

**RAI Volume 3, Chapter 2.2.1.3.1, Fourth Set, Number 1:**

Assess information published in Ashida et al. (2007, 2008), Badwe et al. (2006), Pulvirenti et al. (2003), Dunn et al. (2006), Mann and Arya (2002), and Oka et al. (2007), which indicates that models for general and localized corrosion processes may be sensitive to assumptions regarding dripping versus immersion conditions. Based on these considerations, provide additional support, beyond that provided in the SAR and prior RAI responses, that justifies the assumptions that immersion tests are sufficiently representative of predicted repository environmental conditions to determine corrosion behavior.

**Basis:** The DOE models for representing general and localized corrosion processes rely on experiments conducted using metal coupons immersed in solutions (SAR 2.3.6). However, information in Ashida et al. (2007, 2008), Badwe et al. (2006), Pulvirenti et al. (2003), Dunn et al. (2006), Mann and Arya (2002), and Oka et al. (2007) suggests that general and localized corrosion processes may be affected if environmental conditions changed from immersion to dripping. In SAR 2.3.6, DOE has not addressed how the information in these references might affect the DOE representation of general and localized corrosion processes.

For example, Ashida et al. (2008) observed salt deposit formation, micro-pitting, and intergranular corrosion on Alloy 22 specimens exposed to dripping of simulated concentrated water for 40 days at 90°C. These experimental results were not observed in comparable DOE immersion tests of Alloy 22 in simulated concentrated water (SAR, 2.3.6).

Further, Dunn et al. (2006) reported that after dripping simulated pore waters onto Alloy 22 specimens at 110°C for 10 days, the resulting salt deposit had lower nitrate-to-chloride concentration ratio than the starting dripped solution. Similarly, Pulvirenti, et al. (2003) observed nitrate enrichment in the condensates obtained on distillations of unsaturated pore water, which implies a reduction in the nitrate-to-chloride ratio in the salt. DOE has not addressed how these potential changes in salt chemistry observed for dripping conditions may affect DOE models for corrosion that were developed for immersion conditions.

Moreover, Ashida et al. (2007) observed that dripping induced temperature fluctuation may increase the general corrosion rate of mill-annealed Alloy 22 at 90 °C. DOE has not addressed how the potential effects of these temperature fluctuations are evaluated in the DOE general corrosion model.

Finally, experiments conducted by Oka et al. (2007) and Mann and Arya (2002) indicate that several parameters, including material hardness, impact velocity, impact frequency, and diameter and density of water droplets could affect the extent to which the passive film is damaged by drop-impingement erosion. This

information has not been addressed by DOE in SAR 2.3.6 or in the DOE response to RAI 3.2.2.1.3.1-2-006.

The requested information is needed to assess the use of the Alloy 22 general and localized corrosion models in the DOE performance assessment used to demonstrate compliance with 10 CFR 63.114(f).

## 1. RESPONSE

The initial portion of the response provides a synopsis of each paper noted in the RAI basis statement, followed by a discussion of its relevance to the anticipated conditions on the waste package surface. Following the review of the documents noted in the basis statement, this response provides a discussion illustrating the appropriateness of the immersion testing in conservatively capturing the impact which dripping seepage water might have on the corrosion performance of the drip shield or waste package outer corrosion barrier surfaces.

### 1.1 REVIEW OF REFERENCES DISCUSSED IN BASIS STATEMENT

#### 1.1.1 Ashida et al. 2008

While Ashida et al. (2008) did observe localized attack on Alloy 22, the material evaluated in that study had been thermally aged so as to induce significant second phase precipitation, which as noted in SAR Section 2.3.6.7.1, will result in a reduced resistance to localized corrosion initiation. All of the material utilized for the waste package outer barrier will be solution annealed, and as such the material evaluated by Ashida et al. (2008) is not repository relevant.

Ashida et al. (2008) document an electrochemical evaluation of Alloy 22 when exposed to simulated acidified water (SAW), simulated concentrated water (SCW) and basic saturated water (BSW). Samples were both immersed in solution as well as subjected to a dripping electrolyte where salt deposits accumulated on the surface. The Alloy 22 was received in the mill-annealed condition, and then aged for 100 hours at 800°C prior to being evaluated. This thermal processing treatment resulted in significant topologically or tetrahedrally close-packed (TCP) phase precipitation, as the temperature is just above the temperature of the peak transformation rate indicated on the relevant time-temperature transformation curve (BSC 2004, Figure 89). This exact experimental condition is presented in *Aging and Phase Stability of Waste Package Outer Barrier* (BSC 2004, Figure 64), where the grain boundaries can be seen to be completely decorated with TCP phases, along with some precipitation within the bulk of the grains. The equilibrium volume fraction of P phase produced by this thermal processing treatment is predicted to be approximately 15 vol% (BSC 2004, Figure 80). As discussed in SAR Section 2.3.6.7.1, TCP phase precipitation will result in local depletion of the alloy matrix of Cr and Mo, reducing the corrosion resistance and rendering it less resistant to localized corrosion initiation. The impact that the heat treatment had on the corrosion resistance of the material is also highlighted in the results obtained by Ashida et al. (2008). As illustrated in Figure 8 of Ashida et al. (2008), localized attack is observed consistent with the location of the TCP phases in the microstructure (i.e., some small sites in the bulk of the grains, with the majority of the small sites decorating the grain boundaries).

Experimental results are presented in SAR Section 2.3.6 with respect to localized corrosion that was not observed for Alloy 22 in SCW. In the results discussed in the SAR, the material had been properly solution-annealed. The purpose of this solutionizing heat treatment is to dissolve potentially deleterious second phase particles (such as TCP phases) in the microstructure. This is described in more detail in the response to RAI 3.2.2.1.2.1-2-017. Therefore, the material evaluated by Ashida et al. (2008) and the results they obtained are not repository relevant.

### **1.1.2 Ashida et al. 2007**

The results presented by Ashida et al. (2007) are consistent with the temperature-dependent general corrosion model discussed in SAR Sections 2.3.6.2.2 and 2.3.6.3.3.1.

Ashida et al. (2007) evaluated the impact of small temperature fluctuations on the corrosion potential and passive current density of Alloy 22 exposed to simulated Yucca Mountain groundwater. For a very polarizable (i.e., current density is essentially independent of potential) passivated metal, such as Alloy 22 in simulated groundwater, polarized to an applied potential within the passive region, the passive current density is approximately equivalent to the corrosion current density at open circuit. Ashida et al. (2007) observed that as the temperature decreased, the oxidation rate on the metal surface also decreased, and when the temperature increased, the oxidation rate also increased. This behavior was repeatable for replicate cycles. In other words, the change in the observed passive current density was consistent with the temperature change and did not appear to be a function of the number of temperature cycles.

In the SAR, the general corrosion rate (i.e., passive current density in this example) temperature dependence is represented as an Arrhenius relationship (SAR Sections 2.3.6.3.2.2 and 2.3.6.3.3.1). As presented in SAR Section 2.3.6.3.3.1, the activation energy used in the Alloy 22 general corrosion model ranges from a minimum of 5.54 kJ/mol to a maximum of 64.28 kJ/mol, with a mean value of 40.78 kJ/mol. Using the mean activation energy, if the temperature were to increase from 70°C to 90°C (a similar temperature change to that illustrated in Figure 5 from Ashida et al. 2007), then the corrosion rate should increase by a factor of approximately 2.2; this result is consistent with the data presented in Figure 6 of Ashida et al. (2007). However, the temperature fluctuations observed by Ashida et al. (2007) were due to cooler droplets of solution contacting the sample surface, resulting in a drop in the current density. In the SAR, the Alloy 22 general corrosion model is conservative in that it assumes a gradually decreasing temperature without regard to the short term cooling effects induced by seepage (either directly or by evaporation). In other words, the SAR modeled general corrosion rate remains at the higher-temperature level and is not reduced as a result of intermittent contact with a cooler solution.

In summary, while the temperature dependence of the passive current density observed by Ashida et al. (2007) is consistent with the model presented in the SAR, the Project conservatively ignores the temperature reduction associated with seepage water contacting the waste package surface, and models the corrosion rate as if the temperature remained elevated.

### 1.1.3 Badwe et al. 2006

The material evaluated by Badwe et al. (2006) was not solution annealed, and as such likely had significant second phase precipitation which, as noted in SAR Section 2.3.6.7.1, will result in a reduced resistance to localized corrosion initiation. As such, the material utilized by Badwe et al. (2006) was not repository relevant. However, despite this issue with the material being evaluated, the results observed in the paper are consistent with the Alloy 22 localized corrosion initiation model presented in the SAR (i.e., there is a non-zero probability that localized corrosion could initiate).

Badwe et al. (2006) describes experiments performed to determine if heating the electrolyte versus heating the sample made a difference in the observed electrochemical response. Alloy 22 was evaluated in the mill-annealed condition. As the material was not solutionized, it is reasonable to assume that TCP phases were present in the microstructure. As discussed in SAR Section 2.3.6.7.1, TCP phase precipitation will result in local depletion of the alloy matrix of Cr and Mo, reducing the corrosion resistance and rendering it less resistant to localized corrosion.

The electrochemical results presented in the paper reveal that, in most cases, the passive current density was lower by a factor of 5 to more than 50 (depending on the solution) for the case where the electrode was heated. In addition to experiments where the specimen was completely immersed in the electrolyte, experiments were run where the electrolyte was allowed to dry out, changing compositionally (i.e., concentrating) as it evaporated. In the case where SAW solution was evaporated at a temperature of 80°C, upon completion of the experiment there were what appeared to be metastable pits on the surface. Due to the nature of these tests, the solution chemistry was not monitored as the solution evaporated, and as such, it is not possible to determine what the precise chemistry was at the metal/solution interface at the time the metastable events occurred. However, it is reasonable to assume that the concentration of ions such as chloride would increase substantially as the solution dried. Given the poorly defined solution chemistry, if it is assumed that the chloride concentrations and nitrate concentrations gradually increased to saturation, the model results presented in *General and Localized Corrosion of Waste Package Outer Barrier* (SNL 2007a, Section 6.4.4.6) demonstrate that there will be solutions where the model predicts a non-zero probability of localized corrosion initiation. Furthermore, as the material was not solutionized prior to being exposed to the solution, there were likely locations where TCP phases were present, and the resistance to localized corrosion initiation compromised. As such, taking into account the metallurgical condition of the material being evaluated, combined with the increasingly concentrated solutions, the results observed in the paper are consistent with the Alloy 22 localized corrosion initiation model predictions (i.e., there is a non-zero probability that localized corrosion could initiate).

### 1.1.4 Dunn et al. 2006

In the results presented by Dunn et al. (2006), there was not a significant difference observed between most of the concentration ratios of specific inhibitive ions to chloride when comparing the starting solution and the final solid. Furthermore, Dunn et al. (2006) state that the formation of brines with a boiling point above 110°C was not observed, and that the deliquescence of these salt deposits will not result in brines that promote the environmental degradation of Alloy 22.

In the paper by Dunn et al. (2006), several solutions with different water chemistries were evaporated and the final precipitate compared to the solution from which it was derived. In terms of molar ratios, the results in Table 1 were achieved (comparing slides 14 and 17 from the presentation of Figures 4 and 6 in Dunn et al. 2006).

Table 1. Summary of Approximate Initial Solution and Final Salt Deposit Composition Ratios

Ratio	Test Solutions			Final Salt Deposits		
	Ca-Cl	Neutral	Alkaline	Ca-Cl	Neutral	Alkaline
SO <sub>4</sub> /Cl	0.3 to 0.75	0.35 to 1.8	0.23 to 0.55	0.15 to 1.3	0.32 to 1.8	0.22 to 0.55
NO <sub>3</sub> /Cl	0.03 to 0.6	0.4 to 2.5	0.13 to 0.3	0.02 to 0.4	0.017 to 0.45	0.055
HCO <sub>3</sub> /Cl	0.1 to 0.25	1 to 3	3 to 15	0.12 to 0.17	0.45 to 2.6	1.5 to 10
ΣInh. ions/Cl	0.45 to 1.25	1.75 to 7	3.5 to 15	0.35 to 1.9	0.85 to 4	1.75 to 10.2

As can be seen in Table 1, there is considerable scatter in the reported data (with no connection made between specific starting chemistries and specific ending chemistries), as well as a large degree of overlap between the ranges. Given the scatter of the measurements, there is not a significant difference between most of the concentration ratios of specific inhibitive ions to chloride when comparing the starting solution and the final solid. However, for calcium chloride brines, the sulfate to chloride ratio increased. For neutral brines, the nitrate to chloride ratio decreased. Finally, in alkaline brines, the nitrate to chloride ratio and the bicarbonate to chloride ratios decreased. In addition to the apparent compositional changes, Dunn et al. (2006) also states that the formation of brines with a boiling point above 110°C was not observed, and that the deliquescence of these salt deposits, despite the reported reduction in the nitrate to chloride ratio, will not result in brines that promote either localized corrosion or stress corrosion cracking of the Alloy 22 waste package outer corrosion barrier.

### 1.1.5 Pulvirenti et al. 2003

In this study, solutions based on simulated groundwater systems were concentrated by distillation. As discussed in *Analysis of Dust Deliquescence for FEP Screening* (SNL 2007b, Section 6.2), acid degassing is anticipated to occur as solutions are evaporatively concentrated at elevated temperatures. As the solutions were distilled and concentrated, nitrate concentrations increased relative to the chloride concentration within the most concentrated condensate (Pulvirenti et al. 2003, Table 1). This is consistent with project data, which demonstrates that as the temperature is increased, the nitrate to chloride ion ratio within the liquid phase must increase as halite precipitates (SNL 2007b, Figure 6-7[a]). Halite precipitation was confirmed by Pulvirenti et al. (2003) by X-ray diffraction characterization of the “porous solid deposits” found.

Due to the distillation method utilized in this work, acid gases that were released from the solution as it concentrated were condensed and reincorporated back into the solution. This differs from the conditions anticipated within the repository, where the gas volume over the brine will not be limited and, as such, any acid gas produced would be expected to be diluted by the atmosphere within the drift and dissipate (discussed in SNL 2007b, Section 6.2.3). Thus, the solution chemistries reported, particularly the pH values, by Pulvirenti et al. (2003) are not

repository relevant. In addition, the temperatures of the final solutions used by Pulvirenti et al. (2003) ranged from 77°C to 144°C. As seepage water will not be present until the drift wall is below the boiling point of water (SAR Section 2.3.6.4.4.1), the greater than 120°C surface temperatures utilized to produce these brines are also not repository relevant.

### **1.1.6 Mann and Arya 2002**

In this paper, a series of materials commonly used in hydroturbines were exposed to an impinging jet of electrolyte, the goal being to evaluate conditions relevant to hydroturbines and other applications where high-speed electrolyte flow takes place. In this experiment, the various metal coupons were placed on a disc that was rotated at 4,575 rpm, yielding a tangential velocity of 147 m/s. A water jet was fired at an angle of 90 degrees to the samples, and was 4.24 mm wide with a linear velocity of 20.2 m/s. In contrast, a large raindrop 4 to 5 mm in diameter has a terminal velocity of only about 9 m/s and requires a vertical distance on the order of 20 m to achieve that velocity (Beard 1976). The kinetic energy, KE, (i.e.,  $KE = 1/2 mV^2$ ) of a 4 mm diameter droplet at its terminal velocity of 9 m/s will be approximately 1.4 mJ, so if one drop struck a given area per second, the kinetic energy imparted to the surface would be 1.4 mJ/s. The kinetic energy associated with the high velocity impinging jet used as the experimental basis of the referenced paper is vastly larger (approximately 231 J/s) than a gravity-driven single droplet free-falling from the drift wall. Accordingly, the results of this study cannot be used to assess or compare the effect of mechanical impact of a droplet falling from the drift wall and striking the surface of the drip shield or waste package outer corrosion barrier.

### **1.1.7 Oka et al. 2007**

Oka et al. (2007) evaluated the performance of an aluminum alloy exposed to a high-velocity impinging jet of electrolyte. The goal of this work was to evaluate conditions relevant to steam generator turbine blades. In this experiment, the coupons were placed on a stage and a water jet was fired at an angle of 90 degrees to the samples. The water jet was 0.4 mm wide and velocities ranging from 89 to 248 m/s were utilized. By comparison, a large raindrop has a terminal velocity on the order of 9 m/s and requires a vertical distance on the order of 20 m to achieve that velocity (Beard 1976). As with the experimental procedures used by Mann and Arya (2002), the kinetic energy associated with the high-velocity impinging jet is vastly larger (i.e., 177 to 3833 J/s) than a single gravity-driven droplet falling at its terminal velocity of 9 m/s onto the waste package or drip shield surface (i.e., 1.4 mJ/drop), and the results of this study cannot be used to assess or compare the effect of mechanical impact of a droplet striking the surface of the drip shield or the waste package outer barrier. Even given the extremely high flow velocity, the damage rate dropped to zero at a standoff distance of 500 mm or more.

## **1.2 IMPLICATIONS OF DRIPPING CONDITIONS VERSUS IMMERSION CONDITIONS ON THE SOLUTION CHEMISTRIES EVALUATED IN THE SAR**

As presented in SAR Section 2.3.6.4.4.1, seepage is not projected to occur until the drift wall temperature is below 100°C, at which point the waste package surface temperature will be below 120°C. Experimental results that simulate dripping conditions, combined with waste package surface temperatures above 120°C do not represent a repository-relevant condition, and thus

cannot be used to represent the environments that might form on the waste package or drip shield surface. The solution chemistries that are expected to form due to evaporative concentration of seepage water have been accounted for in the immersion experiments performed to date (SNL 2007a, Sections 6.3.2 and 6.4.3.1.3; SNL 2007c, Sections 6.2 and 6.5.1). Therefore, the solution chemistries which might form on the waste package surface due to dripping of seepage water at repository relevant temperatures have been accounted for. Furthermore, since the implementation of both the general and localized corrosion models applies to the entire surface area of the waste package, rather than just local regions where seepage water may have dripped, the models are more conservative representations of the impact that general and localized corrosion will have on the waste package surface than would otherwise result (i.e., the fact that seepage is anticipated to contact only a small portion of the surface area of a waste package is, conservatively, not considered).

A limited volume of solution, such as would be present under the dripping scenario, could have an impact on the corrosion behavior of the waste package outer barrier by providing an increased availability of oxygen (and hence, a potentially larger cathodic current density), as well as by dictating the available surface area available for anodic and/or cathodic sites. In the bulk electrolyte experiments performed within the Long Term Corrosion Test Facility (LTCTF) tanks, the solution volume was large relative to the surface area of coupons inside the tank, and the tank was continuously stirred such that the solution remained in equilibrium with the oxygen concentration in the air blanket over the top of the tank (which occupied a similar volume to the solution, and was continuously replenished). Farmer et al. (2000, Figure 4) verified that the oxygen concentrations in the SAW, SCW, and simulated diluted water tanks in the LTCTF were in equilibrium with the oxygen partial pressure of air. The low corrosion rate (and thus, the low rate of oxygen reduction) of Alloy 22 is insufficient to result in significant depletion of the oxygen within a bulk electrolyte, and thus a thinner electrolyte layer would not result in an increased availability of oxygen over the bulk case. This result is further confirmed by the observation of samples placed at the waterline of the test solution, where the overall corrosion rates observed were comparable to those observed for completely submerged samples. Also, no increased corrosion was observed at any position on the waterline coupons, including regions close to the waterline itself.

Another result of reduced solution volume (such as would occur under the dripping scenario) is that the available anodic and cathodic surface areas are reduced, along with the spatial separation of anodic and cathodic sites. Further, in a bulk solution, the anode and cathode may be separated spatially, such that reactions occurring at the cathode do not act to alter the chemistry of the bulk electrolyte. As the volume of solution becomes small, so does the potential distance between the anodic and cathodic regions of the surface. Cathodic reactions will act to increase the pH value of the limited volume of solution, further reducing the current-delivering capacity of the cathode and significantly reducing the corrosiveness of any solution that is in contact with the waste package outer corrosion barrier (Kelly et al. 2006).

Finally, in terms of the mechanical impact of seepage water droplets, the droplets will be at a far lower velocity, and possess a far lower kinetic energy, than the impinging jets referenced in the RAI basis statement. As an example, the water jet in Oka et al. (2007) was 0.4 mm wide and had a linear velocity ranging from 89 to 248 m/s. By comparison, a large raindrop has a terminal

velocity on the order of 9 m/s and requires a vertical distance on the order of 20 m to achieve that velocity (Beard 1976). As with the experimental procedures used by Mann and Arya (2002), the kinetic energy associated with the high-velocity impinging jet is vastly larger than a single gravity-driven droplet free-falling from the drift wall onto the waste package or drip shield surface and, as such, the results of this study cannot be used to assess or compare the effect of mechanical impact of a droplet striking the surface of the drip shield or the waste package outer barrier. Further, despite the extremely high flow velocity used by Oka et al. (2007) and the softer material (i.e., aluminum) used, it was found that the damage rate dropped to zero at a standoff distance of 500 mm or more. It can be concluded that droplets of seepage water falling from the drift wall will not result in mechanical damage to the waste package outer corrosion barrier or the protective oxide on its surface.

### 1.3 SUMMARY

Although the dripping scenario differs from the total immersion conditions in tests used as a basis for the models presented in the SAR, based on our review of the experimental data presented in these reports, such dripping conditions would not result in an increase in the observed general or localized corrosion rates. As such, the immersion testing data utilized in the total system performance assessment model adequately accounts for the corrosion behavior that would be expected under dripping conditions.

## 2. COMMITMENTS TO NRC

None.

## 3. DESCRIPTION OF PROPOSED LA CHANGE

None.

## 4. REFERENCES

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SNL 2007c. *General Corrosion and Localized Corrosion of the Drip Shield*. ANL-EBS-MD-000004 REV 02 AD 01. Las Vegas, Nevada: Sandia National Laboratories. ACC: DOC.20060427.0002; DOC.20070807.0004; DOC.20071003.0019; LLR.20080423.0006.

**RAI Volume 3, Chapter 2.2.1.3.1, Fourth Set, Number 2:**

- a) Provide the details of the experimental program referenced in the response to RAI 3.2.2.1.3.1-2-004, including corrosion potential, corrosion rate, and surface characteristics of the test specimens (e.g., morphology and chemical composition).
- b) Provide additional details about the analysis of the Alloy 22 oxide film in mixed-salt solution presented in Section 6.4.1.2.3 of SNL (2007), including: (i) whether silica was present in the chromium-rich oxide layer, (ii) whether the oxide film thickness shown in Figure 6-8 included silica-containing deposits, and (iii) whether the corrosion rate as a function of time was measured for test specimens with silica deposits. If the corrosion rate was measured, provide these data.
- c) Assess information published in Sala et al., (1993, 1996, 1998, 1999), Dixit, et al. (2006), Dunn et al., (2005), and Wong, et al. (2004) which indicates that the presence of silica deposits may affect the long-term stability of the passive film and the general corrosion rate of Alloy 22.

**Basis for (a):** In its response to RAI 3.2.2.1.3.1-2-004, DOE stated that the presence of silicate did not significantly impact the corrosion potential and corrosion rate of Alloy 22 under any of the conditions evaluated. DOE did not provide a reference to, or any details of, this experimental program. The staff requires the details of these analyses to complete their review of the DOE RAI response.

**Basis for (b):** In Section 6.4.1.2.3 of SNL (2007), DOE stated that there was no evidence of silica penetration to the underlying substrate. However, energy dispersive x-ray spectroscopic analyses in Figure 6-6 of SNL (2007) show an apparent silicon peak in the chromium oxide layer, which implies that silica penetrated through the chromium-rich inner oxide layer to the underlying substrate. Further, in Figure 6-8 of SNL (2007), DOE does not state whether silica-bearing deposits were included in measurements of the oxide film thickness. Finally, for the specimens referenced in Section 6.4.1.2.3 of SNL (2007), DOE does not mention if corrosion rates were measured for samples containing silica deposits.

**Basis for (c):** Wong, et al. (2004) noted the presence of silica in salt deposits on Alloy 22 specimens exposed to simulated dilute water (SDW), simulated acidic water (SAW), and simulated concentrated water (SCW) solutions in the Long-Term Corrosion Test Facility (Table 4; Figures 2 and 3). Information presented in Sala et al. (1993, 1996, 1998, 1999) indicates that the presence of silica deposits can be associated with intergranular attack and SCC in nickel-based alloys in steam generator environments. Dixit, et al. (2006) also observed silica deposits on Alloy 22 specimens that experienced localized corrosion. Further, Dunn et al.

(2005) showed that the predominant chromium oxide underlayer was lost beneath silica deposits for Alloy 22 specimens exposed to 4 M simulated groundwater at 95 °C for about 2 years. In SAR section 2.3.6.3, DOE has not addressed how this information on potential silica effects has been considered in the DOE general corrosion model.

The requested information is needed to assess the use of the Alloy 22 corrosion models in the performance assessment used to demonstrate compliance with 10 CFR 63.114(f).

## 1. RESPONSE

Section 1.1 of the response provides the data and discussion requested in Part a) of the RAI statement. The response is based on *Technical Basis Document No. 6, "Waste Package and Drip Shield Corrosion," Appendix J (Waste Package: Effects of Silica on Corrosion)* (BSC 2004a), and focuses on the comparisons between silica-containing and silica-free environments.

Section 1.2 of the response addresses each sub-item in part b) of the RAI statement, providing a discussion of: (i) the analysis and results used to determine where silica was present within the oxide layer; (ii) how the oxide film thickness presented in Figure 6-8 in *General and Localized Corrosion of the Waste Package Outer Barrier* (SNL 2007a) was measured as well as whether the reported oxide thickness included silica-containing deposits; and (iii) the data available on the corrosion rate as a function of time for samples where a silica-rich deposit was observed.

Section 1.3 of the response consists of two sub-parts: (i) a concise review of Sala et al. (1993, 1996, 1998, 1999), Dixit et al. (2006), Dunn et al. (2005), and Wong et al. (2004) and their relevance to the anticipated conditions on the waste package surface, and (ii) a discussion of the impact which silica has on the corrosion performance of Ni-Cr-Mo alloys, with an emphasis on the anticipated in-drift environmental conditions.

### 1.1 PART A: IMPACT OF SILICATE ON THE CORROSION POTENTIAL AND CORROSION RATE OF ALLOY 22

The data that formed the basis of the statements referenced in RAI 3.2.2.1.3.1-2-004 by the current RAI basis statement is taken from *Technical Basis Document No. 6, "Waste Package and Drip Shield Corrosion," Appendix J (Waste Package: Effects of Silica on Corrosion)* (BSC 2004a). The data presented therein are all based upon qualified project data.

#### 1.1.1 Corrosion Potential

Alloy 22 was evaluated in simulated acidic water (SAW) and a series of qualified experiments in NaCl solutions at 90°C and 95°C. Long-term exposure of Alloy 22 in SAW at 90°C with and without silicate illustrated that the corrosion potential was nominally identical at time periods of up to 275 days (BSC 2004a, Figure J-1). Furthermore, shorter-term experiments using Alloy 22 in NaCl-based solutions at 95°C illustrated that the corrosion potential of Alloy 22 was

independent of silicate concentration for sodium silicate ( $\text{Na}_2\text{SiO}_3 \cdot 5\text{H}_2\text{O}$ ) concentrations ranging from 0 to 4,000 ppm (BSC 2004a, Figure J-2).

### 1.1.2 Corrosion Rate

The impact of silicate on the corrosion rate of Alloy 22 was evaluated via short term linear polarization measurements, as well as long term potentiostatic polarization measurements. In the former, Alloy 22 was exposed to NaCl solutions containing from 0 to 4,000 ppm sodium silicate for 24 hours, after which the corrosion rate was assessed via linear polarization resistance. The corrosion rate assessed in this manner was found to be independent of the silicate concentration (BSC 2004a, Figure J-3). In addition potentiostatic polarization experiments were conducted at applied potentials of +0.2V (versus Ag/AgCl reference) and +0.4V (versus Ag/AgCl reference) for approximately 28 days. The resulting current density (i.e., the passive current density at each applied potential) was found to be independent of whether or not silicate was present in the test solution (i.e., 1M NaCl at 95°C).

### 1.1.3 Test Specimen Surface Characteristics

Specimens evaluated for long term electrochemical experiments consisted of Alloy 22 rods with a 600-grit surface finish. There were no remarkable deposits or other features noted upon completion of the test. Short-term electrochemical experiments were performed utilizing Alloy 22 disc specimens which had also been polished to a 600-grit surface finish. As with the long-term experiments, there were no remarkable deposits or other surface features noted upon completion of each test.

### 1.1.4 Summary

Based upon these qualified electrochemical data observations, it was concluded that silica did not impact the corrosion potential and/or corrosion.

## 1.2 PART B: ANALYSIS OF THE OXIDE FILM FORMED ON ALLOY 22 IN A SILICA-CONTAINING, MIXED-SALT SOLUTION

The work referenced in the basis statement was performed at the General Electric Global Research Center, and the results are documented in various qualified annual reports. The results and discussion pertinent to the specific questions from the basis statement are presented below.

### 1.2.1 Interpretation of Data Presented in Figure 6-6 from *General and Localized Corrosion of the Waste Package Outer Barrier*

Figure 6.6 in *General and Localized Corrosion of the Waste Package Outer Barrier* (SNL 2007a) represents a cross-sectional transmission electron microscopy (TEM) image of the oxide layer on an Alloy 22 specimen. Compositional information is also presented for a region well within the bulk metal, in addition to a region which includes the surface oxide layer. The compositional information was acquired via energy dispersive x-ray spectroscopy (EDS). Due to the nature of the technique, the sampling volume is considerably larger than the ~10-nm oxide

thickness. In other words, it is not possible to distinguish the targeted inner layer of the oxide from the outer layer of the oxide via EDS. As such, the results which are presented represent an overall average of the oxide layer, without any positional information – thus, the observation of silicon in the spectrum cannot be used to specify where the silicon was within the oxide.

The basis for the statement that silica did not penetrate down to the underlying substrate was x-ray photoelectron spectroscopy (XPS) depth profiling measurements which were made on similar coupons. Specifically, these measurements established that the oxide film on Alloy 22 was enriched with chromium and nickel, and no significant amounts of molybdenum and tungsten were measured. However, a large amount of SiO<sub>2</sub> with various salts on the outer oxide film was detected, but no evidence of penetration of SiO<sub>2</sub> to the underlying substrate was observed (Andresen et al. 2003, Section 3.3).

### **1.2.2 Oxide Thickness Measurement Technique Used in Section 6.4.1.2.3 of *General and Localized Corrosion of the Waste Package Outer Barrier* (SNL 2007a)**

The oxide thickness measurements reported in Figure 6-8 were made via cross-sectional TEM (SNL 2007a). The TEM specimens were extracted from the test coupons using a focused ion beam (FIB) system. The Alloy 22 TEM specimens were ultrasonically cleaned in acetone, a high-purity water bath, and dried in air. An oxide layer approximately 10-nm thick, enriched with chromium, was observed. Electron diffraction patterns showed a thermodynamically stable Cr<sub>2</sub>O<sub>3</sub>-rich oxide film containing nickel and an Alloy 22 composition substrate. Based upon the cross-sectional TEM images, it was concluded that most of the outer surface silica-rich scale had been removed by the ultrasonic cleaning of the specimens received for TEM analysis before being coated with platinum. As such, the oxide thickness measurements reported in *General Corrosion and Localized Corrosion of Waste Package Outer Barrier* (SNL 2007a) do not include the silica-rich surface scale.

### **1.2.3 Long-Term Corrosion Rate Data in the Presence of Silica**

As presented by Andresen and Kim (2007, Section 6B), a general corrosion rate was determined via weight loss following 62 months of immersion in a test solution nitrate/chloride/bicarbonate solution containing 0.27 m silicate at a temperature of 95°C. Three specimens were evaluated and their measured corrosion rates averaged about 3 to 4 nm/yr. These values are towards the low end of the general corrosion rate distribution for Alloy 22 tested for five years in the Long Term Corrosion Test Facility (LTCTF), and as such indicate that the general corrosion rate was not increased by the presence of silica in the exposure solution.

### **1.3 PART C: THE IMPACT OF SILICA ON THE PASSIVE OXIDE FILM ON ALLOY 22**

#### **1.3.1 Review of References Discussed in Part C of RAI Basis**

##### **1.3.1.1 Wong et al. 2004**

The surface deposits on coupons removed from the LTCTF were analyzed using EDS and XPS for composition. Silica was detected on many of the samples, but no information was presented correlating an increase or decrease in corrosion rate relative to the quantity of silica observed. The silica was attributed to silicate present in the exposure solutions.

##### **1.3.1.2 Sala et al. 1993**

Sala et al. (1993) reports on experiments that were performed on Alloy 600 in aqueous solutions containing 5 ppm morpholine, 5 ppm calcium sulfate, and 2 ppm hydrazine, which were adjusted to a final pH of 9.2 using ammonia. To that base solution, additions of 50 g/l magnetite as well as quantities of acetic acid, zinc oxide, arsenic trioxide, copper, lead oxide calcium phosphate, calcium carbonate, and sodium hydroxide were made, with the quantities/presence varying from experiment to experiment. Samples of Inconel Alloy 600 (Ni - 14-17 Cr - 6-10 Fe) were exposed to each solution within an Alloy 800 autoclave at a temperature of 290°C for 22 hours. Upon completion of each exposure, an aluminosilicate layer was observed on the sample surfaces, consisting of an outer layer composed primarily of silica and organic compounds and an inner layer identified as an aluminosilicate associated with calcium. Intergranular attack (IGA) was observed in the more caustic solutions where a protective surface film was not present on the metal surface (the authors speculated that this may be due to the presence of sulfide or electrochemical conditions which favor the formation of a nickel rich layer free of oxides on the metal surface or other similar phenomena). In the conclusions, Sala et al. (1993) state, “silica rich layers are not believed to be deleterious by themselves, but rather by the species (particularly the organic species) they trap and by the surface reaction they allow.”

Considering the conditions evaluated in this study, neither the materials (i.e., Alloy 600) nor the environmental conditions (very high temperature, organic contamination, etc.) are repository relevant. The waste package outer corrosion barrier material, Alloy 22, is significantly more corrosion resistant than Alloy 600 (particularly in terms of its resistance to localized attack) owing to the greater chromium content relative to Alloy 600, as well as the significant additions of molybdenum and tungsten in Alloy 22. As such, the observations made by Sala et al. (1993) cannot be used to predict the deposits which might form on, or the corrosion performance of, the waste package outer corrosion barrier under the expected repository conditions.

##### **1.3.1.3 Sala et al. 1996**

The work presented in this paper represents a continuation of the test program discussed by Sala et al. (1993). In the text, observations of the formation of a multilayer aluminosilicate deposit on Alloy 600 are presented. Preliminary tests are summarized where the “possibility” of IGA exists for Alloy 600 in a near neutral environment containing silica, alumina, magnetite,

morpholine, acetic acid, and hydrazine at a temperature of 310°C. Information, similar to that which was presented in the previous paper (Sala et al. 1993), again emphasizes the role of the organics.

As with the results presented by the Sala et al. (1993) paper, neither the materials (i.e., Alloy 600) nor the environmental conditions (very high temperature, organic contamination, etc.) are repository relevant. The waste package outer corrosion barrier material, Alloy 22, is a significantly more corrosion resistant material (particularly in terms of its resistance to localized attack) owing to the increased chromium content relative to Alloy 600, as well as the significant additions of molybdenum and tungsten. As such, the observations made by Sala et al. (1996) cannot be used to predict the deposits which might form on, or the corrosion performance of, the outer corrosion barrier under the expected repository conditions.

#### **1.3.1.4 Sala et al. 1998**

This paper evaluates the corrosion performance of Alloy 600 based on testing in solutions at pH values of 4 and 8 containing morpholine, hydrazine, acetic acid, silica, calcium carbonate, alumina, and calcium phosphate. Tests were performed in an Alloy 800 autoclave at a temperature of 290°C. Electrochemical impedance spectroscopy was used to interrogate the samples during the test. As noted by Sala et al. in their papers from 1993 and 1996, the formation of an aluminosilicate layer was observed. Beneath this layer, particularly as the pH was reduced, was a non-protective, chromium containing layer.

As with the results presented in the Sala et al. (1993) and Sala et al. (1996) papers, neither the materials (i.e., Alloy 600) nor the environmental conditions (very high temperature, organic contamination, etc.) are repository relevant. The waste package outer corrosion barrier material, Alloy 22, is a significantly more corrosion resistant material (particularly in terms of its resistance to localized attack) owing to the increased chromium content relative to Alloy 600, as well as the significant additions of molybdenum and tungsten. As such, the observations made by Sala et al. (1998) cannot be used to predict the deposits which might form on, or the corrosion performance of, the outer corrosion barrier under the expected repository conditions.

#### **1.3.1.5 Sala et al. 1999**

In this paper, electrochemical impedance spectroscopy data for experiments similar to those in the Sala et al. (1998) paper are presented. As with the other papers in the series, IGA was observed under the thin, brittle and non-protective layer which forms in the presence of silica, phosphate, and organic species. Tests were performed again on Alloy 600, primarily in an Alloy 800 autoclave and a temperature of 290°C.

As with the results presented in the Sala et al. papers from 1993, 1996, and 1998, neither the materials (i.e., Alloy 600) nor the environmental conditions (very high temperature, organic contamination, etc.) are repository relevant. The waste package outer corrosion barrier material, Alloy 22, is a significantly more corrosion-resistant material (particularly in terms of its resistance to localized attack) owing to the increased chromium content relative to Alloy 600, as well as the significant additions of molybdenum and tungsten. As such, the observations made

by Sala et al. (1999) cannot be used to predict the deposits that might form on, or the corrosion performance of, the outer corrosion barrier under the expected repository conditions.

#### **1.3.1.6 Dixit et al. 2006**

This paper presents the results from a series of autoclave experiments in highly concentrated nitrate and chloride environments. As discussed in *Analysis of Dust Deliquescence for FEP Screening* (SNL 2007b, Section 6.4.2.1.3[a] and Figure 6-7[a]), these experiments were performed under conditions that are not physically possible within the repository. Furthermore, the silicon containing phase (an aluminosilicate with chloride) was only observed in the experiments performed at 220°C with a NO<sub>3</sub>:Cl ratio of 0.5, but localized corrosion was observed in all conditions evaluated in that report. In other words, the presence of the silica does not appear to be the cause of the observed localized corrosion. Furthermore, the source of the silica in this case is believed to be the dissolution of the crevice formers in the high pressure, high temperature environment, and did not originate from the solution. Therefore, while both localized corrosion and the presence of silica were observed by Dixit et al. (2006), they were not demonstrated to be related. Finally, as the experiments were performed under conditions that deviate far from any expected repository environment, they cannot be used to conclusively predict what may or may not take place on the waste package outer corrosion barrier.

#### **1.3.1.7 Dunn et al. 2005**

Dunn et al. (2005, Section 3.3.2) discussed the nature of the oxide on the surface of Alloy 22. The reference states that the surface films formed on Alloy 22 had two surface areas: an outer deposition layer that contained silica as a result of glass test cell dissolution, and an inner layer rich in all elements in the alloy. The inner layers were determined to be rich in the metallic species of nickel and chromium with low concentrations of the oxide species. Following this statement, the authors reference DOE data where Alloy 22 polarized to +250 mV<sub>Ag/AgCl</sub> in basic saturated water (BSW) had a silica-rich oxide surface layer beneath which the chromium rich layer appeared to not be present. The analysis of these specimens is discussed in detail in *Waste Package and Drip Shield Materials: Passive Film Characteristics, Growth, and Stability* (BSC 2004b, Section N.4.5). In alkaline solutions, in this case polarized to +250 mV<sub>Ag/AgCl</sub> in BSW at a temperature of 90°C (BSC 2004b, Table N-3), the specimen surface was found to have a coating of porous silica scale due to dissolution from the glassware. However, a compact oxide layer was found to be present beneath this porous silica layer, composed primarily of chromium oxide. In all cases, a non-crystalline, chromium (III) oxide was found to be responsible for the passive behavior of the metal (BSC 2004b, Section N.4.5).

Dunn et al. (2005) also reported on creviced samples in silica containing environments. For these experiments, areas within crevice sites had an outer deposition layer that contained silica as a result of glass test cell dissolution. These results on passive film characterization of Alloy 22 using x-ray photoelectron spectroscopy suggest that the oxide films formed on Alloy 22 in the passive region consist primarily of the Cr<sub>2</sub>O<sub>3</sub> species, and these Cr<sub>2</sub>O<sub>3</sub>-rich surface films are responsible for the passive behavior of the alloy. As their study did not specifically explore the impact of the porous silica layer, it should be noted that Dunn et al. (2005) did not report an increase in either metal oxidation/corrosion rates or localized corrosion initiation susceptibility in

the presence of silica-rich deposits. Thus, while the results described by Dunn et al. (2005) do illustrate that the nature of the silica deposit and underlying passive oxide film does vary with environmental conditions, this is consistent with results presented in BSC 2004b, and neither states nor implies that passivity is lost resulting in an increase in the general corrosion rate by the presence of a porous, silica rich deposit on the sample surface.

### **1.3.2 Impact of Silica on the Corrosion Performance of Ni-Cr-Mo Alloys**

Silica is likely to be present within every potential repository relevant water composition. The range of measured silica concentrations employed by the LTCTF reflects this (SNL 2007a, Table 6-3 in ERD 02), and as such, any potential impact that repository relevant silica concentrations might have on the general corrosion rate of the engineered barrier system components has been captured, and thus is included in the analyses presented in the SAR. The impact of silicate on the open circuit potential (i.e., no effect) is discussed in SAR Section 2.3.6.4.2.1 and the environments used in SAR Table 2.3.6-13.

As discussed by Dunn et al. (2005), and in *Technical Basis Document No. 6: Waste Package and Drip Shield Corrosion, Appendix J: Waste Package: Effects of Silica on Corrosion* (BSC 2004a) and *Technical Basis Document No. 6: Waste Package and Drip Shield Corrosion, Appendix N: Waste Package and Drip Shield Materials: Passive Film Characteristics, Growth, and Stability* (BSC 2004b), while the presence of silica may lead to the deposition of a silica-rich layer on the surface of Alloy 22, the underlying oxide will remain a protective Cr<sub>2</sub>O<sub>3</sub>-rich layer. Experiments performed under repository relevant conditions have further demonstrated that when a silica-rich surface layer is produced on the Alloy 22 surface, it does not lead to an increase in the general corrosion rate or a reduction in the resistance to localized corrosion initiation. Therefore, the presence of silica in groundwater that may come into contact with the waste package outer corrosion barrier will not increase the general corrosion rate. Furthermore, even when silica concentrations have been increased to levels well above repository-relevant concentrations, no quantifiable impact on the general corrosion rate was observed.

## **2. COMMITMENTS TO NRC**

None.

## **3. DESCRIPTION OF PROPOSED LA CHANGE**

None.

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