## ECP AND LOCALIZED CORROSION OF STEAM GENERATOR TUBE MATERIALS UNDER SIMULATED CREVICE CONDITIONS

#### Y.C. LU

## ABSTRACT

The electrochemical corrosion potential (ECP) of tube materials under faulted SG water and MULTEQ-calculated crevice chemistry conditions, which include crevice conditions resulting from seawater ingress, and simulated Bruce nuclear generator station A (BNGS-A) crevice conditions, was measured in order to provide input into attempts to determine the stress corrosion cracking (SCC), pitting and under-deposit corrosion susceptibility of steam generator (SG) tube materials. The effect of magnetite sludge, and of minor impurities in the sludge, on the ECP of the SG tube materials was studied by comparing the ECP of sludge-coated and uncoated samples. Experimental results revealed that various sludge deposits, including magnetite-containing nickel ferrite, lead oxide and zinc silicate can increase the ECP by more than one hundred mV. Significant positive shifts in the ECP of the SG tube materials were also observed in simulated BNGS-A crevice chemistries. In these simulated chemistry environments, 100 ppm of PbO and 30 g/l of SiO<sub>2</sub> were present.

In most cases, the measured ECP values for tube materials under crevice chemistry conditions were lower than the passive film breakdown potential of SG tube materials. This is because localized corrosion had already taken place in the aggressive crevice electrolyte. The kinetics of localized corrosion depend on many factors. This makes it difficult to reproduce the observed ECP values of SG tube materials. For instance, the measured value in simulated seawater crevice solution varied between -590 and -250 mV (SHE).

The SG tube in the crevice is coupled with the tube free-span. The ECP of tube alloy in the crevice will also be influenced by the ECP at the tube free-span area. The existence of an IR drop will shift the ECP inside the crevice to a more negative value than at the tube free span. Tests performed in a simulated heated crevice showed this potential shift to be in the range 100 to 160 mV. The existence of steam bubbles will also add extra resistance to the ionic path in the corrosion system and influence the crevice ECP.

The rate of crevice corrosion propagation is controlled by both the anodic and cathodic kinetics of corrosion-related reactions. The existence of oxidizing sludge inside the crevice enhances cathodic reactions which would normally take place at the free surface outside the crevice. Therefore the presence of oxidizing sludge inside the crevice will provide a short circuit to the corrosion system and increase the localized corrosion rate significantly

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#### INTRODUCTION

The electrochemical corrosion potential (ECP) is known by several terms: such as open circuit potential, and rest potential. For a corroding system, it is often called corrosion potential. Mixed potential is probably the most appropriate description of the potential. It is based on the physical condition that charge must be conserved in an electrochemical system. The following equation expresses the condition which determines the mixed potential, ECP, of a freely-corroding system:

$$\sum_{j=1}^{n} i_{R/O,j}(E) + i_{Corr}(E) = 0$$

where i  $RO_{i}$  is the partial current density due to the  $j_{th}$  redox couple in the system and  $i_{Corr}$  is the corrosion current density of the substrate. The partial current densities are dependent on the potential drop across the metal solution interface, the equilibrium potential for the reaction, the anodic and cathodic Tafel constants, and the mass-transfer limited current. Therefore, the ECP of an alloy is determined not only by the thermodynamics and electrode kinetics but also by the mass transport conditions at the surface.

ECP modelling has been widely used to predict the corrosion behaviour of various materials in high- temperature water. <sup>1,2,3</sup> It was viewed as a direct measure of the driving force for stress corrosion.<sup>4</sup> Steam generator (SG) crevices are the location where the tube materials are highly susceptible to a variety of localized attack. Sato and co-workers<sup>5</sup> determined ECP of 304 stainless steel and Inconel 600 in a simulated crevice in high-temperature high purity water and the pH of the environment developed in the crevice. They found that the crevice ECP was 300 mV to 400 mV lower than that at the free-surface outside the crevice (with no deposits present) and the crevice pH in high-temperature high purity water was substantially lower than that of bulk water. SG crevice sites in secondary side also have accumulations of sludge with a variety of impurities. It is necessary to study the electrochemical behaviour and corrosion susceptibility of the SG tube material under the faulted simulated SG crevice conditions. This work is an attempt to study the ECP and localized corrosion of SG tube materials in simulated CANDU SG crevice chemistries in the presence of sludge deposits of different chemical composition.

#### EXPERIMENTAL CONDITIONS

Segments of Incoloy 800, Inconel 600, Inconel 690 were mounted onto a specially designed sample holder installed in an autoclave. The ECP values were measured continuously at 260° C

using an internal Ag/AgCl /4M KCl or Ag/AgCl/0.1M KCl high-temperature reference electrode in simulated crevice chemistries following seawater ingress<sup>6</sup> or simulated BNGS-A crevice chemistries.<sup>7</sup> All potentials are converted to the standard hydrogen electrode scale (SHE).<sup>8</sup> The solutions used are defined in Table 1 and Table 2. The pH values quoted in this paper all refer to pH values measured at 25°C.

TABLE 1.	Simulated	Crevice	Chemistries	Following	a Seawater Ingress
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Chemicals	NaCl	KCl	Na₂SO₄	pН
Concentration	5M	5M	1 <b>M</b>	6.1

Crevice Environment Simulated	Suggested experimental solution	MULTEQ Predictions: Solution pH,
	"recipe"	Chemistry and Precipitates, 25C
1. "Base" BNGS-A crevice	0.15M Na2SO4	pH-8.97
environment	0.3M NaCl	Solution Chemistry:
	0.05M KCl	Na: mainly as Na+, Na2SO4
1	0.15M CaCl2	Ca: mainly as Ca2+
	("base" solution #1)	K: as K+
	plus:	Cl: as Cl-
	$\sim 0.5$ mole SiO2 (* not expected to be	H3SiO4:virtually all as SiO2 precipitate
	soluble; solid)	SO4:mainly as Na2SO4, SO42-
	~ 100 ppm Pb 2+ (added as PbO)	Pb: mainly as PbSO4 precipitate, small
		amt. PbCl+ in solution
		Precipitates: CaSiO3, SiO2, PbSO4
2. Deviation from "base"-alkaline	Add 0.4 M NaOH to "base" solution #1	pH-12.58
pH	plus:	Solution Chemistry
	~0.5 mole SiO2 (see * above)	Na: mainly as Na+, Na2SO4
	~ 100 ppm Pb 2+ (added as PbO)	Ca:100% s CaSiO3 precipitate
1		K: as K+
		Cl: as Cl-
		H3SiO4:mainly as SiO2, CaSiO3
	р 	precipitates; some H3SiO4- in solution
		SO4: mainly as Na2SO4, SO42-
		Pb- mainly as Pb(OH)3 in solution
		Precipitates: CaSiO3,SiO2
3. Deviation from "base"-acidic	Add 0.05M NaHSO4 to "base"	pH-2.44
pH	solution #1	Solution Chemistry:
		Na: mainly as Na+, Na2SO4
	plus:	Ca: as Ca2+
	~0.5 moles SiO2 (see * above)	K: as K+
	~100 ppm Pb2+ (added as PbO)	Cl: as Cl-
		H3SiO4-virtually all as SiO2 precipitate
		SO4-mainly as Na2SO4, SO42-, HSO4-,
		NaHSO4
		Pb- mainly as PbSO4 precipitate, small
		amount PbCl+ in solution
		Precipitates: SiO2, PbSO4

TABLE 2 Simulated BNGS-A SG Crevice Chemistry Conditions

Sludge Coating	1	2	3	4	5	6
Composition	Fe <sub>3</sub> O <sub>4</sub> 100%	Fe <sub>3</sub> O <sub>4</sub> 60% + Cu 40%	Fe <sub>3</sub> O <sub>4</sub> 90% + Pb 10%	Fe₃O₄ 90% + PbO 10%	Fe <sub>3</sub> O <sub>4</sub> 80% + Zn <sub>2</sub> SiO <sub>4</sub> 20%	Fe <sub>3</sub> O <sub>4</sub> 80% + NiFe <sub>2</sub> O <sub>4</sub> 20%

Parallel tests were performed with tube and platinum wire samples coated with simulated sludge deposits of various compositions. Details of the preparation of the artificial sludge coating are described elsewhere.<sup>9</sup>

#### EXPERIMENTAL RESULTS AND DISCUSSION

ECP Measurements in Simulated SG Crevice Chemistries Following a Seawater Excursion

The ECP of the SG tube materials was measured under simulated crevice chemistry conditions at 260°C. The ECP data of Incoloy 800, Inconel 600, and Inconel 690, coated with magnetite and uncoated, under simulated crevice chemistry following seawater ingress are shown in Figures 1 through 3.

The ECP of the alloys is higher in the crevice chemistry than in clean SG water when no localized corrosion takes place. This is attributed to the lower pH for the crevice chemistry ( $pH_{25^{\circ}C}$  about 6.0) than in the clean SG water ( pH<sub>25°C</sub> about 9.5 ). The measured ECP values of the alloys in simulated seawater crevices were scattered and had poor reproducibility. This is the consequence of localized corrosion of the tube alloys. In the seawater crevice, samples coated with pure magnetite and magnetite containing 10% Pb or 40% Cu did not show a significant shift in the ECP. However, the addition of 10% PbO or 20% nickel ferrite to the artificial sludge increased the ECP up to more than 100 mV. Zinc silicate additions to the simulated sludge coating may also shift the ECP to a more positive value. After the tests, the samples were cleaned and examined for corrosion. Sludge coated control samples were descaled and examined to make sure that no corrosion was induced by the chemical cleaning process. It was found that magnetite deposit increased the pitting corrosion susceptibility significantly in seawater crevice chemistry. The addition of the copper and lead (both in the metallic and oxide form) and zinc silicate to the magnetite sludge can further enhance the pitting corrosion. Nickel ferrite additions to the magnetite sludge did not show a significant impact on pitting of the tube materials. Inconel 690 was less susceptible to pitting corrosion than Incoloy 800 and Inconel 600 in most surface conditions except in the presence of lead or copper. No direct correlation between the localized corrosion rate and the ECP was found for tube alloys

In order to determine the effect of metallic copper and lead on the ECP, the ECP of metallic copper and lead wires was measured under the simulated seawater crevice chemistry conditions. The ECP of metallic lead was between -650 to -800 mV, and the ECP of metallic copper was between -660 mV to -670 mV. Copper and lead appear to be active in the crevice chemistry at high temperature. Figure 4 shows the time dependence of the ECP of platinum wires with and without a magnetite coating containing 40% metallic copper. The ECP of the coated Pt wire was low at the beginning of the test and increased gradually with time. After 11 hours of exposure to the seawater crevice chemistry at 260°C, the ECP of the coated platinum wire was higher than the uncoated platinum wire. This suggests that the gradual accumulation of the corrosion products (cuprous and/or cupric ion) in the electrolyte makes the crevice electrolyte more oxidizing.

ECP Measurements in Simulated Bruce A SG Crevice Chemistries

The ECP data for Incoloy 800, Inconel 600, Inconel 690 and platinum wire are shown in Figures 5 through 8. The ECP values in "base" BNGS-A and acidic BNGS-A crevice chemistry are

higher than those in clean SG water. The low pH and the addition of the lead oxide and silicon oxide to the simulated Bruce A crevice chemistry are responsible for the ECP shift to the noble (anodic) direction. The ECP value for the alloy materials and platinum are low in the BNGSA alkaline crevice chemistry. It appears that the presence of lead oxide and silicon oxide in the system does not have a significant influence on ECP in alkaline environments. The samples were examined after the tests. (See Figure 9) It is shown that SG tube samples are more susceptible to pitting corrosion in simulated BNGS-A "base" and acidic crevice solutions than in simulated alkaline crevice solution especially under magnetite-coated conditions. A white precipitate was observed on the samples exposed in alkaline crevice solution. EDX analysis indicated that the white precipitate is a silicon compound. No significant pitting corrosion was detected on the three alloys in alkaline crevice solution, whether they were coated with magnetite or not. Pitting was observed on samples exposed to the Bruce A "base" and acidic crevice solution. Magnetite coating enhanced the pitting corrosion significantly in these solutions. Inconel 690 appears less susceptible to pitting corrosion than Inconel 600 and Incoloy 800 in simulated BNGS-A "base" and acidic crevice chemistries.

#### ECP as a Function of Oxygen Concentration in Steam Generator Water

Since the tubes inside crevices are coupled with free-span surface, the ECP of the tube free-span surface will influence the potential of the tube material inside the crevice. It is interesting to examine how the ECP of free-span tube material responds to oxygen and acid excursions.

Oxygen is one of the major oxidants in SG water. The reduction process of oxygen and/or hydrogen peroxide described in equations [3] and [4] will take place at the alloy surface shifting its ECP to a more positive value.

$O_2 + 4H^+ + 4e^- = 2H_2O$	[2]
$H_2O_2 + 2H^+ + 2e^- = 2H_2O$	[3]

Experiments were conducted in simulated normal SG water containing 100 ppb hydrazine and 10 ppm morpholine. The pH of the water was adjusted to 9.5 by adding ammonium hydroxide. The oxygen level was increased gradually at a very slow rate so that a steady potential could be achieved during the measurements. (The average rate of potential increase was about 0.0017 mV/s. Compared to the potential scan rate (ASTM standard) for potentiodynamic polarization, 0.17 mV/s, the rate of potential increase was two orders of magnitudes slower ). Tests were performed on Incoloy 800, Inconel 600, Inconel 690 tube samples and platinum wire. Tests were also performed on samples coated with magnetite to check the effect of the magnetite sludge on the ECP of the tube materials.

The ECP values of Incoloy 800, and platinum, both coated and uncoated, as a function of oxygen concentration in the solution, are presented in Figures 10 and 11. For Inconel 600, and Inconel 690 samples, curves identical to that of Incoloy 800 were obtained.

Due to the oxidation of the alloy materials at high temperature, the response of the ECP to the increase in the oxygen concentration is slower for the alloy materials than for platinum. However, the differences between the alloys are not significant. The ECP of the magnetite-coated samples is more sluggish in responding to the oxygen increase. There is a plateau at about -300 mV. This potential may correlate to the magnetite/hematite conversion. This indicates that under the SG

operating temperature, when an oxygen excursion takes place and the oxygen level exceeds 800 ppb, the SG tube free-span will likely reach a potential of -300 mV if the tube is covered with magnetite. However for alloys not covered with magnetite, the potential may shift to -100 mV as the oxygen concentration in the SG water reaches about 500 ppb. An important point to note is that these tests were performed at relatively low flow rates (in a refreshed-autoclave) compared to those in an operating SG, hence the ECP of the tube materials will be influenced by the mass-transfer process and the ECP value may be under estimated.

## ECP as a Function of pH in Faulted Steam Generator Water Following an Acid Excursion

During a sulfuric acid excursion, the main oxidant in the SG water is protons. The reduction of proton dominates the cathodic reactions on the SG alloy surface. This will increase the ECP of SG tubes significantly.

The ECP of Incoloy 800, Inconel 600, 690 and platinum were measured under simulated faulted SG water condition following a sulfuric acid excursion. The measurement started in a simulated normal SG water containing 100 ppb hydrazine and 10 ppm morpholine. The pH of the water was adjusted to 9.5 by adding ammonium hydroxide. Then the sulfuric acid solution was added to increase the acidity of the solution step by step. Sulfuric acid additions were made only when a steady potential was achieved at a specific pH value. The ECP of Incoloy 800 vs. pH plot is shown in Figure 12. The ECP values of other alloys have similar relations with the solution pH. The ECP of the Incoloy 800 increases with acidity. However, between pH 4.5 and pH 7.5, the variation of ECP with pH is very small. The reason for this is that the high-temperature pH varies very little when pH<sub>25°C</sub> changes from 4.5 to 7.5 during a sulfuric acid excursion.<sup>10</sup> (see Figure 13.) Figure 14. shows that the measured ECP of Incoloy 800 and pH <sub>260°C</sub> are close to a linear relation. The linearity of the plot shows the ECP-pH relation of a hydrogen electrode. It appears that the tube alloys behaves as a hydrogen electrode in high-temperature SG water.

The oxygen excursion may increase the ECP of the SG tube material enough to exceed its pitting potential in the crevice electrolyte and initiate pitting corrosion. However, for a number of reasons the crevice ECP will be lower than the free tube surface: A) The existence of corrosion current flows from the crevice to the free-span resulting in an IR drop across the electrolyte and bubbles; B) In most cases the oxidant concentration is higher outside the crevice during the excursions; and C) The crevice chemistry is very aggressive and the anodic kinetics of alloy materials are enhanced. Tests performed in a simulated heated crevice showed this potential drop inside crevice is from 100 to 160 mV.<sup>11</sup> Although the ECP of tube material in the crevice is lower than pitting potential, localized corrosion can still take place under the crevice conditions. Localized corrosion inside crevices or under deposits is likely initiated through a passive-active trasition mechanism rather than through the passive film breakdown mechanism.<sup>12</sup> As shown in Figure 15, the ECP of the tube free-span,  $E_{FS}$  is located in the passive region, while the ECP of the tube inside the crevice, E<sub>Crevice</sub> is located in the active region due to the potential drop across the crevice electrolyte (IRs), and the bubbles (IRb). When crevice corrosion takes place, the crevice site becomes an anodic area and the free surface outside the crevice becames the cathodic area. The anodic area and the cathodic area are permanently separated. During the propagation stage of the crevice corrosion, the resistance Rs and Rb will limit the corrosion current. However, if oxidant existed inside the crevice site in the form of oxidizing sludge, it will accelerate the

propagation of the localized corrosion. Under this condition, localized corrosion development does not rely solely on the cathodic reaction taking place outside the crevice. The cathodic reduction of the oxidant inside the crevice will provide a shortcut for the corrosion current and thus accelerate the localized corrosion propagation.

#### SUMMARY

- 1. SG tube alloys are susceptible to localized corrosion under simulated SG crevice conditions. Due to the localized corrosion, the measured ECP values of the alloys in simulated seawater crevices chemistries were scattered and had poor reproducibility.
- Magnetite sludge deposits significantly increase the pitting susceptibility of SG tube material in simulated seawater crevice chemistry. Additions of lead, lead oxide, copper, copper oxide, and zinc silicate were found to further increase the pitting corrosion rate. Inconel 690 is less susceptible to pitting corrosion than Incoloy 800 and Inconel 600 in the simulated seawater crevice chemistry at 260°C, except when lead or copper present.
- 3. The ECP of metallic copper and lead in the simulated crevice chemistries at 260°C is below -650 mV. They can not cause anodic polarization of the SG tube materials. However, the accumulation of their corrosion products may turn the crevice electrolyte into an oxidizing environment and shift the ECP of the SG tube materials in the noble direction.
- 4. The ECP of SG tube materials in simulated BNGS-A "base" and acidic solution at 260°C ranges from -220 to -350 mV. This range is at least 250 mV higher than that measured in the alkaline crevice solution. No significant pitting was observed on tube samples, with or without magnetite deposits, after exposure to the simulated BNGS-A alkaline crevice solution at 260°C for up to 24h. Localized corrosion is unlikely to take place in the simulated BNGS-A alkaline crevice environment.
- 5. Incoloy 800, Inconel 600 and 690 are susceptible to pitting corrosion in simulated BNSG-A "base" and acidic crevice solution at 260°C. The presence of the magnetite deposit will enhance the pitting corrosion. However, Inconel 690 is less susceptible to pitting corrosion than Inconel 600 and Incoloy 800 under theses conditions.
- 6. A sulfuric acid excursion can shift the ECP of the SG tube material up to 260 mV, and an oxygen excursion to 1ppm may shift the ECP of SG tube materials up to 450 -600 mV. However, if the tubes are covered with magnetite deposits, the potential of the tube may likely remain at -300 mV due to the magnetite/hematite conversion reaction. The experimental results were obtained at low flow velocity conditions which may result in difference over what might be obtained in a SG. The shift in measured ECP resulting from the acid and oxygen excursion may thus be underestimated for SG conditions.
- 7. The ECP of tube materials inside crevices is likely lower than the ECP at the surface of tube free-span because of the IR drop across the solution and the steam bubbles. Test performed in a simulated heated crevice showed that the potential drop between the tube free span and the area inside the crevice could reach 100 to 160 mV. This potential difference is enough to shift the

tube material inside crevice to the active potential region. The crevice corrosion could be initiated through an active-passive transition mechanism.

- 8. Attempts to use platinum as a probe to detect the faulted condition which may lead to crevice corrosion was not successful. Magnetite deposit on platinum will develop as soon as exposure to high-temperature water. After a few hours of exposure the black deposit becomes visible. The problem of deposits jeopardize the application of platinum as a sensor in high temperature water system.
- 9. Cathodic reactions of oxidizing sludge inside the crevice area provide a short cut to the corrosion circuit during the propagation of crevice corrosion. These reactions will significantly increase the propagation rate of the localized corrosion inside the crevice.

# ACKNOWLEDGEMENTS

CANDU Owners Group, and in particular, Working Party 19, are gratefully acknowledged for their support of this work under WPIR 2019. B. Leger, M. Dupuis and P. Lavoie are acknowledged for carrying out the experimental work. Special thanks are extended to R. L. Tapping, T. S. Gendron, C. W. Turner and K. Franklin for valuable suggestions and support for this work. The author wishes to thank P. V. Balakrishnan and Sandra Pagan of OHN for providing information on high-temperature crevice chemistry conditions and many helpful discussions.

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Figure 1. Measured ECP values of Incoloy 800 tube sample, bare or coated with magnetite and magnetite plus different sludge components, in the simulated seawater crevice chemistry at 260°C.



Figure 2. Measured ECP values of Inconel 600 tube sample, bare or coated with magnetite and magnetite plus different sludge components, in the simulated seawater crevice chemistry at 260°C.



Figure 3. Measured ECP values of Inconel 690 tube sample, bare or coated with magnetite and magnetite plus different sludge components, in the simulated seawater crevice chemistry at 260°C.



Figure 4. ECP vs. time plot of a platinum wire and a platinum wire coated with artificial magnetite sludge containing 40% copper. The Data were obtained in the simulated seawater crevice at 260°C.



Figure 5. ECP values of Incoloy 800 tube sample, bare or coated with magnetite in different simulated BNGS-A crevice chemistries at 260°C.



Figure 6. ECP values of Inconel 600 tube sample, bare or coated with magnetite in different simulated BNGS-A crevice chemistries at 260°C.



Figure 7. ECP values of Inconel 690 tube sample, bare or coated with magnetite in different simulated BNGS-A crevice chemistries at 260°C.



Figure 8. ECP values of platinum wires, bare or coated with magnetite in different simulated BNGS-A crevice chemistries at 260°C.



Figure 9. SG tube samples after the ECP measurements in simulated BNGS-A crevice chemistries at 260°C. The sludge coated samples were examined after chemical cleaning.



Figure 10. The ECP of Incoloy 800 at 260°C as a function of the oxygen concentration in SG water.



Figure 11. The ECP of a platinum wire at 260°C as a function of the oxygen concentration in SG water.



Figure 12. A plot of the ECP of Incoloy 800 at 260°C vs.  $pH_{25°C}$  of the SG water



Figure 13. The  $pH_{260^{\circ}C}$  of the SG water as a function of  $pH_{25^{\circ}C}$  under sulfuric acid excursion conditions. (Results obtained through a MULTEQ type of calculation<sup>9</sup>)



Figure 14: A plot of measured ECP values of Incoloy 800 vs. pH<sub>260°C</sub> of SG water. The dashed line in the plot shows the ECP-pH relationship of a hydrogen electrode. (ECP = - 2.303 RT/F · pH )



Figure 15. A schematic presentation of the mechanism of the crevice corrosion of the SG tube materials.