Activity and Regeneration of Electrodeposited Fe-Ni-Co-Based Electrocatalysts for the Alkaline Oxygen Evolution Reaction

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Activity and Regeneration of Electrodeposited Fe–Ni–Co-Based Electrocatalysts for the Alkaline Oxygen Evolution Reaction

Yujia Zhang, Orhan Kizilkaya, Hubert K. Bilan, Richard Kurtz, and Elizabeth J. Podlaha*

ABSTRACT: A methodology to recover the oxygen evolution reaction (OER) kinetics of aged, electrodeposited Fe–Ni–Co thin films is presented. The electrodeposits were fabricated galvanostatically onto rotating copper electrodes and contained a small amount of Co. The OER characterized in 1 M KOH was a function of the deposit composition. The Tafel slope and the overpotential at 10 mA/cm² observed with ohmically corrected linear sweep voltammetry were both a function of the deposit metal ratio, Fe/(Ni + Co), with the lowest values occurring at a ratio of 1.2. An Fe-rich deposit was purposely altered upon aging to degrade the OER kinetics. The Tafel slope and overpotential were largely recovered when the aged electrocatalyst was treated with a cathodic current density of −100 mA/cm² for 10 min. Soft X-ray absorption spectroscopy provided insight into the changes of the electron density distribution upon aging and regenerating the electrocatalyst. Through X-ray absorption near-edge structure (XANES), the L-edge spectra of Fe, Ni, and Ni. X-ray absorption near-edge structure (XANES), the L-edge spectra of Fe, Ni, density distribution upon aging and regenerating the electrocatalyst. Through absorption spectroscopy provided insight into the changes of the electron density distribution upon aging and regenerating the electrocatalyst. Through X-ray absorption near-edge structure (XANES), the L-edge spectra of Fe, Ni, and K-edge spectra of O, suggested that the recovery is due to the redistribution of the electron density from the oxygen to Fe and Ni.

KEYWORDS: water splitting, OER, electrodeposition, regeneration, Fe–Ni–Co, stability

The oxygen evolution reaction (OER) can limit the efficiency of hydrogen (H₂) production from water due to its sluggish kinetics and hence requires electrocatalysts to lower the activation overpotential. While precious metals and their oxides, such as IrO₂ and RuO₂, are recognized as superior OER catalysts, they are costly and rare. To this end, earth abundant Fe–Ni-based materials, such as oxides, (oxy)hydroxides, and layered double hydroxides (LDHs), are well-recognized alternatives. For example, Ni oxide films doped with even a small amount of Fe (1 wt %) significantly improved the OER kinetics, with low Tafel slopes (e.g., 20–25 mV/dec) reported for Fe contents between 10 and 50 wt %. Merrill and Dougherty observed even smaller Tafel slopes (14.8 mV/dec) of electrodeposited NiFe oxides, containing NiFeO₄ phases and showing magnetic character. Corrigan and Bendert examined the coprecipitation of up to 13 metal dopants to a nickel hydroxide where Ce, Fe, and La additions further promoted OER. An optimum Fe content of 20 at. % in FeNi(OH)₂ electrocatalysts was observed by Li et al., for lowering the overpotential. Nanostructured Fe–Ni LDHs on mildly oxidized multiwalled carbon nanotubes demonstrated excellent OER kinetics, with the OER Tafel slope and overpotential (at 5 mA/cm²) as low as 31 mV/dec and 250 mV, respectively. A PH₃ plasma treated Ni–Fe LDH was used to create an amorphous NiFe hydroxide/NiFeP layer having strong electronic interactions between the layers to lower the adsorption energy of H₂O resulting in enhanced OER performance.

Recently, OER electrocatalysts based on the ternary mixed-metal, Fe–Ni–Co, were also reported in alkaline electrolytes, and in some cases, these studies reported a lower OER overpotential than Fe–Ni. Li et al., for example, electrodeposited Fe–Ni–Co thin films from electrolytes of various concentrations, targeting a range of deposit composition. Their results showed that, with a small amount of Co (~12 at. %), a Ni-rich deposit exhibited an OER Tafel slope of 45 mV/dec, and an OER overpotential of 310 mV at 10 mA/cm². In contrast, Smith et al. found that Fe–Ni oxides were generally more active than Fe–Ni–Co oxides. However, the Fe–Ni–Co oxides examined in their study contained at least 20 at. % Co. When the Fe composition was increased. Stevens et al. found that a Co addition in the Fe-doped NiOOH enhances only electrical conductivity with no improvement in intrinsic OER. However, Thenuwara et al. found that a Co addition in the Fe-doped NiOOH enhances only electrical conductivity with no improvement in intrinsic OER.
reported Co-intercalated Ni–Fe oxides to be more active than their Ni–Fe counterparts due to lower-energy reaction intermediates calculated via density functional theory (DFT), and Bates et al.\textsuperscript{21} attributed improved enhancement with Co due to the formation of the conductive Ni\textsuperscript{III}OOH phase at a lower overpotential, that activates the Fe sites.

Degradation studies of OER transition-metal-based catalysts are commonly investigated with chronopotentiometry/chronoamperometry,\textsuperscript{5,6,19,28,31–33} cyclic voltammetry,\textsuperscript{6} and reported deposition composition changes following a stability test.\textsuperscript{32} For example, McCrory et al.\textsuperscript{3} compared the stability of different transition metal oxides at 10 mA/cm\textsuperscript{2} for 2 h. In a basic electrolyte, the overpotential of the CoFeO\textsubscript{x} electrocatalyst significantly shifted compared to those of the more stable NiFeO\textsubscript{2} and NiCoO\textsubscript{x} deposits. Burke et al.\textsuperscript{33} observed that the stability and mass loss were dependent upon composition in Co\textsubscript{1−x}Fe\textsubscript{x}(OOH) films. The OER current density on Co\textsubscript{1−x}Fe\textsubscript{x}(OOH), when x < 0.5, decreased by half when the sample was chronoaamperometrically aged at an overpotential of 350 mV for 2 h; with large amounts of iron, local regions of FeOOH were present and subsequently dissolved during electrolysis. The dissolution rate increased with overpotential.\textsuperscript{35} Moysiadou and Hu\textsuperscript{32} investigated the OER stability of CoO\textsubscript{y}, CoFeO\textsubscript{y}, CoFeNiO\textsubscript{y}, NiFeO\textsubscript{y}, and NiFeO\textsubscript{2} via both chronopotentiometry (at 5 mA/cm\textsuperscript{2}) and chronoaamperometry (at 1.58 V vs RHE) for up to 14 h with metal ions present in the electrolyte. They noted a dynamic exchange between the solid state of the OER electrocatalyst and metal ions in the alkaline electrolyte and demonstrated that the activity can be retained through this exchange. Electro catalysts containing all three elements Ni, Fe, and Co, demonstrate favorable stability. Li et al.\textsuperscript{36} reported a stable current response of the electrocatalysts with an applied overpotential of 250 mV for up to 8 h to confirm stability. Wang et al.\textsuperscript{28} showed that under a constant current density of 10 mA/cm\textsuperscript{2} a bifunctional Ni–Fe–Co layered triple hydroxide electrocatalyst used as both the anode and cathode resulted in a steady cell potential for up to 19 h of continuous electrolysis. Smith et al.\textsuperscript{20} showed that both a-Fe\textsubscript{90}Co\textsubscript{10}Ni\textsubscript{50}O\textsubscript{30} and a-Fe\textsubscript{90}Ni\textsubscript{10}O\textsubscript{30} had just small increases in potential to maintain the current density over 24 h. Bates et al.\textsuperscript{21} demonstrated the stability of the ternary Ni–Fe–Co on a Raney Ni substrate toward OER during 1 h of continuous operation at an applied potential of 1.54 V, with current fluctuations having a small standard deviation of 0.07 mA/cm\textsuperscript{2}. These studies are used as a guide to establish a lower limit for aging the electrocatalyst. Herein, results are presented where the electrocatalysts are aged in excess of 24 h to purposefully age the electrocatalyst as evidenced by a larger overpotential and Tafel slope. The novelty of the present work is in regeneration of the aged electrocatalyst by applying a cathodic current.

Fe–Ni–Co thin films were electrodeposited galvanostatically from a sulfate/sulfamate–boric acid electrolyte (details in Supporting Information, Table S1), adapted from Kim et al.\textsuperscript{36} and Geng et al.\textsuperscript{37} Copper rotating cylinder electrodes (RCEs) having a diameter of 1 cm and length of 1.2 cm served as the working electrode with Pt as the counter electrode. The RCE is not flush with the top and bottom insulating regions of the plastic holder, but it is recessed, making a right angle between the plastic holder and the copper electrode, to facilitate a uniform current density distribution. The rotation rate of 1000 rpm was used to control the electrolyte mixing in the vicinity of the electrode. A range of applied current densities were selected to deposit Fe–Ni–Co thin films. Table 1 summarizes the deposition conditions, composition, and thickness. The deposition time of each sample was adjusted to target a similar deposit thickness. The bulk composition and thickness were measured using a Kevex Omicron X-ray fluorescence (XRF) instrument. Fe-rich and Ni-rich samples, with a small amount of Co, were realized, having a thickness of 0.51 ± 0.05 μm. To confirm a uniform deposit distribution along the electrode surface, the local deposit composition was determined along the RCE length with XRF and energy dispersive spectroscopy (EDS) with scanning electron microscopy (SEM) elemental mapping (Supporting Information, Table S2 and Figure S1). The Fe, Ni, and Co wt % was found to have a standard deviation of 1.0, 0.8, and 0.5 wt %, respectively. X-ray diffraction (XRD) of the as-deposited Fe–Ni–Co thin films presents reflections that are characteristic of a Ni-rich phase, (111), (200), and (220), and a more crystalline Fe-rich phase, (110) and (200); see Supporting Information, Figure S2.

Figure 1 shows the ohmically corrected polarization behavior of the thin film electrocatalysts in 1 M KOH (at room temperature), the linear sweep voltammetry (LSV) Tafel regions of the Fe-rich (A–C) and Ni-rich (D and E) Fe–Ni–Co deposits, a comparable, Co-free, Fe–Ni electrocatalyst (F), having a similar Fe/Ni weight percentage ratio as deposits fabricated with condition C, with the deduced Tafel slopes and overpotential at 10 mA/cm\textsuperscript{2}, and an assessment of the electroactive surface area. The polarization was initiated at the open circuit potential (OCP) condition and polarized anodically to the OER region. As the potential is swept in the anodic direction, the flat region and the large peak at −0.25 V vs SCE is indicative of a mixed-oxide formation. The smaller peak at +0.35 V vs SCE has been noted to be a signature for nickel hydride restructuring observed in Fe–Ni systems, Ni(OH)\textsubscript{2} + OH\textsuperscript{−} → NiOOH + H\textsubscript{2}O + e\textsuperscript{−}.\textsuperscript{38} With the incorporation of cobalt hydroxide, Ni-Co_1-x(OH)\textsubscript{2}, this peak is reported to shift to a more negative potential.\textsuperscript{39} Such behavior is observed here with an increase of cobalt in the deposits, for conditions C, E, and D. At large, positive applied potentials, the fluctuating current response reflects the high OER and gas bubble dynamics on the catalyst surface, Figure 1a,b. The Tafel plots of the OER region from Figure 1a are shown versus an RHE scale (E\textsubscript{RHE} = E\textsubscript{SCE} + 0.242 + 0.059pH) (Figure 1b). At low current densities, the Fe–Ni–Co deposit Tafel slopes are all similar to the Fe–Ni electrocatalyst Tafel slope. However, at higher current densities, the Tafel region of the Fe–Ni electrocatalyst exhibits two distinct Tafel slopes, becoming unfavorably larger at higher overpotentials. In contrast, all the Co-containing deposits retained their Tafel slopes up to a

<table>
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<th>condition</th>
<th>current density (mA/cm\textsuperscript{2})</th>
<th>deposition time (s)</th>
<th>thickness (μm)</th>
<th>Fe (wt %)</th>
<th>Ni (wt %)</th>
<th>Co (wt %)</th>
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<td>720</td>
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<td>54</td>
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Figure 1. (a) LSV curves and (b) Tafel plots of Fe-rich (A–C), Ni-rich (D and E), and Co-free (F) Fe–Ni–Co electrodeposits in 1 M KOH. (c) Resulting Tafel slopes and overpotential vs RHE at 10 mA/cm²; standard deviations of $\eta$ and $b$ were 0.006 V and 1 mV/dec, respectively. (d) Cyclic voltammetry of deposits in a 0.02 M potassium ferricyanide electrolyte to assess active surface area.

Figure 2. Fresh, aged, and aged + treated Fe$_{54}$Ni$_{37}$Co$_{9}$ deposits on (a) LSV curves, (b) Tafel curves extracted from LSV vs RHE, (c) overpotential and Tafel slopes uncorrected for surface area changes, and (d) ferri/ferrocyanide CV curves that reflect the electroactive areas.
current density of 100 mA/cm². Larger amounts of iron in the deposit were not beneficial in minimizing the overpotential in the OER region, as evidenced by the performance of deposits fabricated with conditions A and B, containing 71 and 65 wt % Fe, respectively. These results are consistent with others where too much iron leads to a lower activity, presumably due to segregated FeOOH phases. Figure 1c summarizes the OER overpotentials (gray), $\eta$, at a current density of 10 mA/cm², and the OER Tafel slopes (black), $b$. The standard deviations of $\eta$ and $b$ were 0.006 V and 1 mV/dec, respectively, obtained through LSV scans on three deposits from condition C in Table 1. Both parameters are plotted as a function of the ratio of Fe deposit bulk composition to Ni and Co, Fe/(Ni + Co) (w/w). The electrodeposit that is slightly Fe-rich outperforms all other deposits for OER activity, exhibiting $\eta$ as low as 0.31 V vs RHE to reach 10 mA/cm², and $b$ as small as 33 mV/dec. The Tafel slope is independent of the catalyst surface area, and thus, a lower value reflects inherent energetic improvement of the electrocatalytic performance and is a descriptor of the rate-determining step in the mechanism.

The overpotential is an area-dependent parameter, and in order to evaluate the intrinsic performance of the catalysts, the electroactive surface areas were characterized. A protocol involving the measurement of the double-layer capacitance using a cyclic voltammetry (CV) technique in a nonfaradaic potential region is widely used as an electrochemical estimation of the active area. However, as Burke et al. pointed out, this method fails for electrodeposited (oxy)hydroxide films with a hydrated surface, due to the ion-permeable nature of the hydrated films that allows ions to polarize against the underlying metal. The same challenge is expected for the electrodeposited Fe−Ni−Co thin films, as the deposit surfaces are converted to hydrous oxide films when exposed to electrolytes at high pH. Therefore, CVs with an electrolyte containing potassium ferricyanide are reported here. The peaks of ferricyanide reduction and ferrocyanide oxidation reflect the electroactive surface area of the deposits. Figure 1d presents the CV results for all the deposits in an unstirred electrolyte (electrolyte composition is provided in Supporting Information). The peaks of the CV, indicative of the transport limited ferri/ferrocyanide reactions, are comparable, indicating that Fe-rich and Ni-rich deposits have almost identical active surface areas. Therefore, comparing the LSV curves (Figure 1a,b) and overpotentials (Figure 1c) with the ferricyanide/ferrocyanide CV results (Figure 1d) confirms that the overpotential at a particular geometric current density is indeed lower for slightly Fe-rich, Fe−Ni−Co electrodeposits and thus is intrinsically less energy intensive for the OER. The overpotential behavior reflects the Tafel slope showing that too much Fe in the deposit slightly increases the overpotential.

A regeneration of aged electrocatalysts was examined with electrodeposits from condition C in Table 1. Electrocatalyst aging was induced by applying an OER current density of 10 mA/cm² for 30 h (Figure 2). Figure 2a,b compares the OER polarization curves of the deposit before (green solid line) and after (blue dash line) aging. The aged deposit clearly shifts to larger overpotentials and has a larger Tafel slope, without accounting for surface area changes. When accounting for the surface area through the ferri/ferrocyanide measure, there is an effective surface area increase of 12%; thus, the OER overpotential of the aged catalyst is even worse than what is measured with a geometrical area. Interestingly, it is noted that when a cathodic current density of −100 mA/cm² was applied to the aged deposit for 10 min, the OER LSV curve was almost recovered to that of the fresh one, as shown by the dotted line (red) in Figure 2a,b. In addition, Figure 2d indicates that the active area of the aged Fe−Ni−Co thin film was also restored.
Therefore, the OER activity of the catalyst can be recovered by this simple reduction process.

XRF measurements show that the composition of the fresh, aged, and regenerated deposits did not significantly differ (Supporting Information, Table S3). EDS spectra taken over a large area, in the region depicted by the SEM (Figure S3) at low magnification, 500X, were consistent with the bulk XRF analysis. No significant changes of porosity were observed at high magnification, 100,000X.

To further understand the mechanism for the improved OER kinetics and correlate the findings acquired from the electrochemical approach to the electronic structure of the electrodeposits, soft X-ray absorption spectroscopy was employed. Figure 3 shows the X-ray absorption near-edge structure (XANES) L-edge spectra of (a) Fe, (b) Ni, (c) Co, and (d) O of the fresh deposit; aged deposit from a 30 h electrolysis; and the aged deposit followed by a reducing current density, with selected reference spectra, with details provided in Supporting Information. Since the 2p → 3d L-edge transition is a dipole-allowed excitation with a strong 2p → 3d wave function overlap, the XANES L-edge spectrum provides a significantly higher intensity than the metal K pre-edge with higher resolution detailing the fine local electronic structure and is better suited for analysis of the electronic composition of the empty d orbitals.41 While the values of reference spectra are useful to identify pure oxide and oxy-hydroxide species, there are expected spectral overlaps for various compounds, such as Fe(III) compounds that vary predominantly in peak shape and satellite intensities. The Fe L-edge spectra of an Fe2O3 reference corresponds well with all samples, consisting of L3 (∼709 eV) and L2 (∼721 eV) spin–orbit split components, that arise from Fe 2P3/2 and 2P1/2 core electron transitions into the unoccupied 3d energy levels of Fe which are split into t2g and eg levels due to the crystal ligand field. Thus, the deposits are predominantly in an Fe(III) oxidation state. The intensity of the peak related to eg states in the XAS spectrum of the aged deposit is higher compared to the other two, which might indicate that more unoccupied d-holes/states are generated by aging.

Figure 3b compares the XANES spectra of the Ni L-edge of the deposits. The Fe–Ni–Co electrodeposit presents very similar spectral features and energy positions of the peaks to those of a NiO reference. Thus, a Ni(II) valency is predominant for each sample. The same trend is observed for the aged deposit, with an enhanced intensity compared to the fresh deposit and the treated, reduced deposit.

The energy positions of the Co L-edge of Co metal and CoO are very close; however, the spectral shape/features are quite different. The deposits show a spectral shape similar to that of Co metal foil, thus with more of a metallic behavior. However, the lower shoulder (∼776 eV) can indicate that there is some oxide present. Interestingly, the aged sample shows features that stem from M4 (783.8 eV) and M5 (799.2 eV) transitions from barium, which do not appear in the fresh and post-treatment spectra, which may indicate a change of porosity with barium as a substrate impurity. However, the higher magnification SEM images (Figure S3) do not show appreciable porosity changes, but the electrochemical surface areas of the aged deposits are slightly larger (Figure 2d), suggesting that the surface is more porous at a scale much below 100 nm. In Figure 3, the intensities are normalized, but the Co L-edge intensities were noted to be significantly lower than those of Fe and Ni, due to a small amount of Co on the surface or in the subsurface region. In addition, the same stability study was performed on the Co-free, Fe–Ni electrocatalyst (condition F in Table 1), and the results are shown in Figure 4. Similar trends of deactivation and regeneration behaviors are observed in the absence of Co. Thus, it is most likely that the role of Co on the electrocatalyst recovery is insignificant, perhaps due to the metallic nature of Co.

XANES data in Figure 3d present the O K-edge spectra of the electrodeposits with Fe2O3 and NiO for reference. The features between 529 and 535 eV reflect the transitions from the oxygen 1s electronic level to its 2p states hybridized with the transition metal 3d states, while the resonances that appear above 535 eV are attributed to the transitions to oxygen 2p states hybridized with the transition metal 4s and 4p states. Comparing the spectra of the deposits with the reference spectra, the pre-edge structures indicate that they are predominantly composed of the oxygen states hybridized with Fe (the bands under double peak features of Fe2O3) and Ni (the peak at 532.8 eV) d states. Here, the opposite behaviors in the intensities are observed compared to the results obtained from Fe and Ni L-edge spectra. This reveals that the aged sample presents a lower pre-edge intensity compared to those of the fresh and reduced films, indicating that there is a dehybridization of the transition metal and oxygen states. This results in a net charge transfer to oxygen and a decrease in the empty oxygen states as revealed by the decrease in the intensity of the pre-edge peak.

Similar trends in the changes of XANES peak intensities were also observed by others; for example, Liu et al.42 reported that vanadium-doped (10%) NiS2 exhibited improved OER activity, which was associated with a lower intensity of the Ni L-edge peak as an indication of a decrease of the unoccupied state.

Figure 4. Fresh, aged, and aged + treated Fe59Ni41 deposits on (a) LSV curves and (b) Tafel curves extracted from LSV vs RHE.
electron density states, and thus a higher electron density at Ni sites. Also, Liang et al.\textsuperscript{43} associated such a decrease in a XANES metal L-edge for cobalt oxides with a higher electron density at metal sites due to a weaker Co–O ionic bond. Such charge redistribution also affects the conductivity of the material.\textsuperscript{44} Therefore, the observed changes in the XANES of Ni and Fe L-edge peaks may be associated with the chemical bonding environment of metal and oxygen atoms. Upon aging, there is a redistribution of electron density favoring the oxygen and creating stronger metal–O bonds decreasing OER activity. Following the cathodic reduction treatment, this redistribution is reset favoring the metal. These changes are also reflected in a change of conductivity, as evidenced by the lower magnitude of the flat current response in Figure 2a during passivation of the metal.

In summary, the electrodeposited Fe–Ni–Co–metal/oxide electrocatalysts improved OER kinetics over that of comparable Fe–Ni–Co electrocatalyst by retaining a low Tafel slope over a much larger current density range. Most importantly, an aged electrocatalyst, having a loss in Tafel slope and overpotential, was found to be regenerated by applying a cathode post-treatment. The reactivation was a result of the change in the chemical bonding environment of metal and oxygen atoms, with Fe and Ni playing an important role. Co was present predominantly in its metallic form.

### ASSOCIATED CONTENT

#### Supporting Information

The Supporting Information is available free of charge at https://pubs.acs.org/doi/10.1021/acsaem.0c00985.

Experimental conditions and bulk, elemental metal composition (PDF)

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### Notes

The authors declare no competing financial interest.

### ACKNOWLEDGMENTS

This work was supported in part from the US National Science Foundation (Grant 9977576) and Clarkson University. The authors acknowledge support in the form of beam time at the synchrotron at the Louisiana State University Center for Advanced Microstructures and Devices (CAMD).

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