Characterization of Corrosion Behavior of Alloy 22 Regarding its Lifetime Performance as a Potential Nuclear Waste Container Material

R.B. Rebak, R.D. McCright

U.S. Department of Energy

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National
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Raúl B. Rebak and R. Daniel McCright
Lawrence Livermore National Laboratory, Livermore, CA 94550, USA

ABSTRACT

Alloy 22 (UNS N06022) was proposed for the corrosion resistant outer barrier of a two-layer waste package container for nuclear waste at the potential repository site at Yucca Mountain in Nevada (USA). A testing program is underway to characterize and quantify three main modes of corrosion that may occur at the site. Current results show that the containers would perform well under general corrosion, localized corrosion and environmentally assisted cracking (EAC). For example, the general corrosion rate is expected to be below 100 nm/year and the container is predicted to be outside the range of potential for localized corrosion and environmentally assisted cracking.

Keywords: nuclear waste container, N06022, general and localized corrosion, stress corrosion cracking

INTRODUCTION

Yucca Mountain is under investigation as the potential site for geological disposal of commercial nuclear spent fuel and some other forms of high-level nuclear waste in the United States. The site is located about 160 km northwest of Las Vegas in the state of Nevada on land owned by the federal government [1]. The repository for the waste disposal would be constructed in a layer of tuff rock, at a depth approximately 300 meters below the ground surface and about 300 m above the permanent water table at this location. The overall strategy in isolating high-level nuclear waste is to make use of the natural barriers present in the host geologic site along with the construction of a series of engineered barriers. The current waste package design consists of two concentric metal containers. The outer container would be made of Alloy 22 or UNS N06022, which is among the most corrosion resistant of all engineering materials. The approximate composition of Alloy 22 (in weight %) is: ~56 Ni, 22 Cr, 13 Mo, 3 W and 3 Fe. The purpose of this outer container is to provide protection against corrosion and its design thickness is 20 mm. The inner container would be made of a 50-mm thick nuclear grade type 316L stainless steel or UNS S31603. The intended purpose of the inner barrier is to provide shield for radiation and mechanical integrity. The engineering barrier also includes a detached drip shield that would be emplaced above the waste package to deflect any falling water from the rock onto the container. The proposed material for the drip shield is Titanium Grade 7 or UNS R52400 [1].

In general, the waste containers have a common diameter (1.8 m), and the length varies according to the type of waste (roughly 3.6 m for the reprocessed waste to 5.7 m for most of the spent nuclear fuel waste packages). By design, it is required that the container material

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(Alloy 22) would not release radioactive material to the surrounding mountain for 10,000 years. Due to radioactive decay, the temperature of the container may rise to a maximum of nearly 160°C during the first period of emplacement (~1,000 years). The magnitude of the temperature depends on the number of containers per unit length of the emplacement tunnels.

The container may corrode only if water is present. The climate at Yucca Mountain is dry and water quantities reaching the waste package surface are limited. In the improbable case that water enters in contact with the container, it would be in the form of a multi-ionic solution. This solution may form through two different mechanisms, namely (1) Dripping from the drift wall and concentrating on the container and (2) Deliquescence of salt or mountain dust that may accumulate on top of the container during dry periods. In both cases the solution that may enter in contact with the container would be concentrated. The ground waters that are associated with Yucca Mountain have been well characterized [2,3]. Table 1 shows the composition of underground water (J-13) from the repository site. This is a case of infiltration water and is a bicarbonate-dominated type of water with significant concentrations of sulfate, nitrate, and chloride. Alkali cations such as sodium and potassium dominate over alkaline earth such as calcium and magnesium. The water also contains some fluoride and is saturated in silica. Its pH is just slightly alkaline in the dilute water, but with concentration of its salts, the pH is raised into the 10-11 range, mainly because of the bicarbonate/carbonate buffering effect. Another type of water that may drip on the container is pore water [3]. This water is characterized by the absence of any carbonate species. It contains significant amounts of sulfate, nitrate, and chloride, but the alkaline earth cations dominate over the alkali cations. It is slightly acidic when diluted but when concentrated, it remains near neutral, since there is no carbonate to alkalize the solution. In both types of water, a high concentration of nitrate develops (Table 1) [3]. The environment may also be altered by microbial activity (e.g. bacteria and fungi) and by radiation, which may cause radiolysis of water. Table 1 also shows the composition of laboratory-prepared concentrated aqueous solutions that could be representative of environments that would enter in contact with the container.

Table 1: Composition of Representative Environments in mg/L

Ion	J-13 Well Water pH 7.4	Unsaturated Zone (UZ) Pore Water pH 5.6	Simulated Concentrated Water (SCW) pH 10.3	Simulated Acidified Water (SAW) pH 2.8	"1000-fold" Pore Water pH 6.3
K^+	5.04	0.01	3400	3400	661
Na^+	45.8	9	40900	40900	727
Mg^{2+}	2.01	12	< 1	1000	470
Ca^{2+}	13	65	< 1	1000	273
F^-	2.18	0	1400	0	<251
Cl^-	7.14	77	6700	24250	681
NO_3^-	8.78	12	6400	23000	501
SO_4^{2-}	18.4	79	16700	38600	25
HCO_3^-	128.9	66	70000	0	<2.2
SiO_3^{2-}	61.1	46	~ 40	~ 40	52

Since the maximum temperature of the containers is approximately 160°C, dry oxidation is not considered a life-limiting degradation mode. If water is present, there are three types of corrosion degradation modes that may occur. These are: (1) General or uniform corrosion, (2) Localized corrosion and (3) Environmentally assisted cracking (EAC). Figure 1 outlines how these three types of corrosion may impact the lifetime performance of the containers. Obviously, all types of corrosion will be influenced by the metallurgical condition of the alloy and the type of environment that is present. Metallurgical condition includes for example welded vs. wrought and annealed vs. aged microstructures. The environmental aspect includes temperature, solution composition (redox potentials) and effect of radiation or microbial activity. Furthermore, both the metallurgical and environmental conditions will determine the free corrosion potential (E_{corr}) of the container.
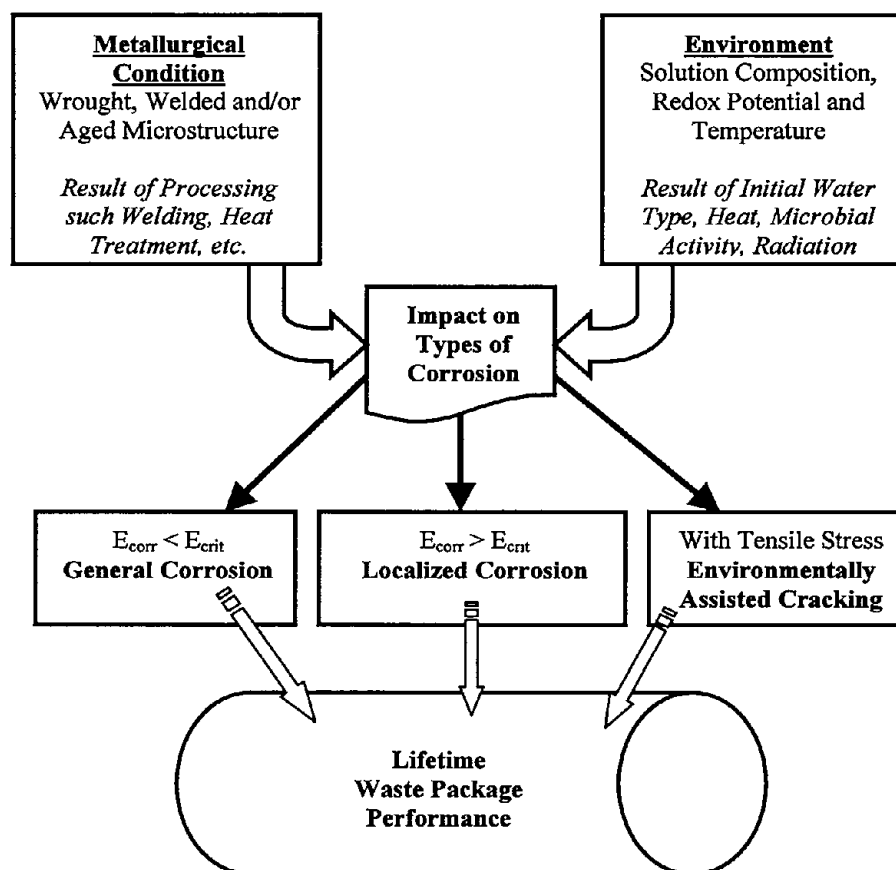
FIGURE 1: General Model For Corrosion Degradation of Alloy 22

General or Uniform Corrosion

General corrosion (or passive corrosion) is the uniform thinning of the container at its corrosion potential (E_{corr}). The degradation model assumes that general corrosion at E_{corr} will progress uniformly over a large surface at a (time-independent) constant rate. The model assumes that the depth of penetration (x in nm) or thinning of the container is equal to the

corrosion rate (CR in nm/year) multiplied by the time (t in years) that the container is exposed to an environment under which general corrosion occurs. That is, $x = CR \cdot t$. General corrosion rates are being measured using long-term weight-loss immersion tests and short-term electrochemical methods. Values of corrosion rate are being determined in realistic multi-ionic solutions in a wide range of temperatures, pH and E_{corr}. Table 2 shows values of corrosion rates determined under different testing conditions using different testing methods. Farmer et al. [4] reported that after 2-year immersion of Alloy 22 coupons in concentrated aqueous electrolytes from pH 2.8 to 10 in the temperature range between 60°C and 90°C, the average corrosion rate by mass loss was approximately 20 nm/year (Table 2). Rebak et al. [5] performed electrochemical impedance studies of MA and aged Alloy 22 at E_{corr} in J-13 water at 95°C. After immersion times of less than three hours in normally aerated solutions, they reported corrosion rates of approximately 200 nm/year (Table 2). Dunn and Brossia [6] performed constant potential tests on Alloy 22 immersed in deaerated 0.028 M and 0.5 M NaCl solutions of pH 2.7 and 8 at 20°C and 95°C. After holding the potential constant for 48 h in the range between 0 and +0.4 V [SCE], they reported passive currentsthat translated into corrosion rates of less than 500 nm/year. Lloyd et al. [7] performed constant potential tests on Alloy 22 in deaerated 1 M NaCl + 0.1 M H_2SO_4 solution at temperatures between 25°C and 85°C. At 75°C and at an applied potential of +200 mV [Ag/0.1MAgCl], they reported a current density of 1.58×10^{-8} A/cm² after 10 h of testing. This current density translates into a corrosion rate of 138 nm/year (Table 2). Evans and Rebak [8] carried out polarization resistance tests at the E_{corr} of Alloy 22 in simulated acidified water (SAW) (Table 1). After an immersion of 1 h in deaerated conditions they reported corrosion rates ranging from 480 nm/year at 30°C to 1440 nm/year at 90°C [8]. However, after one-week immersion in aerated SAW, the corrosion rate decreased more than one order of magnitude to 23 nm/year at 30°C and to 103 nm/year at 90°C (Table 2) [8]. Lian et al. [9] performed constant potential tests in aerated and deaerated SAW and SCW (Table 1) at 90°C and at potential values of +0.1 V and +0.4 V [Ag/AgCl]. For example, after one day testing in deaerated solutions at +0.1 V, they reported corrosion rates of 460 nm/year in SAW and 1250 nm/year in SCW [9]. After they extrapolated the one-day decaying current density values to one year, the corrosion rates became 21 nm/year and 100 nm/year, respectively [9]. The available data in the literature reported above [4-9] shows that the corrosion rate of Alloy 22 in acidic to alkaline brines is, as a maximum, approximately 200 nm/year, which corresponds to a maximum penetration of 2 mm for the design period of 10,000 years.

Table 2: General Corrosion Rates for MA Wrought Alloy 22

Reference	Environment	Measurement Method, Conditions	Corr. Rate (nm/year)
Farmer et al. [4]	Concentrated aerated multi-ionic electrolytes pH 2.8 to 10 at 60°C and 90°C	Weight-loss Immersion Tests for 2 years at E_{corr}	20
Rebak et al. [5]	Aerated J-13, pH 7.4, 95°C	AC Impedance at E_{corr} after ~3 h immersion	200
Lloyd et al. [7]	Deaerated 1 M NaCl + 0.1 M H_2SO_4 solution at 75°C	Constant Potential +200 mV [Ag/AgCl] for 10 h	138
Evans and Rebak [8]	One week in aerated SAW pH 2.8 at 30°C to 90°C	Polarization Resistance at E_{corr}	23 to 103

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Localized Corrosion

Localized corrosion (crevice corrosion) is a type of corrosion in which the attack progresses at discrete sites or in a non-uniform manner. The degradation model (Figure 1) assumes that localized corrosion will only occur when E_{corr} is equal or greater than a critical potential (E_{crit}). That is, if $E_{corr} < E_{crit}$, general corrosion will occur. E_{crit} can be defined as a certain potential above which the current density or corrosion rate of Alloy 22 increases significantly and irreversibly above the general corrosion rate of the passive metal. In environments that promote localized corrosion, E_{crit} is the lowest potential that would trigger localized (e.g. crevice) corrosion. In environments that are benign towards localized corrosion E_{crit} would correspond to a transpassive or oxygen evolution potential. In every case, the margin of safety against localized corrosion will always be given by the value of $\Delta E = E_{crit} - E_{corr}$. The higher the value of ΔE, the larger the margin of safety for localized corrosion. It is important to note here that the values of both E_{corr} and E_{crit} depend of the surface condition of Alloy 22, on the composition of the environment (e.g. chloride concentration) and the temperature. Additionally, the value of E_{crit} depends on the way (method) it is measured.

Alloy 22 is extremely resistant to localized corrosion such as pitting corrosion and crevice corrosion. Critical temperatures for pitting and crevice corrosion determined through immersion tests in aggressive solutions are always among the highest for nickel alloys [10, 11]. Electrochemical tests also showed that Alloy 22 was resistant to crevice corrosion.For example, Dunn et al. [12-14] showed that the repassivation potential for crevice corrosion (E_{crit}) of Alloy 22 was approximately 300 mV [SCE] in 1 M NaCl at 95°C. Rebak et al. [11] performed cyclic polarization tests using seven types Ni-Cr-Mo alloys. They showed that the repassivation potential for MA Alloy 22 in 1 M NaCl solution at 50°C was above 400 mV [SCE]. Evans and Rebak performed cyclic polarization experiments in 5 M $CaCl_2$ pH 6.4 solutions [8]. They reported that the passivity breakdown potential (E_{crit}) was higher than 800 mV [SSC] at 75°C and decreased to 195 mV [SSC] at 90°C [8]. Electrochemical tests reported above [8, 11-14] showed that Alloy 22 was susceptible to localized (crevice) corrosion at high anodic potentials in pure concentrated chloride solutions such as sodium chloride [11-14] and calcium chloride [8]. However, when nitrate was added to the chloride containing solution, the susceptibility of Alloy 22 to crevice corrosion decreased or disappeared [6, 8, 15]. Dunn and Brossia reported that the crevice repassivation potential of welded Alloy 22 in 0.5 M NaCl at 95°C was 0 V [SCE] [6]. When 0.05 M nitrate was added ($Cl^-/NO_3^- = 10$) the crevice repassivation potential remained unchanged; however, when 0.1 M and higher nitrate concentration was added ($Cl^-/NO_3^- \leq 5$), the crevice repassivation potential was near 350 mV [SCE] and the alloy was free from crevice corrosion [6]. Kehler et al. [15] tested the susceptibility of Alloy 22 to crevice corrosion as a function of temperature and pH in 5 M LiCl containing different amounts of oxyanions (sulfate and nitrate). They reported that as the ratio $Cl^-/(NO_3^- + SO_4^{2-})$ decreased from 100 to 10 to 1, the susceptibility to crevice corrosion decreased [15]. Evans and Rebak [8] reported that the breakdown potential of Alloy 22 in 5 M $CaCl_2$ solution at 90°C was 0.195 V [SSC]; however, when 0.5 M $Ca(NO_3)_2$ was added ($Cl^-/NO_3^- = 10$), the breakdown potential (E_{crit}) increased to 0.76 V [SSC] and the alloy was free from localized corrosion.

It is possible to initiate localized attack in Alloy 22, but the environmental circumstances for doing this are extreme. Of particular concern is an environment issuing from evaporative concentration of the pore water composition (Table 1), since this water would be "rich" in calcium chloride. Nevertheless, it is very unlikely that a pure calcium, sodium or lithium chloride environment would ever form under the repository conditions of Yucca Mountain.

By the proper nature of the mountain dust, it is expected that inhibiting oxyanions such nitrate will always be present. Nonetheless, following the modeling criteria requiring a large positive ΔE value as a condition that shows resistance to localized corrosion, Alloy 22 would still be a safe choice. Table 3 lists values of E_{corr} and E_{crit} from the cited references above, showing that in all tested circumstances (even pure high concentration chloride solutions) the value of ΔE was always positive. The values of E_{crit} corresponded to the potential value at which the current density was 20 $\mu A/cm^2$.

Table 3: Parameters for Localized Corrosion Susceptbility

Reference	Tested Conditions. Type of E_{crit}	E_{corr} (mV)	E_{crit} (mV)	ΔE (mV)
Rebak et al. [5]	Potentiodynamic Polarization in aerated J-13, 95°C. Anodic Peak.	-300 [SCE]	250 [SCE]	550
Evans and Rebak [8], Lian et al. [9]	Potentiodynamic Polarization in aerated SAW, pH 2.8, 90°C. Transpassivity	350 [SSC]	700 [SSC]	350
Lian et al. [9]	Potentiodynamic Polarization in aerated SCW, pH 7.8, 90°C. Anodic Peak	-300 [SSC]	200 [SSC]	500
Evans and Rebak [8]	Potentiodynamic Polarization in deaerated 5 M CaCl$_2$, pH 6.4 at 90°C. Localized Corrosion	-410 [SSC]	195 [SSC]	605
Evans and Rebak [8]	Potentiodynamic Polarization in deaerated 5 M CaCl$_2$ + 0.5 M Ca(NO$_3$)$_2$ pH 5.8 at 90°C. Transpassivity	-420 [SSC]	760 [SSC]	1180

Environmentally Assisted Cracking (EAC)

EAC is a phenomenon by which certain ductile metallic materials loose ductility in presence of tensile stresses in specific corrosive environment. That is, for EAC to occur, the simultaneous presence of three factors must be present. These are (1) a susceptible microstructure, (2) tensile stresses and (3) an aggressive environment. If one or more of these variables is eliminated, EAC will not occur. Wrought mill annealed (MA) Alloy 22 is highly resistant to EAC in most environments, including acidic concentrated chloride solutions [14, 16-19]. Dunn et al. did not find EAC when they tested Alloy 22 in 14 molal Cl (as MgCl$_2$) at 110°C and 9.1 molal LiCl at 95°C under controlled potential [14, 16-17]. They used wedge opening loaded double cantilever beam (DCB) and compact tension (CT) specimens at stress intensities in the range 32 to 47 MPa·m$^{1/2}$ for times as long a 52 weeks [14, 16-17]. Rebak reported that Alloy 22 U-bend specimens did not suffer EAC when exposed to 45% MgCl$_2$ at 154°C for up to 6 weeks [18]. Estill et al. performed slow strain rate tests (SSRT) at a 1.6 x 10^{-6} s^{-1} strain rate at the corrosion potential (E_{corr}) in 4 M NaCl at 98°C, saturated CaCl$_2$ (>10 M Cl$^-$) at 120°C and 1% PbCl$_2$ at 95°C [19]. None of these specimens showed a loss of ductility or secondary cracking [19].

Even though Alloy 22 is resistant to EAC in concentrated chloride solutions, it may be susceptible under other severe environmental conditions [20-23]. Andresen et al. tested the susceptibility of Alloy 22 to EAC at the corrosion potential (E_{corr}) in basic saturated water (BSW) at 110°C [20]. This BSW multi-ionic solution is another version of concentrated

solutions that might be obtained after evaporative tests of Yucca Mountain ground waters (more concentrated than SCW in Table 1). Using the reversing DC potential drop technique, Andresen et al. reported a crack grow rate of 5 x 10^{-13} m/s in a 20% cold-worked specimen loaded to a stress intensity of 30 MPa·m$^{1/2}$. This EAC testing was carried out in air saturated BSW water of pH ~ 13. The testing conditions used by Andresen et al. were highly aggressive and, in spite of that, the measured crack growth rate was near the detection limit of the system [20]. Rebak et al. reported that Alloy 22 U-bend specimens suffered transgranular EAC when they were exposed for 336 h to aqueous solutions of 20% HF at 93°C and to its corresponding vapor phase [21]. The liquid phase was more aggressive than the vapor phase [21]. Pulvirenti et al. reported transgranular cracking in one out of four Alloy 22 U-bend specimen exposed for 15 days at 250°C in concentrated ground water contaminated with 0.5 % lead (Pb) and acidified to pH 0.5 [22-23]. Estill et al. performed slow strain rate tests, cyclic loading tests and U-bend tests in large variety of environments (temperature, applied potential and solution composition) [19]. They only reported EAC on MA Alloy 22 through SSRT in saturated concentrated water (SCW) at 73°C and at a potential of +0.4V [SSC] [19]. Even though, Alloy 22 may be susceptible to environmentally assisted cracking in a few environments at E_{corr} [21-23], these are not realistic for the Yucca Mountain emplacement site. There are not published results in which Alloy 22 would suffer EAC in multi-ionic solutions at E_{corr}. King et al. [24] reported cracking in SCW solution only at applied anodic potentials at least 0.3 V higher than E_{corr}.

EAC is unlikely to initiate and grow in Alloy 22 in the repository conditions where the only stresses are residual fabrication (forming and welding) stresses. Nevertheless, it is planned to mitigate further the possibility of EAC by putting compressive stresses on the container surface. Assembly welds made during the container fabrication are expected to be solution annealed to remove residual stress and restore the alloy homogeneity in the welded region. This will help to mitigate any possible EAC initiation. Stress mitigation processes around the final closure weld (top lid onto the container body, filled with the waste form) have also been proposed. The two processes under active consideration are laser peening and induction annealing. Laser peening produces a compressive stress layer on the welded waste package surface, and with optimization of the process, this layer achieves ~3 mm in depth. Induction annealing is local heating of the weld region along with rapid cooling to relieve the stress and to form a more favorable microstructure. This process is also being optimized.

CONCLUSIONS

(1) The three corrosion failure modes that have been identified for the performance of the containers are general corrosion, localized corrosion and environmentally assisted cracking.

(2) It is expected that in relevant environments such as acidic and alkaline multi-ionic brines, Alloy 22 will remain passive. Under these conditions, general corrosion rates are expected to be below 100 nm/year.

(3) In multi-ionic brines, Alloy 22 is immune to localized corrosion. Crevice corrosion may occur in conditions of pure concentrated chloride solutions near the boiling point at anodic potentials that are at least 300 mV more positive than E_{corr}.

(4) Alloy 22 is resistant to environmentally assisted cracking (EAC) in relevant conditions of Yucca Mountain. The elimination of residual stresses should further mitigate the occurrence of EAC.

ACKNOWLEDGMENTS

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