Reaction Rates of Amorphous Iron Hydroxide with Nickel and Cobalt Ions in High Temperature Water

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Reactions of Ni and Co ions with amorphous iron(III) hydroxide, which was synthesized by electrolysis of iron electrode, have been studied in high temperature water using an in situ method of a modified magnetic balance and using a reaction model. Formation of NiFe$_2$O$_4$ and CoFe$_2$O$_4$ from the amorphous iron(III) hydroxide were explained by the reaction model incorporating two phenomena, i.e. dehydration of the iron(III) hydroxide and diffusions of Ni and Co ions into it. The dehydration rate followed Avrami's equation. Apparent activation energy of the dehydration was 4.39×10$^4$ J/mol for the amorphous iron(III) hydroxide used in the experiments. The diffusions of Ni and Co ions into the particle were evaluated from the diffusion equation for spherical coordinates. The diffusion coefficient of Ni ion into the amorphous iron(III) hydroxide was much higher than that of Co ion, while its apparent activation energy was about 5.5×10$^4$ J/mol, a value close to that of Co ion. However, the diffusion rate of Co ion into α-Fe$_2$O$_3$ was faster than that of Ni ion. Calculated values from the reaction model showed good agreement with experimental values.

KEYWORDS: reaction rate, reaction model, nickel ferrite, cobalt ferrite, hematite, amorphous iron hydroxide, iron crud, dehydration, diffusion, cobalt ions, nickel ions, Avrami's equation, activation energy, in situ measurement

I. INTRODUCTION

Corrosion products (Fe crud)$^{(13)(14)}$ contained in BWR cooling water are considered to play a significant role in the radioactive concentration of the reactor water$^{(5)}$~$^{(6)}$. Metallic ions which are the parent nuclides of the radioactive species are deposited together with Fe crud on the fuel cladding surface$^{(6)}$~$^{(8)}$. Release rates of radionuclides in these deposits depend on the deposits chemical form$^{(9)}$. Therefore, it is important to study the reactions of Fe crud with metallic ions in high temperature water in order to clarify the mechanism of radionuclide deposition-dissolution on the fuel rod surface. In recent crud-suppressed BWRs, both $^{59}$Co and $^{60}$Co radioactivities, which are respectively produced by the reactions $^{58}$Ni($n$, $p$)$^{58}$Co and $^{59}$Co($n$, $\gamma$)$^{60}$Co, have merited attention$^{(7)}$~$^{(8)}$.$^{(10)}$~$^{(12)}$.

In a previous paper$^{(13)}$, NiFe$_2$O$_4$ formation from iron(III) hydroxides and oxide, γ-FeOOH, α-FeOOH, amorphous iron(III) hydroxide and α-Fe$_2$O$_3$, which are the components of Fe crud in BWR condensate water was studied in high temperature water. An in situ method, using a magnetic balance, was developed to measure the mass of NiFe$_2$O$_4$. The iron(III) hydroxides react with Ni(OH)$_2$ to form NiFe$_2$O$_4$ and α-Fe$_2$O$_3$ through a dehydration reaction in water.$^{*}$

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above 553 K.

In this paper, reaction rates of amorphous iron(III) hydroxide with nickel and cobalt hydroxides are studied in high temperature water. The amorphous iron(III) hydroxide is the main component of Fe crud in BWR condensate water\(^{(12)(24)}\), and can be experimentally generated by electrolysis of iron electrode. Data of NiFe\(_2\)O\(_4\) and CoFe\(_2\)O\(_4\) formation measured by the in situ method are analyzed by a reaction model. Reactivity of Ni ions with amorphous iron(III) hydroxide is compared with that of Co ions according to the reaction model analysis.

II. REACTION MODEL

Iron(III) hydroxides are transformed into \(\alpha\)-Fe\(_2\)O\(_3\) (hematite) in high temperature water through a dehydration reaction\(^{(13)(15)}\). Since NiFe\(_2\)O\(_4\) and CoFe\(_2\)O\(_4\) are more stable than \(\alpha\)-Fe\(_2\)O\(_3\) thermodynamically\(^{(14)}\), NiFe\(_2\)O\(_4\) or CoFe\(_2\)O\(_4\) is formed by dehydration when Ni or Co ions exist at the dehydration sites in iron(III) hydroxide particles. To produce NiFe\(_2\)O\(_4\) or CoFe\(_2\)O\(_4\) requires both the dehydration reaction of iron(III) hydroxide and diffusion of Ni or Co ions into the particles. The dehydration reaction is assumed to proceed homogeneously everywhere inside the iron(III) hydroxide particles, while Ni and Co ions diffuse into the particle through their surfaces. The dehydration reaction is considered to occur in the area of Ni or Co ion diffusion with the same probability as in other areas inside the particles. Therefore, the NiFe\(_2\)O\(_4\) or CoFe\(_2\)O\(_4\) weight fraction \(f(t)\) in the particle at any reaction time can be expressed as follows:

\[
f(t) = \alpha(t) \cdot p(t),
\]

where \(\alpha(t)\) is the dehydration fraction of the iron(III) hydroxide at the reaction time \(t\), and \(p(t)\) the permeation fraction of Ni or Co ions. The value of \(p(t)\) is 1 when Ni or Co ion concentration in the whole particle is the same as NiFe\(_2\)O\(_4\) or CoFe\(_2\)O\(_4\).

The previous paper\(^{(11)}\) showed that the dehydration rates of iron(III) hydroxides follow Avrami's equation\(^{(17)(18)}\):

\[
\alpha(t) = 1 - \exp(-kt^n),
\]

where \(k\) is the dehydration rate constant and \(n\) the constant for each iron hydroxide species.

The value of \(p(t)\) is calculated from the profile of Ni or Co ion concentration as follows:

\[
p(t) = \frac{\int_0^r 4\pi r^2 C \, dr}{\int_0^r 4\pi r^2 C_0 \, dr},
\]

where \(C\) is the concentration of Ni or Co ions at the radius coordinate \(r\) in the particle, \(C_0\) the concentration of Ni or Co in NiFe\(_2\)O\(_4\) or CoFe\(_2\)O\(_4\), and \(r_s\) the particle radius. Assuming a constant spherical shape of the particle, the profile of the concentration \(C\) is obtained by solving the diffusion equation:

\[
\frac{\partial C}{\partial t} = \frac{1}{r^2} \frac{\partial}{\partial r} \left( D_e \frac{\partial C}{\partial r} \right),
\]

where \(D_e\) is the effective diffusion coefficient of Ni or Co ion. Constituents of the particle, other than NiFe\(_2\)O\(_4\) and CoFe\(_2\)O\(_4\), are iron(III) hydroxide and \(\alpha\)-Fe\(_2\)O\(_3\). The iron(III) hydroxide is considered to be distributed uniformly inside the particle because the dehydration proceeds homogeneously. Content of \(\alpha\)-Fe\(_2\)O\(_3\) in the outer region of the particle is smaller than in the inner region because diffusion of metallic ions from the particle surface results in a larger amount of NiFe\(_2\)O\(_4\) and CoFe\(_2\)O\(_4\) in the outer region. Therefore, \(D_e\) is defined as a function of time and \(r\):

\[
D_e(t, r) = D_{OH} f_{OH}(t) + D_{Fe} f_{Fe}(t, r), \quad (5)
\]

where subscripts OH and Fe denote the iron(III) hydroxide and \(\alpha\)-Fe\(_2\)O\(_3\), respectively, and \(f\) is the weight fraction and \(D\) the diffusion coefficient. It was seen that iron(III) hydroxides reacted with Ni(OH)\(_2\) to form NiFe\(_2\)O\(_4\) within a few hours, but \(\alpha\)-Fe\(_2\)O\(_3\) did not form NiFe\(_2\)O\(_4\) at 553 K even after a 100 h reaction\(^{(13)(14)}\). It is considered that the diffusion rate in iron(III) hydroxide is much higher than that in \(\alpha\)-Fe\(_2\)O\(_3\):

\[
D_{OH} \gg D_{Fe}. \quad (6)
\]

This leads to \(D_e\) as a function of only time as follows:

\[
D_e(t) = D_{OH} f_{OH}(t) = D_{OH} \exp(-kt^n). \quad (7)
\]
The concentration of Ni or Co ions at the particle surface is assumed to be held constant at \( C_0 \). The boundary conditions of Eq. (4) are expressed as

\[
\begin{align*}
C &= C_0; \quad t > 0, \quad r = r_0 \\
C &= 0; \quad t = 0, \quad 0 < r < r_0.
\end{align*}
\]

Under these conditions, the solution of Eq. (4) has been given\(^{(19)}\)\(^{(20)}\) and \( \rho(t) \) is expressed as follows:

\[
\rho(t) = 1 - \frac{6}{\pi^2} \sum_{n=1}^{\infty} \frac{1}{n^2} \exp\left(-\frac{Dt n^2 r_0^2}{r_0^2}\right)
\]

where \( Dt \) is defined as follows\(^{(21)}\):

\[
Dt = \int_0^t D_e dt = D_{OH} \int_0^t \exp(-kt^2) dt.
\]

In this way, the weight fraction of NiFe\(_2\)O\(_4\) or CoFe\(_2\)O\(_4\) generated at any reaction time is calculated from Eqs. (1), (2) and (9).

### III. EXPERIMENTAL

#### 1. Preparation of Sample

Amorphous iron(III) hydroxide was synthesized from Fe ion by electrolysis of an iron electrode. This Fe ion was oxidized while bubbling air through 303 K water for 1 h to precipitate the iron(III) hydroxide. The precipitated iron(III) hydroxide was filtered and dried in a room temperature vacuum desiccator for 20 h. The generated iron(III) hydroxide was confirmed to be amorphous by X-ray diffraction analysis. Its chemical composition was determined to be Fe\(_2\)O\(_3\)-2.10H\(_2\)O by elemental analysis and measurement of dehydration by thermogravimetry (Shinku Riko, DTG-7000). The specific surface area of the amorphous iron(III) hydroxide was 198.9 m\(^2\)/g as measured by the BET method (Simadzu, Accusorb-4000) and the average particle diameter was 1.78 \( \mu \)m as measured by centrifugal automatic particle analyzer (Horiba, Capa-500).

Hematite (\( \alpha \)-Fe\(_2\)O\(_3\)) was synthesized by the dehydration of the above amorphous iron(III) hydroxide in 553 K autoclave water for 8 h. The synthesized \( \alpha \)-Fe\(_2\)O\(_3\) was confirmed by X-ray diffraction analysis and its specific surface area was 12.2 m\(^2\)/g and the average particle diameter 1.08 \( \mu \)m.

#### 2. Dehydration Measurement

The autoclave equipment shown in Fig. 1 was used to measure the dehydration rates of the amorphous iron(III) hydroxide. Amorphous iron(III) hydroxide (about 300 mg) was dispersed in the autoclave water (700 ml) which was maintained at a constant temperature. The dissolved oxygen concentration [DO] of the water was 0.1~0.15 g/m\(^3\) and the pH was 6.9~7.0. At appointed time intervals, 100 ml samples were withdrawn and were filtered through a 0.1 \( \mu \)m pore size membrane. After drying in a vacuum desiccator, the amount of \( \alpha \)-Fe\(_2\)O\(_3\) generated by the dehydration reaction of the amorphous iron(III) hydroxide was determined by X-ray diffraction analysis.

![Fig. 1 Experimental apparatus for measurements of dehydration of iron(III) hydroxides](image)

#### 3. NiFe\(_2\)O\(_4\) and CoFe\(_2\)O\(_4\) Formation Measurements

The modified magnetic balance shown in Fig. 2 was used to measure the amounts of NiFe\(_2\)O\(_4\) and CoFe\(_2\)O\(_4\) produced in high temperature water. Among the reactants and products, only NiFe\(_2\)O\(_4\) and CoFe\(_2\)O\(_4\) are ferromagnetic. When NiFe\(_2\)O\(_4\) or CoFe\(_2\)O\(_4\) is formed in the reactor vessel, set in the magnetic field, the sample receives a force in accordance with the amount of NiFe\(_2\)O\(_4\) or CoFe\(_2\)O\(_4\). This force can be measured by Faraday's method\(^{(22)}\). In the horizontal magnetic field \( H_x \) and vertical magnetic field gradient \( \partial H_z / \partial z \), a sample receives the force \( F \) in accordance with the
following formula:

\[ F = m \chi g \mu_0 H_z \frac{\partial H_z}{\partial z} \]  

(11)

where \( m \) is the weight of NiFe\(_2\)O\(_4\) or CoFe\(_2\)O\(_4\), \( \chi g \) the mass magnetic susceptibility of NiFe\(_2\)O\(_4\) or CoFe\(_2\)O\(_4\), and \( \mu_0 \) the magnetic permeability in vacuum. Since \( H_z \), \( \partial H_z/\partial z \), \( \chi g \) and \( \mu_0 \) are known values, the weight \( m \) can be obtained from the measured force \( F \).

The amorphous iron(III) hydroxide or \( \alpha \)-Fe\(_2\)O\(_3\) was thoroughly mixed with Ni(OH)\(_2\) or Co(OH)\(_2\) of a molar quantity twice that needed to convert it into NiFe\(_2\)O\(_4\) or CoFe\(_2\)O\(_4\). Then, each powder sample was put in the reactor vessel together with the appropriate amount of deionized water. The value of [DO] was 0.1 ± 0.01 g/m\(^3\) and the pH was 6.9~7.0. Heating was done through PID control and the detected values were recorded by a small computer. Details of the equipment have been reported previously(13).

IV. RESULTS AND DISCUSSION

1. Dehydration Rates of Amorphous Iron(III) Hydroxide

Several kinds of iron(III) hydroxides exist in BWR cooling water and all of them undergo a dehydration reaction in high temperature water(14). The dehydration rates of iron(III) hydroxides follow Avrami's equation, expressed as Eq. (2). The values of \( k \) and \( n \) in Eq. (2) should be determined from experiments. The iron(III) hydroxides are transformed into \( \alpha \)-Fe\(_2\)O\(_3\) in high temperature water through the dehydration reaction when no other metallic ions coexist. Dehydration fraction \( \alpha(t) \) can be determined by the amount of \( \alpha \)-Fe\(_2\)O\(_3\) generated through the dehydration reaction. Figure 3 shows variations with reaction time in \( \alpha(t) \) obtained by X-ray diffraction analysis of \( \alpha \)-Fe\(_2\)O\(_3\) generated from the amorphous iron(III) hydroxide used in experiments. The dehydration fraction \( \alpha(t) \) increases with time and the dehydration rate is higher as temperature rises. Values of \( n \) and \( k \) are provided by plotting values of ln[-ln(1-\( \alpha \))] against ln\( t \). The value of \( k \) increases with the reaction temperature, but \( n \) takes the same value, 0.55, among the four reaction temperatures. The value of \( n \) is determined for each kind of iron(III) hydroxide particle and is independent of temperature, as observed previously(14). Figure 4 shows Arrhenius plots of the dehydration rate constant \( k \). Temperature dependency of \( k \) can be expressed as the following Arrhenius
Fig. 3 Variation of dehydration fraction of amorphous iron(III) hydroxide with reaction time

\[ k = 1.20 \times 10^5 \exp(-4.39 \times 10^4 / RT), \]  
\( (12) \)

where \( R \) is the gas constant and \( T \) the absolute temperature. The apparent activity energy of dehydration of this amorphous iron (III) hydroxide is \( 4.39 \times 10^4 \) J/mol. Since \( n \) and \( k \) values are determined, the value of \( \alpha(t) \) at any reaction time and temperature can be calculated from Eq. (2).

2. Diffusion Rates of Ni and Co Ions into Iron(III) Hydroxide

Figure 5 shows changes with reaction time in the fraction of NiFe\(_2\)O\(_4\) and CoFe\(_2\)O\(_4\) generated from the amorphous iron(III) hydroxide in 444 K water. It appears that the reactivity of Ni ions with the iron(III) hydroxide is higher than that of Co ions under these reaction conditions. This difference is attributable to the difference in the diffusion rate between Ni and Co ions into the iron(III) hydroxide particles.
The ion permeation fraction \( p(t) \) can be obtained by dividing experimental values of \( f(t) \) with \( a(t) \) calculated from Eqs. (2) and (12). Figure 6(a),(b) indicate changes with reaction time in \( p(t) \) of Ni and Co ions, respectively. The diffusion rate of Ni ions is about 10 times faster than that of Co ions by comparing the two plots.

![Variation of fraction permeated by Ni and Co ions with reaction time](image)

**Fig. 6(a),(b)** Variation of fraction permeated by Ni and Co ions with reaction time

The diffusion coefficients of Ni and Co ions into the particle, \( D_{OH,Ni} \) and \( D_{OH,Co} \), can be calculated from \( p(t) \) values shown in Fig. 6(a),(b) by Eq. (9). Change of the diffusion coefficients with the reaction temperature are shown as Arrhenius plots in Fig. 7.

![Arrhenius plots of diffusion coefficients of Ni and Co ions into amorphous iron(III) hydroxide](image)

**Fig. 7** Arrhenius plots of diffusion coefficients of Ni and Co ions into amorphous iron(III) hydroxide

The Ni ion diffusion coefficients \( D_{OH,Ni} \) are higher than \( D_{OH,Co} \) under these conditions. However, the slopes of the two lines are almost the same. Temperature dependancy of the diffusion coefficient for Ni ion, \( D_{OH,Ni} \), is determined as follows:

\[
D_{OH,Ni} = 1.14 \times 10^{-8} \exp(-5.40 \times 10^4/RT),
\]

(13)

and that for Co ion, \( D_{OH,Co} \), is

\[
D_{OH,Co} = 9.29 \times 10^{-11} \exp(-5.69 \times 10^4/RT).
\]

(14)

The apparent activation energy for diffusion of Ni ion is approximately equal to that of Co ion. The values of \( D_{OH,Ni} \), however, are much larger than \( D_{OH,Co} \) under the primary cooling water conditions.

3. Comparison of Experiment and Model Calculation

Since the values of \( n, k \) and \( D_{OH} \) are determined by experiments, the fractions of NiFe\(_2\)O\(_4\) and CoFe\(_2\)O\(_4\) generated can be calculated at any time and temperature. To begin with, the dehydration rate constant \( k \) and the ions diffusion coefficient \( D_{OH} \) are determined.
at each reaction temperature from Eqs. (12)~(14). Then, $a(t)$ for any time is calculated from Eq. (2) by using the obtained $k$. The values of $p(t)$ are calculated from Eqs. (9) and (10) by using the acquired $D_{\text{OH}}$. The fraction of the generated NiFe$_2$O$_4$ or CoFe$_2$O$_4$, $f(t)$ is evaluated by substituting $a(t)$ and $p(t)$ into Eq. (1). The calculated values of NiFe$_2$O$_4$ fraction $f_{\text{Ni}}(t)$ are compared in Fig. 8 with experimental values. The calculations agree closely with experimental data. This indicates that the presented model is appropriate for NiFe$_2$O$_4$ formation from amorphous iron(III) hydroxide. For the case of CoFe$_2$O$_4$ formation shown in Fig. 9, the calculated values are in approximate agreement with the experimental data, but the agreement is not as good as for NiFe$_2$O$_4$ formation. In particular, the experi-

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Fig. 8 Comparison between model calculation and experiment of NiFe$_2$O$_4$ formation from amorphous iron(III) hydroxide

Fig. 9 Comparison between model calculation and experiment of CoFe$_2$O$_4$ formation from amorphous iron(III) hydroxide
mental values are higher than the estimated values later in the reactions. This is considered to be caused by Co ion diffusion in the crystal lattice of $\alpha$-Fe$_2$O$_3$ formed by dehydration of the amorphous iron(III) hydroxide. In the presented model, the diffusions of Ni and Co ions are assumed to occur only in the iron(III) hydroxide. Therefore, the fractions of NiFe$_2$O$_4$ and CoFe$_2$O$_4$ saturate after the dehydration of the iron(III) hydroxide, according to the model calculation. Good agreement is observed for the NiFe$_2$O$_4$ formation because Ni ions do not diffuse into $\alpha$-Fe$_2$O$_3$. On the basis of these results, Co ions are predicted to diffuse into $\alpha$-Fe$_2$O$_3$ under these conditions.

The formation of NiFe$_2$O$_4$ and CoFe$_2$O$_4$ from $\alpha$-Fe$_2$O$_3$ are examined to confirm the diffusion of Co ions into $\alpha$-Fe$_2$O$_3$. If Ni or Co ions diffuse into the crystal lattice of $\alpha$-Fe$_2$O$_3$, the formation of NiFe$_2$O$_4$ or CoFe$_2$O$_4$ which is more stable than $\alpha$-Fe$_2$O$_3$ thermodynamically should be observed. Figure 10 shows the changes in the fractions of NiFe$_2$O$_4$ and CoFe$_2$O$_4$ formed from $\alpha$-Fe$_2$O$_3$ in 561 K water. Preparation of $\alpha$-Fe$_2$O$_3$ used in the experiments is done by the dehydration of the amorphous iron(III) hydroxide in 553 K water for 8 h. The fraction of CoFe$_2$O$_4$ increases slowly with time. On the other hand, no formation of NiFe$_2$O$_4$ is observed under these conditions. Then the diffusion of Ni ions into the $\alpha$-Fe$_2$O$_3$ crystal lattice is considered to be very slow at 561 K. Thus, the cause of disagreement between the model calculations and experiments in the latter reaction period shown in Fig. 6(a) is considered to be the diffusion of Co ion into $\alpha$-Fe$_2$O$_3$.

![Figure 10](image)

**V. CONCLUSION**

Formation of NiFe$_2$O$_4$ and CoFe$_2$O$_4$ from amorphous iron(III) hydroxide, which was synthesized by electrolysis of iron electrode, were observed in high temperature water by the *in situ* method of a modified magnetic balance. The reactions of the iron(III) hydroxide with Ni and Co ions in high temperature water could be explained by a reaction model incorporating two phenomena, the dehydration of iron(III) hydroxide and the diffusion of ions into the iron(III) hydroxide. The dehydration rate of the amorphous iron(III) hydroxide followed Avrami's equation. The diffusion coefficient of Ni ions into the amorphous iron(III) hydroxide, which was determined from a diffusion equation for spherical coordinates, was much higher.
than that of Co ions, but both apparent activation energies were close. The reactivity of Ni ions with the amorphous iron(III) hydroxide was higher than that of Co ions under BWR cooling water conditions. However, the diffusion rate of Co ions into $\alpha$-Fe$_2$O$_3$ was faster than that of Ni ions.

[NOMENCLATURE]

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(Subscripts)

- $e$: Effective
- $Fe$: $\alpha$-Fe$_2$O$_3$
- OH: Iron hydroxide

REFERENCES