## Supplementary Information

## Gamma-Radiation induced synthesis of freestanding nickel nanoparticles

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## Details for the catalyst ink preparation and working electrodes modification:

The sample was firstly prepared as an ink slurry and then attached onto a glass carbon (GC) electrode by a drop-casting method. The inks were prepared by mixing different amount of the corresponding catalyst powders (2.5 mg for Ni nanoparticle, 25 mg for commercial Ni powder (KEBO, <10  $\mu$ m), respectively), 0.75 mL Milli-Q water (18.2 M $\Omega$  cm<sup>-1</sup>), 0.25 mL IPA (99.5%, Sigma-Aldrich) and 10  $\mu$ L Nafion solution (5 wt. %, Sigma-Aldrich), the mixture was then sonicated for 5 min to make a homogeneous ink. 20  $\mu$ L of the asprepared inks were drop-casted onto the mirror polished GC electrodes (5 mm in diameter) and were dried at room temperature. It should be noted that the dispersed commercial Ni powder could be precipitated easily. To avoid this, the drop-casting was done immediately after the ink was taken from the sonicator.

## Details for ECSA measurement and calculation:

Electrochemical surface area (ECSA) of the synthesized Ni nanoparticles and the commercial Ni powder has been measured using double-layer capacitance ( $C_{dl}$ ) method. The double layer charging current ( $i_c$ ) was obtained from CVs, see Fig. S1 (A1, B1) at multiple scan rates (v) at 1.02 V (vs RHE), which was in the potential region where the presence of faradaic currents was minimized and the total current dominated by capacitive currents. By plotting  $i_c$  as a function of v, the slope of the linear fit was equal to  $C_{dl}$  ( $i_c = v C_{dl}$ ) (Fig. S1 (A2, B2)). The ECSA of the catalyst was calculated by dividing  $C_{dl}$  by 0.040 mF · cm<sup>-2</sup>, a typical value reported in alkaline aqueous solution. Based on the catalyst ink recipe, the mass of Ni on the electrode could be calculated and thereafter the ECSA normalized to Ni mass was calculated.



Figure S1. Double-layer capacitance measurements performed for the electrochemical surface area (ECSA) determination. Cyclic voltammetry (A1 for the Ni nanoparticles, B1 for the commercial Ni powder) measured at a non-Faradaic region in 0.1 M NaOH electrolyte; Cathodic (red solid circle) and anodic (black solid square) currents measured at 1.02 V vs RHE are plotted as a function of scan rate (A2 for the Ni nanoparticles, B2 for the commercial Ni powder). The determined double-layer capacitance of the system is taken as an average value of the slope of the linear fits to the data.



Figure S2 HRTEM images of the agglomerated Ni particles. The images are recorded at different magnification.