Cryomilled Ni-Co-Se Enables Water Oxidation Electrocatalysts Durable at High Current Densities

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Article

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Abstract

The oxygen evolution reaction (OER) limits electrocatalysis due to the high overpotential incurred by the poor reaction kinetics; this problem worsens over time if the performance of the OER electrocatalyst diminishes during operation. Here, we report the synthesis of immiscible Ni-Co-Se nanoparticles (<10 nm) for alkaline OER using milling at a cryogenic temperature. Milling at such low temperatures promotes thermodynamically stable nanocrystalline intermetallics with a high density of coordinatively unsaturated active sites. Using operando synchrotron spectroscopy, electron microscopy, and density functional theory we found that during the OER, Se ions leaches out of the nanocrystalline structure activating the electrocatalyst by hydrating and transforming defective Ni and Co sites into active and stable oxyhydroxides. Activated $(\text{NiCo})_3\text{Se}_4$ electrocatalyst required only an overpotential of 279 mV at 0.5 A.cm^{-2} and 329 mV at 1 A.cm^{-2} for 500 hours in 1M KOH. Using anion exchange membrane, we report the lowest cell voltage for an alkaline water electrolyser delivering 2 A.cm^{-2} at 2 V.

Introduction

To date, benchmark noble electrocatalysts based on RuO_x and IrO_x are considered to be state-of-the-art OER electrocatalysts in view of their superior performance; unfortunately, their high price and scarcity limit their widespread application. In contrast, earth abundant metal oxides, selenides, nitrides and phosphides derived from the first-row transition metals Ni, Co, and Fe can offer a cheaper alternative for efficient OER electrocatalysts in anion exchange membrane (AEM) systems, which offer an alkaline environment that is more compatible with non-noble metal electrocatalysts than is the acidic environment.

One of the most efficient non-noble OER electrocatalysts in alkaline systems is Fe doped NiOOH .^{6–8} While a small amount of Fe can boost the electrocatalytic activity of NiOOH , it has been reported that under OER conditions, Fe segregates in Ni-Fe oxyhydroxide lattice forming FeOOH secondary phase and deactivating the electrocatalyst.⁹ Berlinguette et al. found that at 200 mA.cm^{-2} in strong base electrolytes, NiFeO_x OER electrocatalyst had a similar degradation problem as a significant amount of Fe left the structure and diffused to the cathode.¹⁰ Furthermore, Markovic et al. investigated the activity and stability trends for monometallic oxyhydroxides and observed that Fe is the least stable among Ni and Co.¹¹ These findings motivated us to develop an Fe-free electrocatalyst with the goal of an earth abundant OER electrocatalyst that is both active and stable in alkaline conditions. Among the best candidates to replace Fe, Co emerged as a promising option because – when used in combination with NiO_x – it lowers the overpotential and improves stability.^{12,13} In addition to Co, chalcogenides namely sulfides, tellurides, and selenides are known to enhance the activity when used with mixed transition metals compared to oxides. The improved activity has been attributed to an increase in covalency in the metal-chalcogenide bond due to the smaller associated electronegativity and better conductivity.^{14–21} However, several reports have shown that S and Se, in particular, are thermodynamically unstable at high pH during OER and ascribed the outstanding activity to increased nanoporosity in the structure,

disordering in the lattice, or high number of defective surface sites after S and Se ions leave the structure.^{22–24}

In this work we will use cryomilling in addition to a chalcogenide (Se) scaffold to synthesize nanocrystalline Ni-Co-Se with high concentrations of Co in Ni. Making such materials has previously proven challenging due to the immiscibility of the constituent elements. The role of Se on the OER activity will be studied using operando X-ray absorption spectroscopy (XAS) and high-resolution transmission electron microscopy (TEM) and complemented by density functional theory (DFT) studies.

First, cryomilling, ball milling at cryogenic temperatures, will be used to synthesize the Ni-Co-Se since milling at such low temperatures provides us with control over a wide compositional range of amorphous mixed-transition-metal compounds that are reported to be more active and stable than their crystalline counterparts.²⁵ Amorphous metals have been produced by a variety of methods: atomization, electrodeposition, electroless deposition, planar and flow casting. However, alloys produced by these methods are limited to compositions near low melting point eutectics unlike cryomilling which has a broader compositional range centered about high melting point intermetallics. Accordingly, targeting high melting point Ni-Co-Se favors disordering and the formation of abundant defective and coordinatively unsaturated sites (CUS) that are known to be catalytically active and can boost the overall performance of the OER electrocatalyst.^{26–28} Second, we will activate Ni-Co-Se in situ during OER and evaluate its half-cell activity and stability at high current densities. Finally, the overall performance of Ni-Co-Se as OER electrocatalyst against industrial requirements for water splitting and CO₂R in 5 cm² AEM electrolyser.

Main Text

Synthesis and activation of the electrocatalyst.

(NiCo)₃Se₄ and (NiCo)Se were synthesized using a two-step ball milling process consisting of: 1) mechanical alloying of two or more elements at a cryogenic temperature (< -196 °C) to produce disordered crystal structures and 2) surfactant-assisted ball milling (SABM) at room temperature to reduce the size of the alloy to nanoparticles (**Figure 1a**). The combination of high-speed mixing (30 Hz) and low temperature (-196 °C) facilitates the formation of nanocrystalline structures centered at high-melting point compositions (see supplementary information for synthesis description).

First, Ni-Se and Co-Se baseline samples were successfully synthesized to understand the individual milling behaviour of Ni and Co with Se. We found that despite the possibility of particles welding together due to excessive collisions during milling, Se played an important role in stabilizing single phase nanoparticles (<100nm) with Ni and Co (see supplementary information and Figures S1 and S2 for details on synthesis and characterization). Afterwards, we synthesized two Ni-Co-Se compounds centered at high-melting point composition: (NiCo)Se and (NiCo)₃Se₄ (**Figure 1b**). The broad diffraction peaks in XRD indicated that nano-sized crystallites (1.71-1.9 nm) were produced. The particle size distribution of Ni-Co-Se compounds had an average size greater than 1 μm and a small surface-to-

volume ratio, which is undesirable for efficient electrocatalysis. Therefore, a surfactant was used in SABM step to reduce the particle size of the alloyed compounds to 37 nm for (NiCo)Se and 67 nm for (NiCo)₃Se₄ (**Figure 1c**). Finally, the surfactant was removed by centrifuging at 10'000 rpm (11'000 rcf) for 1 hour and nanoparticles were collected for characterization. In summary, Se plays an important role in the size, morphology, and crystal structure of the starting composition of the cryomilled alloy. Cryomilling Ni-Co without Se produces large (> 6 μ m) crystalline particles which are not amorphized even under prolonged milling times; when Se was milled with Ni and Co, the crystal structure of the alloy was disordered while the size of the particles was significantly reduced, exposing a large active area of unsaturated defective sites for the OER.

According to the Pourbaix diagram of Se, Se oxidizes to soluble SeO₄²⁻ at high pH. This has been reported to facilitate the complete transformation of Ni and Co to disordered oxyhydroxides rich in active sites (**Figure 2a**).^{12,23,29} In our work we activated (NiCo)Se and (NiCo)₃Se₄ electrochemically by oxidizing and selectively leaching Se out of the nanocrystalline structure. The activation process was conducted in a three-electrode cell in 1M of pre-electrolyzed KOH, to purify the electrolyte from Fe ions that could participate in the reaction (**Figure S4**), and initiated by applying a constant current density of 10 mA.cm_{geo}⁻² for 2 hours, a sufficient time to warrant a complete removal of Se.²²

Scanning electron microscopy (SEM) images were taken for (NiCo)₃Se₄ before (dry) and after (post) activation (**Figure 2b**). Clusters of nanoparticles covered the entire carbon fiber in the dry sample and Se accounted for 56 at.% in the structure. After activation, the morphology of the clusters was altered and Se was completely removed (0 at.%, as confirmed by EDS), while the atomic ratio between Ni and Co remained unchanged (1:1). TEM and electron energy loss spectroscopy (EELS) mapping were conducted on dry (**Figure 2c**) and activated (post) samples (**Figure 2d**). EELS maps confirmed that Ni and Co were homogeneously distributed in *ca.* 10 nm nanoparticles without phase segregation and without oxidation. This was also confirmed using scanning transmission electron microscope (STEM) and energy dispersive spectroscopy (EDS) (**Figure S3**). After activation, EELS spectrum showed no contributions from Se while Ni and Co oxidized, indicating that Se had been completely leached out of the structure during electrochemical activation.

To further support these observations, soft X-ray absorption spectroscopy (sXAS) was conducted to investigate the local chemical and electronic changes to individual Ni, Co and Se atoms after activation. The X-ray absorption near edge structure (XANES) of Se L-edge confirmed that Se was incorporated in the dry nanoparticles as selenide (Se²⁻) and then completely left the structure during activation (post) as inferred by the lack of the Se L-edge signal after 2 h (**Figure 2e**).³⁰ The oxidation states of Ni and Co were inspected after activation as shown in XANES of L₃-edge results (**Figures 2f and 2g**). Two different modes of X-ray detection were used to investigate the oxidation state: fluorescence X-rays using silicon drift detector (SDD) and total electron yield (TEY). The TEY mode is dependent on the conductivity of the surface but is more surface sensitive (<10 nm) compared to SDD (within 100 nm). Using both detection modes allowed us to investigate the changes in oxidation state within the bulk and on the surface of the

electrocatalysts before and after activation. The XANES of Ni L₃-edge of the dry sample revealed that Ni had an oxidation state of 2+ before activation and increased to 3+ after activation suggesting the transformation of Ni to NiOOH.³¹ Co had a mixed oxidation state of 2+/3+ on the surface implying that Co was in a spinel structure.³² The oxidation state increased to 3+ after activation suggesting possibly the transformation of Co to CoOOH.³³

Electrocatalytic performance and stability

The performance of (NiCo)Se and (NiCo)₃Se₄ after activation was evaluated by measuring the overpotential at 10 mA.cm_{geo}⁻² on a glassy carbon electrode as in (**Figure 3a**). The activity of (NiCo)Se and (NiCo)₃Se₄ after SABM dramatically improved due to an increase in surface-to-volume ratio of the nanoparticles. Alloying Ni and Co in the electrocatalyst lowered the overpotential compared to NiSe and CoSe alone by *ca.* 50 mV confirming that Ni and Co have synergistic effects when alloyed together.^{12,34} The best performance was seen for (NiCo)₃Se₄ after SABM with an overpotential of 268±2 mV at 10 mA.cm_{geo}⁻² and a Tafel slope of 42 mV.dec⁻¹ compared to IrO₂ and NiFeOOH (**Figures S5 and S6 and Table S4**).

To examine the specific catalytic activity, we normalized the current density by the electrochemical surface area (ECSA) using double layer capacitance measured by cyclic voltammetry (CV) (**Figures S7 and S8**). (NiCo)₃Se₄ demonstrated ECSA current densities 44 times higher than NiFeOOH (prepared by Sol-Gel) and 4 times higher than commercial IrO₂ using the same catalyst loading of 0.21 mg.cm⁻² for all electrocatalysts (**Figure 3b**). The turnover frequency (TOF) of (NiCo)₃Se₄ was calculated as 50.76 x 10⁻³ s⁻¹ per site, considering only Ni and Co as active sites, *ca.* two folds higher than IrO₂ 28.09 x 10⁻³ and NiFe 23.98 x 10⁻³ s⁻¹ per site inferring that (NiCo)₃Se₄ is intrinsically more active.

We then investigated the stability of (NiCo)₃Se₄ using chronopotentiometry at 10 mA.cm_{geo}⁻² (Figure S9). (NiCo)₃Se₄ maintained a steady overpotential value for 100 h with only 4 mV of additional overpotential. At higher current densities and prolonged testing conditions, excessive oxygen bubbling can artificially increase the overpotential by shielding the active sites on the surface and within the pores of the electrocatalyst. Rotating disk electrodes (RDEs) are often used to overcome this problem and improves the detachment of bubbles from the surface of the electrocatalyst by rotation. However, vigorous rotation for long duration has been reported to affect the adhesion of the electrocatalyst to the surface of the RDE inferring fallacious conclusions about the intrinsic stability of the electrocatalyst.³⁵ Therefore, we proposed an alternating current test (ACT) to remove bubbles without mechanical rotation and better assess the intrinsic stability of the electrocatalyst. In ACT, a constant current density of 10 mA.cm_{geo}⁻² was applied to (NiCo)₃Se₄ for 10 h and then it was allowed to rest at OCP for 1 h to release O₂ bubbles on the surface before evaluating the performance using linear sweep voltammetry (LSV); this resembles one

cycle of testing (**Figure 3c**). The potential slowly increased during the first few hours of each cycle because of bubble accumulation on the surface, however the initial potential was retained in the following cycle suggesting that bubbles were partially or completely released. The test was repeated for 34 cycles (340 hours of operation). After each cycle, overpotential and Tafel slope were extracted and plotted with respect to time (**Figure 3d**). The electrocatalyst experienced a marginal increase in overpotential (<10 mV) during the entire test while the Tafel slope remained unchanged suggesting that the reaction mechanism was not altered.

The performance of $(\text{NiCo})_3\text{Se}_4$ on nickel foam (NF) was tested at high current densities, the overpotential was 279 mV at $0.5 \text{ A.cm}^{-2}_{\text{geo}}$ and 329 mV at $1 \text{ A.cm}^{-2}_{\text{geo}}$ (**Figure S10**). Next, the stability of the electrocatalyst was evaluated under constant current density (**Figure 3e**). The electrocatalyst had an average potential degradation of 0.17 mV.h^{-1} in 500 hours compared to 0.21 mV.h^{-1} for commercial IrO_2 . We attempted to compare the performance of activated $(\text{NiCo})_3\text{Se}_4$ with literature at $10 \text{ mA.cm}^{-2}_{\text{geo}}$ and high current densities ($>100 \text{ mA.cm}^{-2}_{\text{geo}}$). Over 69 earth-abundant electrocatalysts were listed in Table S8 and mapped using overpotential, Tafel slope, and electrochemical potential degradation as performance metrics (Figures S11). Activated $(\text{NiCo})_3\text{Se}_4$ had one of the best combined apparent (normalized by geometric area) and intrinsic (normalized by ECSA) activities at 10 mA.cm^{-2} on glassy carbon electrode (Fig. S11a). It also had the lowest electrochemical potential degradation rate of 0.17 mV.h^{-1} at $>100 \text{ mA.cm}^{-2}$ (**Figure 11d**).

Electronic structure of Ni-Co-Se and their influence on OER performance

To investigate the active species during OER and study the dynamics of the electrochemical activation of $(\text{NiCo})_3\text{Se}_4$, in situ XAS on the K-edge of Ni, Co and Se was conducted on the sample at four conditions: dry, OCP, at 1.1 V, and during OER at 1.5V (**Figure 4**). XANES showed that Se, in the dry sample, was in selenide (2-) form (**Figure 4a**) while Ni and Co had a mixed oxidation state of 2+/3+ suggesting that they exist in a spinel structure (**Figures 4b and 4c**), confirming sXAS findings.^{36–38} During OER, Se was oxidized to Se (VI) at 1.5 V as evident by the emergence of the peak at 12,666 eV. The edge position of Ni and Co continuously increased to higher energy values indicating an increase in oxidation state to 3+.³⁹ The increase in the edge position of Ni was accompanied by a decrease in the white line intensity implying that the electrocatalyst evolved to a disordered octahedral structure upon oxidation.⁴⁰ Extended X-ray absorption fine structure (EXAFS) was conducted to investigate local structure changes in the vicinity of Ni, Co, and Se sites during OER (**Figures 4d, 4e, and 4f**). A progressive increase in the Se-O peak at 1.2 Å upon oxidation was observed confirming that Se was leaching out as SeO_4^{2-} . For Ni, the peaks at *ca.* 1.5 Å and *ca.* 2.5 Å corresponds to the single scattering path of Ni-O and Ni-Ni/Co/Se. The Ni-O shifts to the left at 1.38 Å during OER suggesting that Ni oxidized to the active $\gamma\text{-NiOOH}$ structure.⁴¹ Three peaks were observed for Co in the dry sample at $\sim 1.5 \text{ Å}$ from Co-O, while the path from Co-Co/Ni/Se was observed at two distances $\sim 2.5 \text{ Å}$, and $\sim 3 \text{ Å}$ suggesting that Co occupies an

octahedral site in the spinel structure.⁴² The Co-O peak at 1.43 Å after the OER confirms the formation of CoOOH.⁴³

The experimental results were supported by DFT calculations which suggested the possibility of SeO desorption due to the observation of short Se-O interatomic distances within the oxide structure on both NiSe and NiCoSe surfaces (**Table S6**). Here, we compared the potential energy differences associated with SeO desorption and the formation of the peroxide intermediate (OOH); both processes have the oxide intermediate as a reactant. While SeO desorption was energetically favoured on the NiSe (001) and (101) surfaces, it was not energetically favoured on NiCoSe (001) and (101) (**Figure 5b**). This suggests Se leaching is an entropically driven process. Furthermore, Se leaching was less favoured near a Co atom. This reduced favourability of Se leaching near a Co atom likely occurs because the Se atom stabilizes the electron hole created by substituting Co by Ni. No SeO formation and therefore possibility of desorption was observed at the oxide step for surfaces with a Se vacancy. The potential energy differences associated with the potential determining step do not decrease significantly with Co doping or a Se vacancy (**Figure S13 and S14**). The combination of Co-doping and a Se vacancy on the (101) surface, however, changed the potential determining step to the oxygen molecule desorption. The significantly higher O₂ desorption energy for this surface is due to the second oxygen atom also binding to two metal atoms. For this surface, the desorption broke three Ni-O bonds and one Co-O bond unlike other (101) surfaces investigated where, at most, two metal-oxygen bonds were broken.

The interatomic distances were also investigated through DFT calculations (**Figure 5c**). It shows that H₂O does not strongly interact with the (001) and (101) surfaces unless there is a Se vacancy. Regardless of Co-doping, the presence of a Se vacancy in conjunction with a Ni adsorption site causes the surface to strongly adsorb water to a surface Ni atom. The calculated Ni-O bond lengths of 2.169 Å for Ni-Se_{vac} and 2.265 Å for Ni-Co-Se_{vac} on (001) surface and 3.525 Å for Ni-Se_{vac} and 3.266 Å for Ni-Co-Se_{vac} on (101) surface, are typical of hydrate complexes, where experimental values for these distances range from 2.053 Å to 2.127 Å.^{45,46} The Se vacancy on a Ni-Se or Ni-Co-Se (001) surface allows a stronger Ni-O interaction as indicated by shorter Ni-O distances for all OER intermediates (**Table S5**). The Ni-O interatomic distances vary from 2.135 Å to 2.179 Å except at the O intermediate, which has a Ni-O distance of 1.942 Å, suggesting this intermediate formed NiO_x rather than NiOOH.⁴⁷ For the (101) surface, the range of Ni-O distances during OER is between 1.797 Å and 2.101 Å for all surfaces except at Co-site on the Ni-Co-Se surface. The distinction between a Ni and a Co site is lost on the Ni-Co-Se_{vac} (101) surface due to the adsorbed oxygen species filling the Se vacancy and bridging a surface Co and Ni atom. The calculated Ni-O bond lengths suggest a transformation to NiOOH structures as these values are consistent with EXAFS result, which have shown that Ni is coordinated to oxygen at 1.88 Å (γ -NiOOH).⁴⁸ For Ni-Co-Se_{vac} (001) and (101) surfaces, the range of Co-O bond length is between 1.726 Å and 2.107 Å indicating a transformation to CoOOH (**Table S7**).⁴³ Se vacancies on certain surfaces permit two-site binding mechanism steps because there is no Se atom obstructing the O bonded to different metal atoms. Alternatively, a Se vacancy is a step towards forming a NiO_x surface as suggested by the

calculated results for the (101) Ni-Co-Se_{vac} surface. Originally the Se atom coordinates with three surface metal atoms and one metal atom in the layer below. As shown by the adsorbed molecular oxygen structure, the single Se is replaced by two O atoms (**Figure S14**). Both O atoms form bonds with surface metal atoms with bond lengths between 1.736 Å and 1.888 Å. This suggests that the formation of the active NiO_x layer is a two-step process. The first step removes surface Se at the oxide intermediation of the OER cycle and the second step rearrange the surface to form a NiO_x layer.

AEM water and CO₂R electrolyser

To further evaluate the performance of (NiCo)₃Se₄ as an OER electrocatalyst for industrial water splitting and CO₂R, we used a 5 cm² AEM electrolyser with Pt/C on carbon paper for hydrogen evolution and Cu on PTFE for CO₂R (**Figures 6a and 6d**). The polarization curves (without iR correction) indicated that (NiCo)₃Se₄ lowered the cell voltage to 2 V at 2 A.cm⁻² for water splitting (**Figure 6b**), which outperforms commercial IrO₂, and 3 V at 1 A.cm⁻² for CO₂R for (**Figure 6e**) using 1 M KOH. The performance was stable for 50 h at 1 A.cm⁻² of water splitting (**Figure 6c**) and 5 h at 0.5 A.cm⁻² of CO₂R (**Figure 6f**). These results demonstrated the potential of utilizing Cryomilled (NiCo)₃Se₄ for industrial applications

Conclusions

In summary, we have reported in this work the synthesis of homogenously dispersed Ni-Co-Se nanocrystals with varying compositions using a two-step milling process. Electrochemical leaching of Se from the structure enabled the transformation to Ni/Co-oxyhydroxides as validated by DFT, TEM, sXAS, and in situ XAS. The best performing activated (NiCo)₃Se₄ electrocatalyst showed a stable OER performance drawing 10 mA.cm_{geo}⁻² at an initial overpotential of 268 mV in 1M Fe-free KOH and maintaining this current for 350 h with a marginal degradation in electrochemical potential (<10 mV). At higher current densities (0.1-1 A.cm⁻²), the electrocatalyst exhibited a stability of 0.17 mV.h⁻¹ comparable with state-of-the-art IrO₂ (0.21 mV.h⁻¹). Using (NiCo)₃Se₄ as OER electrocatalyst in AEM electrolyser, we could demonstrate outstanding water splitting performance delivering 2 A.cm⁻² at 2 V and 1 A.cm⁻² at 3 V. The results encourage the utilization of cryomilling and chalcogenides such as Se to mass produce efficient earth-abundant electrocatalysts with tailored nanostructures and long-term durability

Declarations

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Figures

Figure 1

Schematic illustration of the two-step synthesis process a) step 1: milling at a cryogenic temperature -196 °C to alloy elements, step 2: milling in wet environment using surfactant to reduce the size of the alloyed

particles. b) XRD of (NiCo)Se and (NiCo)₃Se₄ after 6 hours of cryomilling. c) Particle size distribution for (NiCo)Se and (NiCo)₃Se₄ after SABM for 10 hours followed by centrifuging at 6000 rpm for 1 hour.

Figure 2

a) A schematic of the activation process for Ni-Co-Se alloys. b) BSE-SEM images and c) and d) TEM images of the (NiCo)₃Se₄ catalyst at 0 h (dry) and after 2 h of activation at 10 mA.cm⁻² (post). The

elemental content of the catalyst is shown in the inset tables. Soft X-ray L-edge XANES of e) Se, f) Ni, and g) Co using TEY and SDD.

Figure 3

a) iR-corrected polarization curves on glassy carbon electrode with a loading of 0.21 mg.cm⁻². b) Specific activity of (NiCo)₃Se₄ and (NiCo)Se compared to IrO₂ and NiFe, normalized by ECSA at 300 mV overpotential (left axis) and normalized by geometric area (right axis). The roughness factor (RF) value

for each catalyst is noted at the top of the bar. c) (NiCo)3Se4 NP was deposited on carbon paper and tested using alternating stability test. Inset is a schematic illustration of O2 bubble accumulation on the surface of the electrocatalyst during oxidation. c) The change in key electrochemical parameters every 10 hours during the alternating stability test. d) a) Activated (NiCo)3Se4 was deposited on Nickel foam and tested for long term stability at high current densities. All tests were conducted in 1M Fe-free KOH electrolyte.

Figure 4

In situ XANES K-edge of a) Se, b) Ni, and c) Co of (NiCo)3Se4 during OER. In situ EXAFS radial distribution function of d) Se, e) Ni, and f) Co of (NiCo)3Se4 during OER. Interatomic distances noted in the figures are shorter than real interatomic distances because Fourier Transform (FT) spectra were not phase corrected. All tests were done in 1M KOH.

Figure 5

a) OER mechanism on the NiSe (001) surface, the Se vacancy is highlighted in magenta, Co doping sites are given in green, and Ni and Se are shown in blue and yellow, respectively. b) Comparing the energies of OOH formation and SeO desorption. c) Oxygen bond length changes during OER for NiSe, NiSevac, NiCoSe, and NiCoSevac using (001) and (101) surfaces.

a AEM Water Electrolyser

d CO₂ Reduction

Figure 6

a) Schematic diagram of AEM water splitting b) Polarization curve and c) Chronopotentiometry test using 1 mg.cm⁻² Pt/C on carbon paper for HER and 2 mg.cm⁻² (NiCo)₃Se₄ on Nickel foam for OER compared to commercial IrO₂. d) Schematic diagram of AEM water splitting. e) Polarization curve and f) Chronopotentiometry test using 1 mg.cm⁻² Cu on PTFE for HER and 2 mg.cm⁻² (NiCo)₃Se₄ on Nickel foam for OER compared to commercial IrO₂.

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