Numerical Simulation of the anomalous electrodeposition of Nickel-Iron alloy on rotating disk electrode

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Abstract. Electrodeposition was originally used for the preparation of metallic mirrors and corrosion resistant surfaces. In this paper, we study a stationary one dimensional system modelling nickel-iron alloy electrodeposition. This system takes into consideration the following phenomena: dissociation, diffusion, electromigration, convection and deposition of multiple ion species. In this work, we present the finite element discretization of the main model supported with a numerical algorithm to effectively handle the nonlinearities. We compare the obtained numerical result with experimental data reported in the literature and show that our simulation method can predict the characteristic features of the Nickel-Iron system.

Keywords: Numerical analysis, Finite element method, Electrodeposition, reaction-diffusion, Nickel-Iron (NiFe) alloy

AMS Subject Classification : 97N40, 65M60, 35Q99.

1. Introduction
The electrodeposition of metals and metallic alloys has been used for nearly a century. It is currently used industrially in many application and combines both art and science. Among the advantages of Electrodeposition as a tool of material technology is its ability to grow films using a wide variety of materials including semiconductors and other materials for bio-stimulation. The main purpose of electrodeposition is to give metals some crucial properties they lack like wear resistance, corrosion protection (Using chromium electrodeposition which adds a corrosion resistant layer). Electrodeposition of Iron alloys is one of the most recent developments in the alloy domain due to their wide spectrum of physical properties. Its application range from recording, memory and storage devices because of the easily magnetized and demagnetized property of Permalloy, to watch springs, space technology, scientific instruments due to the negligible coefficient of expansion of Invar. Also, it is used as a decorative material. The electrodeposition of NiFe provides an additional challenge, as a possible appearance of an anomalous co-deposition. Investigations of the NiFe electrodeposition date back to 1927, when Glasstone and Symes studied plating of iron group metals to try to understand this anomalous co-deposition. In 1960, Brenner gave an extensive review summarizing bench of studies on NiFe electrodeposition. After that, many scientists tried to propose an explanation of the
mechanism of anomalous co-deposition. The most viable one was proposed in 1965 by Dahms and Croll, according to the experimental results they suggested that the outbreak of anomalous co-deposition matches with a rise in surface PH. Their conclusion was the increased hydroxide ion concentration near the surface should lead to the formation of ferrous hydroxide, when it is adsorbed on the cathode enabling the deposition of nickel and giving a high discharge rate of Fe$^{2+}$ (Iron oxide). Lately, mathematical models and experimental results exhibited anomalous behaviour even with low hydrogen evolution rates, which means that the rise in surface pH is not necessarily required for the occurrence of anomalous co-deposition.

The anomalous phenomena happen due to the presence of iron hydroxide (FeOH$^+$) is close to the electrode at higher concentration than nickel hydroxide (NiOH$^+$). In spite of the achievement of the works [2], and [3]. They found that even if when they applied this model on a rotating disk electrode, it still gives rise to anomalies for different cases. An effective model proposed by Maltosz in [4] required the reduction of each metal ion appears in two steps: with electro-absorption of an intermediate whose valence is equal to one in the first and its reduction in the 2nd one. The abnormal process results from the odsorption of the iron intermediate without having to consider the presence of nickel hydroxide and iron hydroxide. In [2] numerical approach using Cramer’s rule was proposed to provide a system of ordinary differential equations. They applied a mechanism for the anomalous co-deposition of NiFe in order to evaluate the sensitivity of the model. Alaa et al. in [6] studied the phenomenon for different types of models, boundary conditions and a variety of numerical approaches. In fact, their mathematical model used an electroneutrality condition on the species that we replaced here by a partial differential equation. We imposed mixed boundary condition while they considered a Dirichlet type for all the species. Furthermore, we suggest a finite element method discretization. Finally, we are more interested in the anomalous co-deposition of iron rather than the evolution of the concentration of the species. In this work, we studied a type of modelling similar to the one done by Pritzker et al. [5]. For this particular steady state model we didn’t find any well-posedness study. The layout of the paper is organized as follows, in the next section we describe the mathematical model of our main problem. Next, we exhibit our proposed numerical scheme. In the last section, numerical results are given accommodated by some interpretation and comparisons with experimental data. A conclusion is last in order to provide a summary and further remarks.

2. Modelling and Numerical scheme

2.1. Modelling

We worked with a co-deposition problem of NiFe in a solution of SO$_4^{2-}$. This particular process produces magnetic devices from permalloy. For this electrolysis bath nickel sulphate (NiSO$_4$), iron sulphate (FeSO$_4$) and boric acid (H$_3$BO$_3$) are dissolved in water and sulphuric acid (H$_2$SO$_4$) is used for the adjustment of PH. Therefore, the considered species in the solution are $A_1 = Ni^{2+}$, $A_2 = Fe^{2+}$, $A_3 = H^+$, $A_4 = SO_4^{2-}$ and $A_5 = HSO_4^-$. We consider the species conservation equation in one dimension case as

$$\frac{\partial w_i}{\partial t} = -\partial_x (J_i) + S_i,$$

(1)

Where, $w_i$ the concentration of species $A_i$, $S_i$ is the production rate, $J_i$ is its molar transport flux given by

$$J_i = -d_i\partial_x w_i + vw_i - m_i w_i \partial_x \phi,$$

(2)

Where, $d_i$ is the diffusion coefficient of the species, $v$ is the fluid velocity vector, $\phi$ the electric potential and $m_i$ the mobility or the species given as follows,

$$m_i = \frac{d_i z_i F}{RT},$$
Where, $z_i$ the valency of each species, $F$ the Faraday constant, $R$ the universal gas constant and $T$ the local temperature.

The electrical potential must satisfy the electroneutrality condition everywhere in the system,

$$-\partial_{xx} \phi = \frac{4\pi F}{\varepsilon_0} \sum_{j=1}^{5} z_j w_j,$$  \hspace{1cm} (3)

With, $\varepsilon_0$ is the permittivity.

We consider a one dimensional system in steady-state case of the species with simultaneous homogeneous reaction.

$$SO_4^{2-} + H^+ \overset{k_1}{\underset{k_{-1}}{\rightleftharpoons}} HSO_4^-,$$  \hspace{1cm} (4)

The above equation is the only homogeneous reaction considered in the solution. This reaction is described as bimolecular in the direct sense with $k_1$ is reaction rate constants in this sens, and monomolecular in the backward one, with $k_{-1}$ being its reaction rate constants.

Thus, the reaction terms are:

$$S_3 = -k_1 w_3 w_4 + k_{-1} w_5,$$

$$S_4 = -k_1 w_3 w_4 + k_{-1} w_5,$$

$$S_5 = k_1 w_3 w_4 - k_{-1} w_5$$

$$K = \frac{k_1}{k_{-1}}$$ are the equilibrium constant for homogeneous reaction and $w_i$, $i = 1, ..., 5$ are respectively the concentrations of $Ni^{2+}$, $Fe^{2+}$, $H^+$, $HSO_4^-$ and $SO_4^{2-}$.

At the electrodeposition surface we consider these two electrode reactions:

$$Ni^{2+} + 2e^- \rightarrow Ni,$$  \hspace{1cm} (5)

$$Fe^{2+} + 2e^- \rightarrow Fe,$$  \hspace{1cm} (6)

We consider all the species to be inert except for the hydrogen because its formation is involved only in the reduction of the protonium ion,

$$2H^+ + 2e^- \rightarrow H_2,$$  \hspace{1cm} (7)

Fluxes at the cathode surface $(x = 0)$ are given as follows,

$$J_k(0) = \frac{I_k}{2F} = -\beta_k w_k(0) exp(-\alpha_k z_k FV \frac{R}{RT}),$$  \hspace{1cm} (8)

With, $I_k$, $k = 1, 2, 3$ are the current densities for reactions (5), (6) and (7), $V$ is the cathode potential after ohmic correction and $\beta_k$, $\alpha_k$ are respectively the rate constants and transfer coefficients.

The considered bulk region is an area $\Omega$, the phenomenon occurs at the distance $L = 3\delta$ from the electrode surface, with $\delta$ is the hydrodynamic boundary layer thickness. According to Ying et al. (1988) in [11] $L = 3\delta$ is the limit surface for electrodeposition to take place, i.e above it the electrodeposition process does not happen.

In this model, we didn’t explicitly include the presence of boric acid. Under normal operating conditions for NiFe co-deposition, these species remain undissociated and therefore are only moved by diffusion and convection.
Boundary conditions have to be added as follows:
At the cathode surface $x = 0$,

$$-d_i w'_i(0) - m_i(0) \phi'(0) = -\gamma_i w_i(0), \quad \text{for } i = 1, \ldots, 5 \text{ and } \phi(0) = V,$$

where $\gamma_i = \beta_i \exp(-\alpha_i z_i FR \frac{V}{RT})$, $\gamma_i = 0$ for $i = 4, 5$.

At the surface $x = L$,

$$-d_i w'_i(L) - m_i w_i(L) \phi'(L) = \eta_i w_i(L) \quad \text{for } i = 3, 4, 5 \text{ and } \phi(L) = 0,$$

$$w_k(L) = w_k^* \quad \text{for } k = 1, 2.$$

According to Cochran, Von Karman and Levich [10, 12, 13], the fluid velocity component $v_x$ of $v$ can be expressed in term of power series expansion,

$$\xi = x \sqrt{\frac{\omega}{v}},$$

$$v_x = \sqrt{vH(\xi)} = \sqrt{v(\omega(-a\xi^2 + \frac{1}{3}\xi^3 + \frac{b}{6}\xi^4 + \ldots))},$$

Where, $a = 0.51023$, $\omega$ is the rotation speed of the RDE, and $v$ is the kinetic viscosity of the solution. The expression yields also to the boundary layer thickness [9]:

$$\delta = \left(\frac{3d}{a}\right)^{\frac{1}{3}} \frac{v}{v^2 \sqrt{v}}.$$

Where, $d$ is a reference diffusivity taken to correspond to the species in solution with the smallest diffusion coefficient.

### 2.2. Numerical scheme

| Symbols | Parameters |
|---------|------------|
| $d_i$  | Diffusion coefficient for each species |
| $m_i$  | Mobility for each species |
| $z_i$  | The velocity for each species |
| $\beta_i$ | The rate constants |
| $\alpha_i$ | The transfer coefficients |
| $V$  | The cathodic potential |
| $w^*_k$ | The concentrations of the species at $x = L$ |

Table 1: Key parameters

The system satisfied by the concentration:

For $i = 1, \ldots, 5$

$$\begin{cases}
-d_i \frac{\partial^2 w_i}{\partial x^2} + v_x \frac{\partial w_i}{\partial x} - m_i \frac{\partial}{\partial x}(w_i \frac{\partial \phi}{\partial x}) = S_i(w), & \text{in } \Omega \\
- \frac{\partial \phi}{\partial x} = \frac{4\pi F}{\varepsilon_0} \sum_{j=1}^{5} z_j w_j, & \text{in } \Omega \\
-d_i \frac{\partial w_i}{\partial x}(0) - m_i w_i(0) \frac{\partial \phi}{\partial x}(0) = -\beta_i w_i(0) \exp[-\alpha_i z_i FR \frac{V}{RT}], \\
-d_i \frac{\partial w_i}{\partial x}(L) - m_i w_i(L) \frac{\partial \phi}{\partial x}(L) = \eta_i w_i(L), & \text{for } i = 3, \ldots, 5 \\
w_k(L) = w_k^*, & \text{for } k = 1, 2 \\
\phi(0) = V, \phi(L) = 0,
\end{cases}$$

(9)
Where, $\Omega = [0, L]$ and $L = 3\delta$.

For $i = 1, 2$ we have

\begin{equation}
\begin{aligned}
\left\{
\begin{array}{ll}
-d_i \frac{\partial^2 w_i}{\partial x^2} + v_i \frac{\partial w_i}{\partial x} - m_i \frac{\partial}{\partial x}(w_i \frac{\partial \phi}{\partial x}) = 0, & \text{in } \Omega \\
-d_i \frac{\partial w_i}{\partial x}(0) - m_i w_i(0) \frac{\partial \phi}{\partial x}(0) = -\beta_i w_i(0) \exp[-\alpha_i z_i \frac{FV}{\pi T}], & \\
w_i(L) = w_i^*.
\end{array}
\right.
\end{aligned}
\end{equation}

(10)

For $x \in [0, L]$, we set

$$\psi(x) = \phi(x) - \frac{V}{L}(L - x),$$

and,

$$v_i(x) = w_i(x) - w_i^*.$$  

Then, system (10) is equivalent to the following system

\begin{equation}
\begin{aligned}
\left\{
\begin{array}{ll}
-d_i v''_i + (v_x + m_i \frac{V}{L}) v'_i - m_i (v_i \psi')' = m_i w_i^* \psi'' & \text{in } \Omega \\
-d_i v'_i(0) - m_i (v_i(0) + w_i^*)(\psi'(0) - \frac{V}{L}) = -\gamma_i (v_i(0) + w_i^*) , & \\
v_i(L) = 0.
\end{array}
\right.
\end{aligned}
\end{equation}

(12)

To give the numerical formulation of the problem (12), Let $V_1 = \{\phi \in H^1(\Omega) \mid \phi(L) = 0\}$ be the space of approximate solutions and the space of tests functions. Let $V_1^h$ be a finite element space of Lagrange $P1$ included in $V_1$. The Faedo-Galekrin formulation for the problem is given by finding $v_i^h \in V_1^h$ for $i = 1, 2$.

\begin{equation}
\begin{aligned}
\left\{
\begin{array}{ll}
\text{for every } \xi_h \in V_1^h & \\
\int_0^L d_i \frac{\partial v_{ih}^h}{\partial x} \frac{\partial \xi_h}{\partial x} + \int_0^L m_i v_i^h \frac{\partial \psi_h}{\partial \xi_h} \frac{\partial \xi_h}{\partial x} + \int_0^L (v_x + m_i \frac{V}{L}) \frac{\partial v_{ih}^h}{\partial \xi_h} & + [v_i^h(0)(\gamma_i + m_i \frac{V}{L})] \xi_h(0) \\
+ \gamma_i w_i^* \psi'(0) & = 0.
\end{array}
\right.
\end{aligned}
\end{equation}

Set,

$$a_i^1(v_i, \psi, \xi) = \int_0^L d_i \frac{\partial v_{ih}^h}{\partial x} \frac{\partial \xi_h}{\partial x} + \int_0^L m_i v_i^h \frac{\partial \psi_h}{\partial \xi_h} \frac{\partial \xi_h}{\partial x} + \int_0^L (v_x + m_i \frac{V}{L}) \frac{\partial v_{ih}^h}{\partial \xi_h} \xi_h$$

$$+ [v_i^h(0)(\gamma_i + m_i \frac{V}{L})] \xi_h(0),$$

$$b_i^1(\psi, \xi) = \int_0^L m_i w_i^* \psi'(0).$$

For $i = 3, 4, 5$ we have

\begin{equation}
\begin{aligned}
\left\{
\begin{array}{ll}
-d_i \frac{\partial^2 w_i}{\partial x^2} + v_i \frac{\partial w_i}{\partial x} - m_i \frac{\partial}{\partial x}(w_i \frac{\partial \phi}{\partial x}) = S_i(w), & \text{in } \Omega \\
-d_i \frac{\partial w_i}{\partial x}(0) - m_i w_i(0) \frac{\partial \phi}{\partial x}(0) = -\beta_i w_i(0) \exp[-\alpha_i z_i \frac{FV}{\pi T}], & \\
-d_i \frac{\partial w_i}{\partial x}(L) - m_i w_i(L) \frac{\partial \phi}{\partial x}(L) = \eta_i w_i(L).
\end{array}
\right.
\end{aligned}
\end{equation}

(13)

To show the numerical formulation of the problem (13), we set $V_2 = H^1(\Omega)$ to be the space of approximate solutions and the space of tests functions and $V_2^h$ be a finite element space of
Lagrange $P1$ included in $V_2$. To solve the problem (13) the Faedo-Galekrin formulation is used to find $w^h_i \in V^h_2$ for $i = 3, 4, 5$.

\[
\begin{aligned}
&\text{for every } \xi_h \in V^h_2 \\
&\int_0^L d_i \frac{\partial w^h_i}{\partial x} \frac{\partial \xi_h}{\partial x} + \int_0^L m_i w^h_i \frac{\partial \phi_h}{\partial x} \frac{\partial \xi_h}{\partial x} + \int_0^L v_x \frac{\partial w^h_i}{\partial x} \xi_h + \gamma_i w_i(0) \xi(0) \\
&\quad + \eta_i w_i(L) \xi(L) = f^h_0 S_i(w) \xi.
\end{aligned}
\]

Define,

\[
\begin{aligned}
a^2_i(w_i, \phi, \xi) &= \int_0^L d_i \frac{\partial w^h_i}{\partial x} \frac{\partial \xi_h}{\partial x} \\
&\quad + \int_0^L m_i w^h_i \frac{\partial \phi_h}{\partial x} \frac{\partial \xi_h}{\partial x} + \int_0^L v_x \frac{\partial w^h_i}{\partial x} \xi_h + \gamma_i w_i(0) \xi(0) + \eta_i w_i(L) \xi(L),
\end{aligned}
\]

\[
b^2_i(w, \xi) = \int_0^L S_i(w) \xi.
\]

And for the electric potential, we use Faedo-Galerkin formulation in $V^h_1$ such as

\[
\begin{aligned}
&\text{Find } \phi_h \text{ such that } \phi_h(0) = V, \text{ for every } \xi_h \in V^h_1 \\
&\int_0^L \frac{\partial \phi_h}{\partial x} \frac{\partial \xi_h}{\partial x} = \frac{4\pi F}{\varepsilon_0} \sum_{j=1}^5 \int_0^L z_j w^h_j \xi_h.
\end{aligned}
\]

2.3. Algorithm

We used the following algorithm to calculate $w_{i,h}$ and $\phi_h$ we denote $w_i = w_{i,h}$ and $\phi = \phi_h$

- Initializing : $\phi^0$ and calculate $w^1_i$, $i = 1, ..., 5$ such that,

  For $i = 1, 2$, $w^1_i = v_1^i + w_1^i$ where $v_1^i$ solution of

  \[
a^1_i(v_1^i, \psi^0, \xi) = b^1_i(\psi^0, \xi), \quad \text{with } \psi^0(x) = \phi^0(x) - \frac{V}{L}(L - x),
\]

  For $i = 3, 4, 5$, $w_1^i$ solution of

  \[
a^2_i(w^1_i, \psi^0, \xi) = 0,
\]

- Loop over n

  Calculate $\phi^n$ solution of

  \[
  \int_0^L \frac{\partial \phi^n}{\partial x} \frac{\partial \xi_h}{\partial x} = \frac{4\pi F}{\varepsilon_0} \sum_{j=1}^5 \int_0^L z_j w^j_n \xi_h,
  \]

  For $i = 1, 2$ calculate $v^n_i$ solution of

  \[
a^1_i(v^n_i, \psi^n, \xi) = b^1_i(\psi^n, \xi), \quad \text{with } \psi^n(x) = \phi^n(x) - \frac{V}{L}(L - x),
\]

  and finding $w^{n+1}_i = v^{n+1}_i + w^*_i$

  For $i = 3, 4, 5$ calculate $w_i$ solution of

  \[
a^2_i(w^{n+1}_i, \psi^n, \xi) = b^1_i(w^{n+1}_k, w^{n+1}_j, w^n_i, \xi), \quad \text{with } i \leq j \text{ and } k \leq i.
\]
2.4. Numerical result
The approached problem obtained by the finite element method was applied to a real experimental data. The parameter of hydrodynamic boundary layer thickness is given as $\delta = 1.1341 \times 10^{-5}$.

The diffusion coefficients $d_i$, the velocity $z_i$, the electric mobility $m_i$, and the parameters $\beta_i, \alpha_i, w_i^*$ are given as follows:

| species | $z_i$ | $d_i \times 10^9 (m^2/s)$ | $m_i \times 10^9$ | $\beta_i \times 10^9$ | $\alpha_i$ | $w_i^* (Mol/liter)$ |
|---------|-------|-----------------------------|-------------------|------------------------|------------|------------------|
| $Ni^{2+}$ | +2 | 0.68 | 52.133 | 350 | 0.175 | 0.5 |
| $Fe^{2+}$ | +2 | 0.76 | 58.27 | 270 | 0.269 | 0.01 |
| $H^+$ | +1 | 9.31 | 356.9 | 7.5 \times 10^3 | 0.130 | # |
| $SO_4^{2-}$ | -2 | 1.065 | 51.79 | 0 | 0 | # |
| $HSO_4^-$ | -1 | 1.33 | 82.95 | 0 | 0 | # |

Table 2: Electrodeposition - Experimental Data

The values of the solutions at the boundary $x = 3\delta$ are given by $w_i^*$.

The cathode potential on the (SHE) scale after ohmic correction is chosen as

$$-1.1 \leq V \leq -0.4$$

The current densities for the reactions considered in the model are

$$I_k(V) = -Fz_k\beta_kw_k(0)exp(-\alpha_kz_k\frac{FV}{RT}) \text{ for } k = 1, 2, 3$$

The total current is

$$I(V) = \sum_{k=1}^{3} I_k(V)$$

Moreover we define

- the composition of the iron deposit

$$w_0(V) = \frac{i_2(V)MF_e}{100(i_2(V)MF_e + i_1(V)MNi)}$$

- the effective current

$$\eta(V) = 100 \frac{i_1(V) + i_2(V)}{I(V)}$$

Finally, $T = 298K$, $PH = 3$ and $F = 96487$.

In this study, we compare the model prediction to experimental data reported in the literature. The first figure represents $I(V)$ the total current, the comparison of the composition of the iron deposit $w_0$ with experimental data is given in the second figure, and the effective current is shown in the last one.
Figure 1: $I(V)$ The total current
Figure 2: Comparison of the composition of the iron deposit $w_0$ with experimental data reproduced from the experiments done by Andricacos et al. (1989) in [7]
3. Conclusion
For a better understanding of the nickel-iron electrodeposition process, we presented a numerical analysis solving a system of reaction diffusion equations coupled with a potential equation modeling an electrodeposition process. The discretised problem obtained by the finite element method was applied to a real experimental data. The comparison between the numerical simulation and experimental results leads to the conclusion that our scheme can predict the characteristic features of the nickel-iron system.

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