Supporting Information

Amorphous Ni-Based Nanoparticles for Alkaline Oxygen Evolution

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Table S1: List of materials used for synthesis and as standards for analysis

Material	Source	Purity	Particle Size
Ni	Alfa Aesar	99.9 wt %	< 149 µm
Co	Alfa Aesar	99.8 wt %	< 149 µm
Nb	Alfa Aesar	99.99 wt %	< 44 µm
Y	Goodfellow	99.9 wt %	<500 µm*
NiO	Fisher Scientific	99.8 wt %	---
β-$Ni(OH)_2$	Sigma Aldrich	---	---
β-NiOOH	This work**	---	---
Co_3O_4	Sigma Aldrich	99.5 wt %	< 50 nm
$Co(OH)_2$	Sigma Aldrich	95 wt %	---
Y_2O_3	This work***	---	---
$Y(OH)_3$	Muse Chem	99.99 wt%	---

*Milled at room temperature to reduce the particle size to < 149 µm prior to alloying

**Synthesized by adding a solution of 47.5 g KOH and 11 mL Br_2 in 500 mL of H_2O dropwise to 50 g of $Ni(NO_3)_2{\cdot}H_2O$ in 750 mL of H_2O at 100 °C. The sample was then rinsed with H_2O, centrifuged, filtered, and dried under vacuum for ca. 48 hours. The presence of β-NiOOH was verified using XRD.

***Produced by oxidizing Y powder and verified using XRD

Table S2: Nominal and measured (ICP-OES) compositions for as-produced nanoparticles

Material		Ni (at %)	Co (at %)	Nb (at %)	Y (at %)
Crystalline $Ni_{95}Co_5$	Nominal	95	5	---	---
	ICP-AES	95.2 ± 2.1	4.8 ± 0.3	---	---
Amorphous $Ni_{79.2}Nb_{12.5}Y_{8.3}$	Nominal	79.2	---	12.5	8.3
	ICP-AES	82.0 ± 2.0	---	10.8 ± 2.6	7.3 ± 0.8
Amorphous $Ni_{74.2}Co_5Nb_{12.5}Y_{8.3}$	Nominal	74.2	5	12.5	8.3
	ICP-AES	73.4 ± 2.1	4.9 ± 0.3	12.1 ± 2.8	9.6 ± 1.4

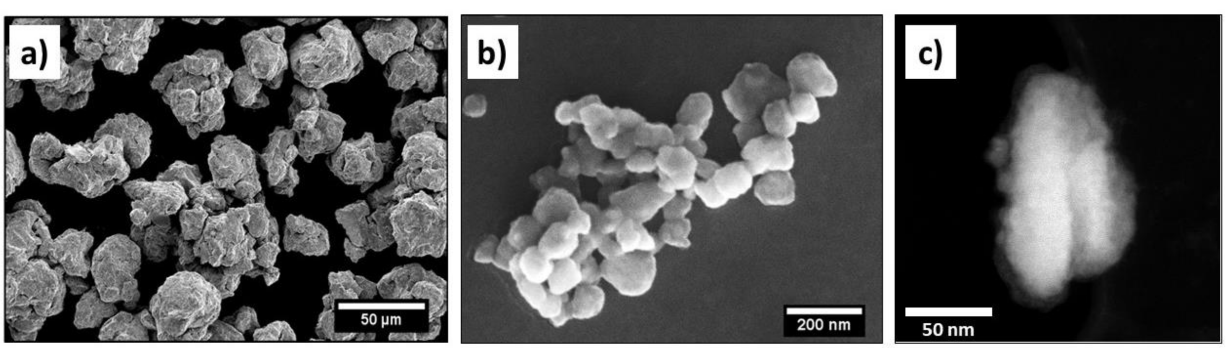

Figure S1: SEM images of amorphous $Ni_{74.2}Co_5Nb_{12.5}Y_{8.3}$ particles after (a) cryomilling and (b) SA-HEBM, along with (c) TEM image of a single particle.

Table S3: Tafel slope, current density at η = 400 mV, and η at j = 10 μA cm^{-2}_{ECSA} values for the OER of crystalline Ni, $Ni_{95}Co_5$ and amorphous $Ni_{79.2-x}Co_xNb_{12.5}Y_{8.3}$ (x = 0 and 5 at %) after anodically cycling 50 and 10000 times with a scan rate of 50 mV s^{-1} between 0.1 and 0.7 $V_{Hg/HgO}$. Experiments were conducted in deaerated 1 M KOH at 30 °C. Values and error bars are based on a minimum of three tests.

Material	Cycles	Tafel Slope (mV)	$j_{\eta = 400\ mV}$ (μA cm^{-2}_{ECSA})	$\eta_{j = 10\ \mu A\ cm^{-2}_{ECSA}}$ (mV)
Crystalline Ni	50	59 ± 8.2	252 ± 89.7	299 ± 1.80
	10000	71 ± 0.2	169 ± 44.4	276 ± 13.4
Crystalline $Ni_{95}Co_5$	50	49 ± 1.0	852 ± 180	235 ± 3.50
	10000	81 ± 5.9	93.3 ± 43.6	297 ± 22.1
Amorphous $Ni_{79.2}Nb_{12.5}Y_{8.3}$	50	50 ± 3.8	299 ± 18.8	281 ± 17.0
	10000	73 ± 2.7	76.1 ± 18.1	288 ± 12.5
Amorphous $Ni_{74.2}Co_5Nb_{12.5}Y_{8.3}$	50	71 ± 3.1	287 ± 48.1	267 ± 17.0
	10000	58 ± 1.3	289 ± 73.3	265 ± 18.1

X-ray Photoelectron Spectroscopy (XPS) Analysis

Tables S4 (Ni 2p) and S5 (O 1 s) were used to aid with the identification of surface species produced on crystalline $Ni_{95}Co_5$, amorphous $Ni_{79.2}Nb_{12.5}Y_{8.3}$, and amorphous $Ni_{74.2}Co_5Nb_{12.5}Y_{8.3}$ after anodic cycling 50 times. The standards produced and tested within this report were confirmed to be in good agreement with literature (NIST X-ray Photoelectron Spectroscopy Database, NIST Standard Reference Database Number 20, National Institute of Standards and Technology, Gaithersburg MD, 20899 (2000), doi:10.18434/T4T88K).

Table S4: Ni $2p_{3/2}$ XPS fitting parameters for alloys after anodic cycling and relevant standards

Material	Binding Energy (eV)	Peak Width FWHM (eV)	Δsplit (eV)	Δsat (eV)	Reference
Crystalline $Ni_{95}Co_5$	852.1, 854.6, 855.1	1.57, 1.76, 2.83	18.4, 17.6, 17.8	6.10	This work
Amorphous $Ni_{79.2}Nb_{12.5}Y_{8.3}$	851.9, 855.0	1.24, 2.70	17.3, 17.5	5.88	This work
Amorphous $Ni_{74.2}Co_5Nb_{12.5}Y_{8.3}$	852.0, 854.9	1.13, 3.03	17.2, 17.5	5.95	This work
Ni	852.5	1.08	17.3	5.65	This work
NiO	854.1, 855.7	0.98, 3.30	17.3, 17.3	6.30, 7.10	This work
β-$Ni(OH)_2$	855.4, 856.2	2.06, 3.13	17.4, 18.0	5.38, 6.10	This work
β-NiOOH	854.8, 856.0	1.33, 2.05	17.4	5.11	This work
γ-NiOOH	855.3	2.42	17.5, 18.3	5.80	1

Δsplit is the difference between Ni $2p_{3/2}$ and Ni $2p_{1/2}$ peaks

Δsat is the difference between Ni $2p_{3/2}$ and Ni $2p_{3/2}$ satellite peak

Table S5: O 1 s XPS fitting parameters for alloys after anodic cycling and relevant standards

Material	Binding Energy(eV)	Peak Width FWHM (eV)	Reference
Crystalline $Ni_{95}Co_5$	528.9, 530.8, 533.1	1.37, 1.85, 1.98	This work
Amorphous $Ni_{79.2}Nb_{12.5}Y_{8.3}$	529.2, 530.6, 532.7	1.31, 1.68, 1.49	This work
Amorphous $Ni_{74.2}Co_5Nb_{12.5}Y_{8.3}$	529.3, 530.7, 531.4	1.25, 1.61, 2.30	This work
NiO	529.7, 531.2, 531.3	0.88, 3.19, 2.14	This work
β-$Ni(OH)_2$	530.7, 530.9	1.39, 2.14	This work
β-NiOOH	529.3, 530.7, 532.1	0.87, 1.74, 2.68	This work
γ-NiOOH	528.9, 530.8, 532.6	1.27, 1.92, 2.18	1
Co_3O_4	529.4, 529.8, 531.2, 532.5	1.49, 0.92, 1.86, 2.36	This work
$Co(OH)_2$	531.2, 532.7	1.55, 1.85	This work
CoO_2	528.7, 531.2, 533.0	---	2
Y_2O_3	529.2, 531.5, 533.3	1.45, 2.01, 1.99	This work
$Y(OH)_3$	531.4, 532.4, 533.2	1.72, 1.76, 2.28	3

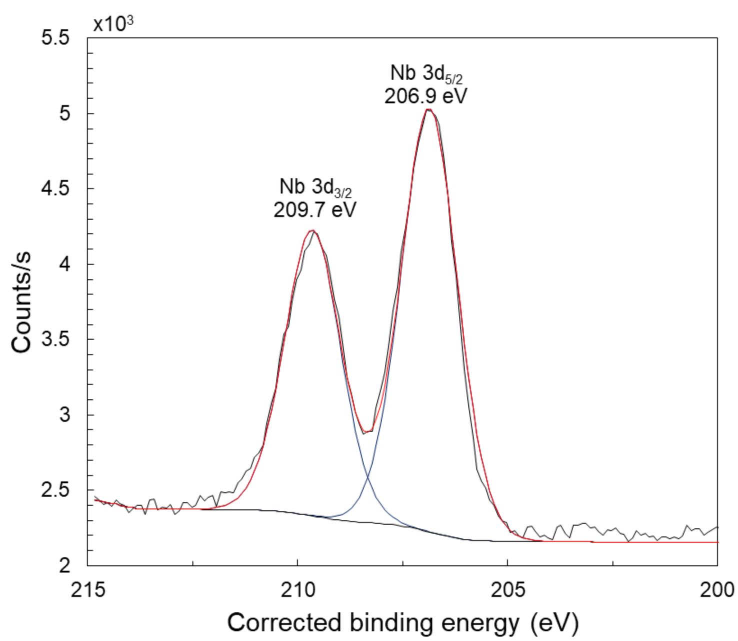

Figure S2: Nb 3d XPS spectra corresponding to the presence of Nb_2O_5 on the surface of amorphous $Ni_{74.2}Co_5Nb_{12.5}Y_{8.3}$ prior to anodic cycling

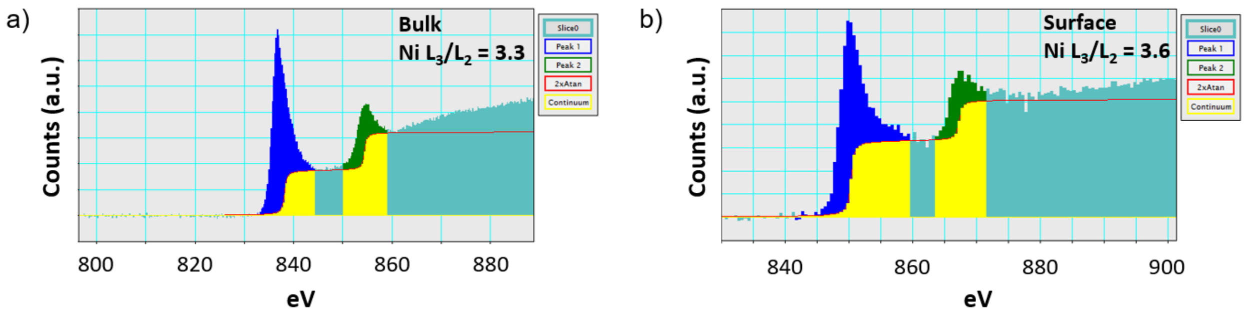

Figure S3: Ni L_3/L_2 white-line intensity ratios from electron energy-loss near-edge spectroscopy for pristine amorphous $Ni_{74.2}Co_5Nb_{12.5}Y_{8.3}$ a) bulk and b) surface

Table S6: Comparison of L_3/L_2 white-line intensity ratios for pristine $Ni_{74.2}Co_5Nb_{12.5}Y_{8.3}$ bulk and surface with Ni, NiO and $Ni(OH)_2$ references

Sample	Estimated L_3/L_2 ratios	Reference
$Ni_{74.2}Co_5Nb_{12.5}Y_{8.3}$ bulk	3.3	This work
$Ni_{74.2}Co_5Nb_{12.5}Y_{8.3}$ surface	3.6	This work
Ni reference	3.3	4
NiO reference	3.8	5
NiO reference	4.0	6
$Ni(OH)_2$ reference	3.6	5

Table S7: Quantification of Nb and Y elemental edges in EELS for surface and core regions in pristine, 1000 cycled, 5000 cycled, and submerged $Ni_{74.2}Co_5Nb_{12.5}Y_{8.3}$ nanoparticles.

Area	Sample	Nb Concentration (at%)	Y Concentration (at%)
Core	Pristine	45	55
	Submerged	39	61
	1000 Cycles	41	59
	5000 Cycles	48	52
Surface	Pristine	69	31
	Submerged	68	32
	1000 Cycles	25	75
	5000 Cycles	0	100

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