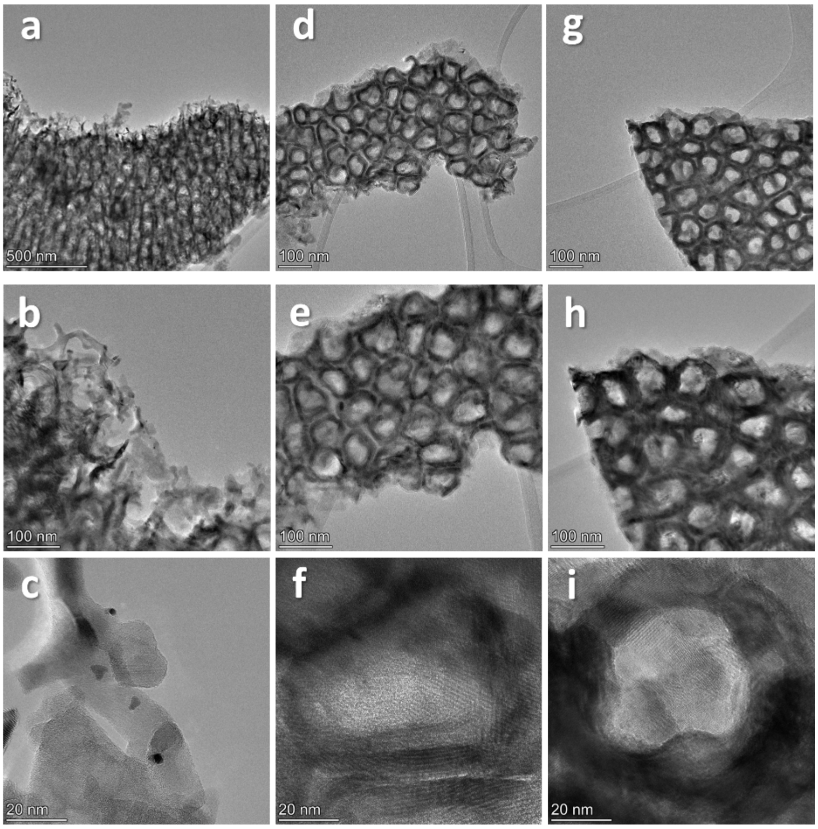



**Figure S7.** HR-TEM images at different magnification of the Pt-containing samples

| Catalyst | Ref | Overpotential / mV | J / mA cm$^{-2}$ | Stability / h |
|---|---|---|---|---|
| Pt(4)/TiNT (ALD) | This work | 17 / 51 / 168 | 1 / 10 / 50 | >50 |
| NiO(180)/TiNT (ALD) | This work | 283 / 399 | 1 / 10 | 25 |
| Pt(4)NiO(180)/TiNT (ALD) | This work | 13/ 43 / 132 | 1 / 10 / 50 | >50 |
| $Ni_xCo_\gamma$ P | [S1] | 36 / 161 / 251 | 10 / 500 / 1000 | 120 |
| NiMoB | [S2] | 68 / 257 | 50 / 500 | 300 |
| NiCoP-120 | [S3] | 56 / 128 / 247 | 10 / 100 / 1000 | 100 |
| Ni nanorods | [S4] | 271 | 100 | 36 |
| Pt-modified S-doped NiFe LDH | [S5] | 71 | 100 | 200 |
| NiRu alloy | [S6] | 15 | 10 | — |
| Pt/$NiO_xO_v$ | [S7] | 19.4 | 10 | 100 |
| D-NiO–Pt | [S8] | 20 | 10 | 100 |
| Pt-Ni(N) | [S9] | 13 | 10 | — |

**Table S3.** Literature comparison with similar HER materials in 1M KOH

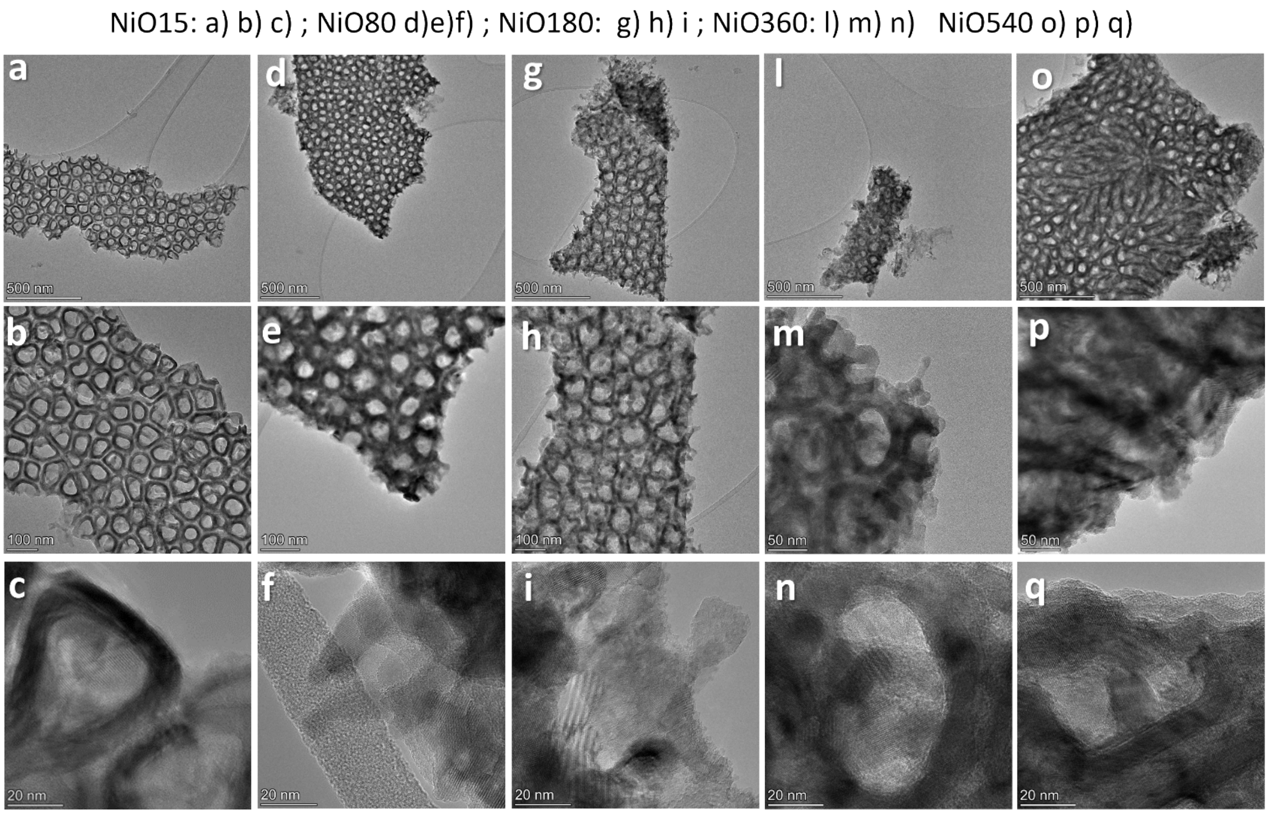



**Figure S8.** HR-TEM images at different magnification of the NiO-containing samples

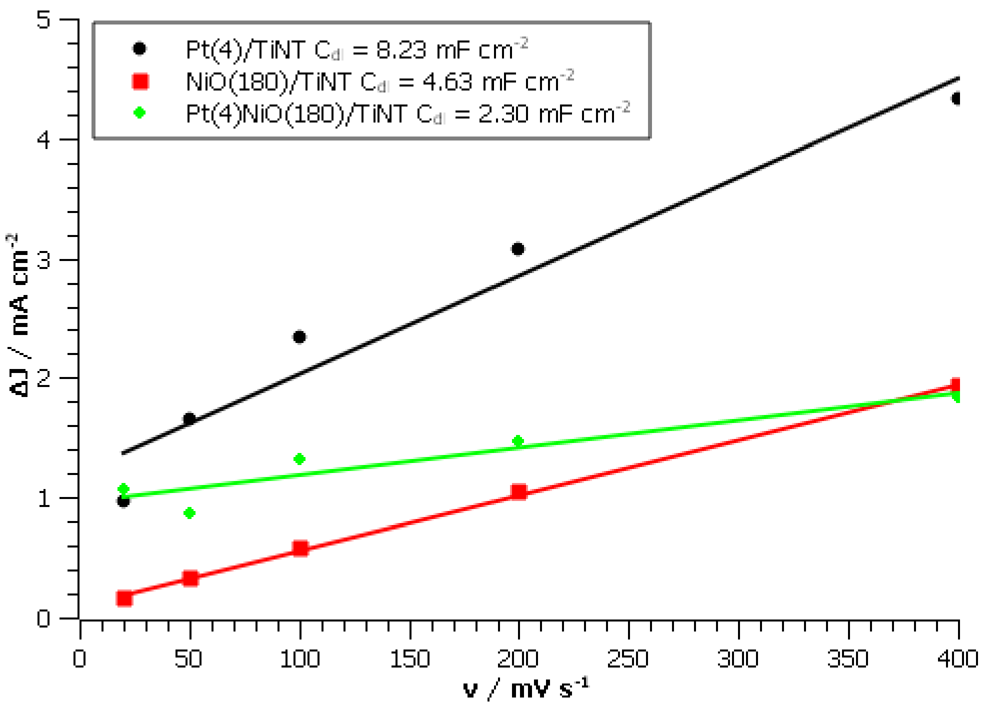



**Figure S9.** Differential capacitance measurements of Pt(4)/TiNT, NiO(180)/TiNT and Pt(4)NiO(180)/TiNT materials

Supporting References: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