Repassivation Behavior of Nickel-Base Alloys in High Temperature Acidic Solution[†]

Kazuo Yamanaka

Corrosion Research Laboratory, Iron & Steel Research Laboratories,
Sumitomo Metal Industries, Ltd., Amagasaki 660, Japan

The initial repassivation processes of anodic reaction on newly created surfaces of high nickel alloys containing 0–30 mass% chromium were investigated by a rapid straining electrode technique in a 10%Na₂SO₄+H₂SO₄ (pH 3.0 at room temperature) aqueous solution at 553 K at constant anodic potentials. The results obtained are as follows:

- (1) The decrease in current density with time after rapid straining consists of two stages. Namely, the current density in a short period (within around 20 ms) after rapid straining decays to the formula of $i \propto \exp(-t^n)$ (i : current density, t : time, n : constant), and then the density at least up to 1000 ms decays to the formula of $i \propto t^{-n}$.
- (2) The repassivation rate increased with increasing chromium content, and so the amounts of charge caused by the metal dissolution decreased with increasing chromium content.
- (3) It was considered that the reason for which nickel-base alloys containing high chromium content such as Alloy 690 (60%Ni–30%Cr–10%Fe) had high SCC resistance in a high temperature acidic solution containing sulfate ions is due to both the promotion of the repassivation and the suppression of the film dissolution by the formation of the chromium oxide film.

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I. Introduction

It has been reported that nickel-base alloys such as Alloy 600 have the intergranular attack (IGA)^{††} susceptibility in high temperature acidic solution as well as caustic solution⁽¹⁾, and Alloy 690 containing a higher chromium content than Alloy 600 has high corrosion resistance in accelerated primary water and off chemistry water of PWRs^{(2)–(4)}.

However, the reason why the SCC resistance and corrosion resistance of Alloy 690 has not yet been made clear; especially the relation between IGA resistance and surface oxide film formed in a high temperature acidic solution, is unclear.

Thus, in the present paper, using a rapid straining electrode technique⁽⁵⁾ which can quantitatively analyze a transient anodic dissolution process on a newly created surface, the repassivation behavior of nickel-base alloys containing 0–30 mass%Cr was investigated to make clear the effect of chromium content on the initial repassivation process of anodic reaction on a newly created surface at the crack tip.

II. Experimental Method

1. Specimen

The materials used in this study were vacuum-melted 75%Ni–bal. Fe alloys having 0, 5, 15 and 23%Cr, and 60%Ni–30%Cr–10%Fe alloy. Table 1 shows the chemical composition of the alloys.

The alloys were forged, hot-rolled, drawn to a thin wire of 0.50 mm in diameter and annealed in argon atmosphere at 1373 K for 1.8 ks (0.5 h) and then water quenched. Then the wires were polished by emery paper up to No. 1000 and degreased with acetone.

2. Experimental equipment

A schematic drawing of rapid straining electrode equipment used in this investigation is shown in Fig. 1. The autoclave with the volume of about 0.18 m³ is made of Alloy 690. The wire specimen is attached to the chucks made of Hastelloy C, and zirconia coated zirconium plates are inserted between the chuck and the specimen in order to electrically isolate the specimen from the autoclave. One side (upper part) of the specimen holding chuck is connected to a pull rod made of Hastelloy C, and the pull rod passes through the autoclave head plate with water cooled pressure balanced fitting seal and is connected to a piston in a cylinder. When high pressure argon gas is led into the cylinder, the piston is pulled up rapidly and the pull rod is moved up, and then the wire specimen is elongated. A constant amount of strain is given by a stopper set in the cylinder, the amount of

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^{††} Intergranular attack (IGA) is composed of intergranular corrosion (IGC) and intergranular stress corrosion cracking (IGSCC) following IGC.

Table 1 Chemical composition of alloys.

Alloy	Chemical compositions (mass%)									
	C	Si	Mn	P	S	Ni	Cr	Ti	Al	Fe
1	0.014	0.13	0.31	0.002	0.001	74.38	0.01	0.20	0.10	24.85
2	0.018	0.11	0.29	0.001	0.001	74.55	4.86	0.21	0.13	19.83
3	0.018	0.11	0.29	0.001	0.001	75.15	15.05	0.21	0.14	9.03
4	0.018	0.12	0.29	0.001	0.001	74.96	22.75	0.21	0.14	1.51
5	0.020	0.18	0.33	0.002	0.002	60.87	30.05	0.26	0.15	8.13

Fig. 1 Schematic drawing of strain electrode equipment in high temperature aqueous solutions.

strain is measured by a transducer mounted on the top of a piston. 10 mass% Na_2SO_4 aqueous solution adjusted to pH 3.0 by the addition of H_2SO_4 at room temperature was used as a testing solution, and test temperature was 553 K. A reference electrode for electrochemical measurement is used an internal Ag/AgCl reference electrode (a liquid inside an electrode: $0.1 \text{ kmol} \cdot \text{m}^{-3}$ KCl). The transient anodic current and the output of the strain transducer are recorded in 2 channel digital memory recorder (Kawasaki Electronica, Type TMR-10).

3. Straining electrode testing method

A wire specimen was mounted to a specimen holding chuck. The autoclave was filled with a testing solution, and the solution was deaerated by high purity argon gas. Then the autoclave was heated to a desired temperature. After reaching to the desired temperature, straining electrode tests were carried out several times at applied constant potential. When a rapid strain is applied to the specimen, the surface film is ruptured and transient cur-

rent is observed due to the appearance of newly created surface. Then the current decays immediately after stopping elongation. The current density on a newly created surface, i , is defined by the following equation:

$$i = \frac{\Delta i(t)}{\Delta S} \quad (1)$$

Here, $\Delta i(t)$ is calculated by reducing residual current that is measured just before straining from transient current measured. ΔS is a small amount of increase of specimen surface. Assuming that total area is constant, ΔS is calculated by the following equation⁽⁴⁾.

$$\frac{\Delta S}{S_0} = \frac{1}{2} \frac{\Delta l}{l_0} \quad (2)$$

where S_0 is initial surface area, Δl is a small amount of elongation and l_0 is initial length. In the present work, as $l_0 = 25 \text{ mm}$, $S_0 = 3.9 \times 10^{-5} \text{ m}^2$ and $\Delta l = 0.40 \text{ mm}$ were adopted, ΔS calculated by an eq. (2) is $3.1 \times 10^{-7} \text{ m}^2$. Since the time from the start to the end of an elongation was about 7 ms, the strain rate being about 2.3 s^{-1} .

4. Analysis of corrosion film

The corrosion films of the alloys were mainly analyzed by ion micro mass analyzer (IMMA, ARL made), and also by ESCA (VG made, ESCA3 Mark) in some alloys. The samples for film analysis were made in the 10 mass% $\text{Na}_2\text{SO}_4 + \text{H}_2\text{SO}_4$ (pH 3.0 at room temperature) aqueous solution at 598 K for 7.2 ks (2 h) at a constant potential in the platinum container put into the autoclave in order to avoid the contamination by the dissolution from the autoclave container.

III. Experimental Results

1. Anodic polarization curves

Anodic polarization curves for 75%Ni-0%Cr-25%Fe, 75%Ni-15%Cr-10%Fe (Alloy 600) and 60%Ni-30%Cr-10%Fe (Alloy 690) alloys in deaerated 10% $\text{Na}_2\text{SO}_4 + \text{H}_2\text{SO}_4$ aqueous solution (pH=3.0) at 553 K is shown in Fig. 2. The curve of Alloy 600 in Fig. 2 exhibits the presence of the active peak, the first passive state and the second passive state in near -0.50 V (vs Ag/AgCl), in the range from about -0.40 V to $+0.1 \text{ V}$ and from about $+0.2 \text{ V}$ to $+0.6 \text{ V}$, respectively. As chromium content was increased from 0% to 30%, current density of the active peak and passive

Fig. 2 Anodic polarization curves and intergranular attack zone of high nickel alloys in 10%Na₂SO₄ + H₂SO₄ (pH = 3.0) aqueous solution at 553 K.

region in the potential range from about -0.6 V to 0 V lowered. The presence of the current density peak in the potential range from $+0.1$ to 0.6 V in both 75%Ni-15%Cr-10%Fe and 60%Ni-30%Cr-10%Fe alloys is considered to be due to chromium dissolution. Straining electrode tests were carried out by applying each fixed potential $E_{\text{corr.}} + 100$ mV [where $E_{\text{corr.}}$ is around -0.65 V (vs Ag/AgCl)] corresponding to the active state, $E_{\text{corr.}} + 300$ mV and $E_{\text{corr.}} + 500$ mV in the first passive state, where IGA of Alloy 600 occurs⁽⁶⁾.

2. Straining electrode behavior

The effect of chromium content on changes in transient current densities with time of nickel base alloys was measured in deaerated 10%Na₂SO₄ + H₂SO₄ (pH = 3.0) aqueous solution at 553 K at each fixed potentials $E_{\text{corr.}} + 100$ mV, $E_{\text{corr.}} + 300$ mV and $E_{\text{corr.}} + 500$ mV.

Figure 3 shows the transient current decay curves of nickel-base alloys at the fixed potential of $E_{\text{corr.}} + 100$ mV. After the elongation was stopped, the distinct regions in the current decay with time were observed. That is, the current decay consists of two stages. The current density in a short period (within around 20 ms) immediately after rapid straining was stopped decays to the formula of $i \exp(-t^n)$ (i : current density, t : time, n : constant) (named stage I), and then the density at least up to 1000 ms decays to the formula of $i \propto t^n$ (stage II).

Figure 4 shows the effect of chromium content on i_{max} , $i_{10 \text{ ms}}$ and $i_{100 \text{ ms}}$, in which the maximum current density i_{max} immediately after rapid straining, the current densities $i_{10 \text{ ms}}$ at 10 ms and $i_{100 \text{ ms}}$ at 100 ms after the elongation

Fig. 3 Effect of chromium content on current transient curves in 10%Na₂SO₄ + H₂SO₄ (pH = 3.0) aqueous solution at 553 K. (Applied potential: $E_{\text{corr.}} + 100$ mV).

Fig. 4 Effect of chromium content on i_{max} , $i_{10 \text{ ms}}$ and $i_{100 \text{ ms}}$ of nickel base alloys in 10%Na₂SO₄ + H₂SO₄ (pH = 3.0) aqueous solution at 553 K (Applied potential: $E_{\text{corr.}} + 100$ mV).

from Fig. 3 are plotted against the chromium content. From this figure, it can be seen that i_{max} showed slightly larger with increasing chromium content. On the other hand, both $i_{10 \text{ ms}}$ and $i_{100 \text{ ms}}$ became smaller with increasing chromium content.

Typical examples of the formula of change in anodic current density with time for stage I ($t < 20$ ms) and stage II ($t \geq 20$ ms) obtained in this experiment are shown in Table 2.

Table 2 Formula of change in anodic current density with time (10%Na₂SO₄ + H₂SO₄ (pH=3.0), $E_{\text{corr.}}$ + 100 mV, 553 K).

Alloy	Current density (A/m ²)	
	Stage I ($t < 20$ ms)	Stage II ($t \geq 20$ ms)
75%Ni- 0%Cr-25%Fe	$0.71 \times 10^2 \exp(-27.88t^{0.683})$	$3.98 \times 10^0 t^{-0.401}$
75%Ni- 5%Cr-20%Fe	$0.80 \times 10^2 \exp(-30.21t^{0.645})$	$1.77 \times 10^0 t^{-0.450}$
75%Ni-15%Cr-10%Fe	$0.85 \times 10^2 \exp(-15.87t^{0.514})$	$1.23 \times 10^0 t^{-0.465}$
75%Ni-23%Cr- 2%Fe	$1.03 \times 10^2 \exp(-68.99t^{0.707})$	$3.36 \times 10^{-1} t^{-0.388}$
60%Ni-30%Cr-10%Fe	$1.13 \times 10^2 \exp(-17.92t^{0.401})$	$2.78 \times 10^{-1} t^{-0.408}$

 Fig. 5 Effect of chromium content on current transient curves in 10%Na₂SO₄ + H₂SO₄ (pH=3.0) aqueous solution at 553 K. Applied potential: (a) $E_{\text{corr.}}$ + 300 mV, (b) $E_{\text{corr.}}$ + 500 mV.

On the other hand, the effects of chromium content on current transient curves caused by a repassivation phenomenon after the elongation in deaerated 10%Na₂SO₄ + H₂SO₄ (pH=3.0) aqueous solution at 553 K at the potentials of $E_{\text{corr.}}$ + 300 mV and $E_{\text{corr.}}$ + 500 mV are shown in Figs. 5(a) and (b), respectively. In both figures, i_{max} of all of the five alloys varying in chromium content showed larger value than that of the potential of $E_{\text{corr.}}$ + 100 mV. And also the current attenuation showed faster than that of the potential of $E_{\text{corr.}}$ + 100 mV in the four alloys containing above 5% chromium.

The power n in stage II, which the current decayed with the power law ($i \propto t^{-n}$), varied with potential and chromium content as shown in Fig. 6. The value of n means the repassivation ability because the larger n shows the higher repassivation rate.

It can be seen from Fig. 6 that the value of n was increased from about 0.5 to 0.7~0.9 with increasing the chromium content in the concentration range from 0 to 30%Cr at the fixed potentials of both $E_{\text{corr.}}$ + 300 mV and

 Fig. 6 Effect of potential and chromium content on the value of n of the current decay curves in stage II ($i \propto t^{-n}$) of nickel-base alloys in 10%Na₂SO₄ + H₂SO₄ (pH=3.0) aqueous solution at 553 K.

Fig. 7 Effect of chromium content on amounts of charge in stage (I+II) of nickel base alloys in 10%Na₂SO₄+H₂SO₄ (pH=3.0) aqueous solution at 553 K.

$E_{\text{corr.}} + 500 \text{ mV}$.

Amount of charge passed in 1 s following a film rupture repassivation process can be obtained by integrating the current decay curves shown in Figs. 3 and 4. Figure 7 shows the effect of chromium content on amounts of charge of nickel base alloys in deaerated 10 mass%Na₂SO₄+H₂SO₄ aqueous solution (pH=3.0) at 553 K. This figure showed that the amounts of charge decreased with increasing chromium content and that of Alloy 690 containing 30 mass%Cr was lowered to 1/4–1/6 as compared with Alloy 600 containing 15 mass%Cr. This behavior seems to correspond to the effect of chromium content on the acidic IGA resistance of nickel-base alloys⁽⁶⁾.

3. Corrosion film

Figure 8 showed the depth profile of Ni, Cr, Fe, Si, Mn and O elements by IMMA analysis of the corrosion film formed on both 75%Ni–15%Cr–10%Fe (Alloy 600) and 60%Ni–30%Cr–10%Fe alloy (Alloy 690) in deaerated 10%Na₂SO₄+H₂SO₄ aqueous solution (pH=3.0) at 553 K at the potential of $E_{\text{corr.}} + 300 \text{ mV}$.

The corrosion film of 75%Ni–15%Cr–10%Fe alloy (Alloy 600) had rather thick film of chromium oxide as shown in Fig. 8(a). On the other hand, the corrosion film of the 60%Ni–30%Cr–10%Fe alloy (Alloy 690) had a considerably thin film of chromium oxide as compared with Alloy 600 as shown in Fig. 8(b).

According to ESCA analysis, it is thought that Cr was composed of mainly Cr₂O₃ in both alloys. The fact that chromium oxide can exist stably in a high temperature acidic solution can be clearly seen from the pH-potential diagram of the Cr–H₂O system⁽⁷⁾.

Fig. 8 IMMA analysis of the corrosion film of nickel-base alloys at the potential $E_{\text{corr.}} + 300 \text{ mV}$ in 10%Na₂SO₄+H₂SO₄ (pH=3.0) aqueous solution at 553 K for 7.2 ks (2 h).

IV. Discussion

1. Repassivation behavior in a high temperature acidic solution

Figure 9 shows a schematic representation of the anodic current decay behavior obtained by the measurement of 60~75%Ni–0~30%Cr–25~2%Fe alloys in a high temperature acidic aqueous solution after rapid straining.

That is, the current decay consists of two stages: the current density in a short period (within around 20 ms) immediately after rapid straining decays to the formula of $i \propto \exp(-t^n)$ (i : current density, t : time, n : constant) (named stage I), and then the density at least up to 1000 ms decays to the formula of $i \propto t^{-n}$ (stage II). The fact that the current density follows the formula of $i \propto t^{-n}$ was already obtained by Shibata and Fujimoto⁽⁸⁾ in high temperature Na₂SO₄ and H₃BO₃+Na₂B₄O₇ aqueous solution, and also obtained by Yamanaka and Murayama⁽⁹⁾ using nickel base alloys in high temperature NaOH aqueous solution. Surmising from the current decay curves obtained, it is considered that the repassivation behavior of nickel base alloys in both high temperature caustic and acidic aqueous solution is similar to some extent, although the compositions and structure of oxide film formed on the alloys in acidic aqueous solution are different from that in caustic aqueous solution. Namely, in a high temperature acidic aqueous solution such as this

Fig. 9 Schematic figure of change in anodic current density with time.

investigation, as can be clearly seen in the pH-potential diagram for Cr-H₂O system⁽⁷⁾, chromium oxide (Cr₂O₃) exists in a stable state and so the film formation of chromium oxide may be mainly caused by the following reaction:

This is supported by the film analysis by IMMA as shown in Fig. 8. On the other hand, the corrosion film formed in a high temperature caustic aqueous solution, as have already been reported⁽⁹⁾, had a double layer structure in which nickel hydroxide or oxide in the outer layer and chromium oxide in the inner layer existed. Since chromium oxide cannot exist stably in high temperature caustic aqueous solutions, the nickel hydroxide or oxide layer may be mainly formed in the initial film formation process by the following reaction:

Thereafter, supposing that this outer nickel hydroxide or oxide layer may act as a protective film such as a cation selective film, the chromium oxide layer can be formed in the inner layer by the following reaction (the

same as eq. (3)):

By considering this, it is thought that the inner chromium oxide layer can be formed by decreasing pH beneath the cation selective outer nickel hydroxide or oxide film.

It is concluded that chromium is effective to both the increase in the repassivation rate and the suppression of the film dissolution in a high temperature acidic aqueous solution such as this investigation.

It can be clearly seen that the repassivation behavior has some difference between low chromium (below around 15%Cr) and high chromium (around 30%Cr) nickel-base alloys as shown in Figs. 3, 5 and 8.

In just after the film rupture condition, high chromium alloys, which containing much active element (chromium), are occurred high metal dissolution as compared with low chromium alloys.

However, at only 10~100 ms after film rupture, high chromium alloys may be repassivated by the dense chromium oxide film which has high suppression efficiency.

Figure 10 shows a schematic figure of the repassivation behavior of both low chromium and high chromium nickel-base alloys in high temperature acidic aqueous solution.

Therefore, it was considered that the reason, why nickel base alloys containing high chromium content such as Alloy 690 (60%Ni-30%Cr-10%Fe) had high SCC resistance in high temperature acidic solution containing sulfate ion, is due to both the promotion of the repassivation and the suppression of the film dissolution by the formation of the dense chromium oxide film.

2. Estimation of the average SCC propagation rate

It has been already reported that IGSCC of Ni-base Alloy 600 occurs in such acidic environments as high concentrated SO₄²⁻ ions at the crevice of tube/tube sheet or

Fig. 10 Schematic figure of the repassivation behavior of Ni-base alloys in high temperature acidic solution.

tube support plate⁽¹⁰⁾. The contamination source of SO_4^{2-} ions is caused by leak of the ion exchange resin and thermal decline of resin itself used for the demineralizer, and condenser leak of the secondary water⁽¹⁾. Newman⁽¹⁾ has measured by using C-ring test method in such environment that the average SCC propagation rate of Alloy 600 is an order of around 1×10^{-7} m/s under the condition of applied stress equivalent to the yield strength at 563 K.

Here, the average SCC propagation rate of Alloy 600 was estimated by using amounts of charge Q measured by the rapid straining electrode technique.

According to Faraday's law, the relation between corrosion depth da and charge Q is given by

$$da = \frac{M}{N\rho F} Q \quad (6)$$

where Q : charge, M : atomic weight, ρ : density, F : Faraday's constant (96500 coulomb), N : valence change. In the case of Alloy 600, $M=57.4$ g/mol, $\rho=8.45 \times 10^3$ kg/m³ and $N=3$, assuming that the Cr_2O_3 oxide film is formed. Substituting these values for eq. (6), eq. (7) is obtained:

$$da = 2.35 \times 10^{-7} Q \text{ (m)} \quad (7)$$

Charge Q in eq. (7) is expressed by the accumulation of charge $Q_{\text{stage I}}$ in stage I and charge $Q_{\text{stage II}}$ in stage II.

Repeating time of film rupture/repassivation is t_f , SCC propagation rate da/dt is given by

$$\frac{da}{dt} = 2.35 \times 10^{-7} \frac{Q}{t_f} \text{ (m/s)} \quad (8)$$

here, t_f is given by

$$t_f = \frac{\varepsilon_f}{\dot{\varepsilon}} \quad (9)$$

ε_f is fracture strain of the oxide film at the crack tip and $\dot{\varepsilon}$ is strain rate at the crack tip and is given by

$$\dot{\varepsilon} = \alpha \sigma^{-n} \quad (10)$$

where σ : stress at the crack tip, α , n : constant (n is normally 4~5). Assuming that ε_f of Alloy 600 is the same as ε_f value of FeO (3×10^{-3}), and using that $\dot{\varepsilon}$ is the creep rate formula of 800°F (699 K) from the literature data⁽¹¹⁾ ($\dot{\varepsilon} = 10^{-10.78} \sigma^5$), eq. (11) is obtained by substituting these values for eq. (9):

$$t_f = 3 \times 10^{-7.78} \sigma^{-5} \text{ (s)} \quad (11)$$

Assuming that σ is yield stress of Alloy 600 (around 30 kgf/mm²), and using Q value of Fig. 7, the average SCC propagation rate is obtained from eq. (8):

$$\frac{da}{dt} \approx 2.2 \times 10^{-7} \text{ (m/s)} \quad (12)$$

This value is roughly good agreement with the mea-

sured value by the C-ring test method mentioned above.

Therefore, it can be concluded that the rapid straining electrode technique in this investigation surely reappears the SCC initial stage of Alloy 600 which is composed of the film rupture/repassivation process in a 10%Na₂SO₄+H₂SO₄ (pH=3.0) aqueous solution at high temperature.

V. Conclusions

Using a rapid straining electrode technique, the initial repassivation processes on newly created surface of 60~75%Ni-0~30%Cr-25~2%Fe alloys in a high temperature 10 mass%Na₂SO₄+H₂SO₄ (pH 3.0 at room temperature) aqueous solution at 553 K were investigated at constant anodic potentials, and the following results were obtained.

(1) The decrease in current density with time after rapid straining consists of two stages. Namely, the current density in a short period (within around 20 ms) after rapid straining decays to the formula of $i \propto \exp(-t^n)$ (i : current density, t : time, n : constant), and then the density at least up to 1000 ms decays to the formula of $i \propto t^{-n}$.

(2) The repassivation rate increased with increasing chromium content, and so the amounts of charge caused by the metal dissolution decreased with increasing chromium content.

(3) It was considered that the reason for which nickel-base alloys containing high chromium content such as Alloy 690 (60%Ni-30%Cr-10%Fe) had high SCC resistance in a high temperature acidic solution containing sulfate ions is due to both the promotion of the repassivation and the suppression of the film dissolution by the formation of the chromium oxide film.

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