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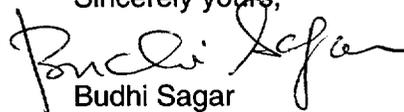
Subject: Programmatic review of Materials Research Society 25<sup>th</sup> International Symposium on the Scientific Basis for Nuclear Waste Management paper titled "Localized Corrosion Susceptibility of Alloy 22 as a Waste Package Container Material"

Dear Mrs. DeMarco:

Attached is a paper to be published in the Proceedings of the Material Research Society (MRS) 25<sup>th</sup> International Symposium on the Scientific Basis for Nuclear Waste Management held on November 26–29, 2001 in Boston. The paper is focused on the experimental investigations of the localized corrosion susceptibility of alloy 22, the material preferred by the DOE for the waste package outer container. In addition to the mill annealed material, welded and thermal aged conditions were investigated to evaluate the effect of fabrication processes on the resistance to localized corrosion through the measurement of the repassivation potential for crevice corrosion. The beneficial effect of nitrate as an efficient inhibitor of crevice corrosion promoted by chloride was also investigated. The paper updates information presented in IM 1402.571.170 titled Effect of Environment on the Corrosion of Waste Package and Drip Shield Materials.

Please contact Gustavo Cragnolino at (210) 522-5539 if you have any questions regarding this paper.

Sincerely yours,

  
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## **Localized Corrosion Susceptibility of Alloy 22 as a Waste Package Container Material**

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### **ABSTRACT**

Alloy 22 is the material preferred by the U.S. Department of Energy for the waste package outer container for geological disposal of high-level radioactive waste at the proposed repository site in Yucca Mountain, Nevada. The susceptibility of Alloy 22 to localized corrosion is an important consideration in the evaluation of the waste package behavior and the assessment of the overall performance of the proposed repository. Factors significant to localized corrosion susceptibility include temperature, the chemical composition of the environment in contact with the waste package, and microstructural effects related to container fabrication processes. Environmental chemical composition and temperature effects were examined by measuring the repassivation potential for crevice corrosion in chloride-containing solutions at temperatures ranging from 80 to 150 °C. The effect of potentially inhibiting anionic species, such as nitrate, was also determined. In addition to the mill annealed material, tests were conducted on both welded and thermally aged material to evaluate the effects of waste package fabrication processes. The resistance of Alloy 22 to localized corrosion decreased with increasing temperature and chloride concentration. Welding and thermal aging also decreased the localized corrosion resistance of the alloy. Results of these tests indicate that localized corrosion can be initiated on Alloy 22 in chloride-containing environments at temperatures lower than those at which a liquid film can be formed on the container surface by the presence of deliquescent salts.

### **INTRODUCTION**

One of the principal factors in the U.S. Department of Energy (DOE) Repository Safety Strategy for the proposed repository at Yucca Mountain (YM) is the long-term performance of the waste package (WP) [1]. It is expected that the integrity of the WPs, predicated on the use of a highly corrosion-resistant alloy as an outer container material, will prevent exposure of the radioactive waste (spent nuclear fuel and vitrified reprocessed waste) to the environment for times longer than the regulatory performance period of 10,000 years. During this period, the environment in contact with the WPs, initially hot dry-air and later humid air, may become an aggressive aqueous solution as a result of dripping of groundwater, altered in its chemical composition by interactions with the host rock and evaporative concentration due to radioactive decay heating.

The currently proposed DOE WP design consists of a 2-cm thick Alloy 22 (58Ni-22Cr-13Mo-3W-4Fe) outer container enclosing a 5-cm thick inner container made of type 316 nuclear grade (NG) stainless steel. The main purpose of the inner container is to provide structural strength to the WP. Alloy 22 became the material preferred by the DOE for the outer container because it is extremely resistant to localized corrosion. Nevertheless, Alloy 22 is susceptible to localized corrosion in the form of crevice corrosion within certain ranges of chloride concentration, redox potential, and temperature as are other Ni-Cr-Mo alloys previously

considered by the DOE, such as Alloys 825 and 625 [2, 3].

The objective of this paper is to provide a summary of the results of our experimental investigations on the localized (crevice) corrosion of Alloy 22. Crevice corrosion is one of the modes of corrosion that may significantly affect the performance of the containers in the repository. Penetration of the container can occur in a relatively short time interval once localized corrosion is initiated. Repassivation potential measurements were conducted to determine the minimum potential for the initiation and propagation of crevice corrosion. Information previously reported on the localized corrosion of Alloy 22 in aqueous environments relevant to the proposed Yucca Mountain repository [4–6] is updated with new experimental results.

## EXPERIMENTAL

Chemical compositions of the two heats (A and B) of Alloy 22 used in these measurements, as well as the composition of the Alloy 622 filler rod (C) used for welded specimens, are provided in Table I. The geometry and dimensions of the creviced specimens used in all the tests are reported elsewhere [2]. Flat specimens, machined from as-received (A: Heat 2277-8-3175) mill annealed plate, were fitted with two polytetrafluoroethylene (PTFE) crevice-forming washers. The welded specimens were machined from 12.7-mm-thick plate (B: Heat 2277-8-3235) and welded along the crevice area using the tungsten inert gas procedure with argon as the shielding gas. The weld was performed using a double V-notch as the joint geometry and Alloy 622 (C: Heat XX1045BG11) as the filler rod, using 8 passes per side (16 passes total). After welding, flat creviced specimens were machined from the welded plate.

**Table I.** Chemical composition (wt %) of heats of Alloy 22 (A and B) and Alloy 622 (C).

Heat	Ni	Cr	Mo	W	Fe	Co	Si	Mn	V	P	S	C
A	57.8	21.40	13.60	3.00	3.80	0.09	0.030	0.12	0.15	0.008	0.002	0.004
B	56.5	21.40	13.47	2.87	3.94	1.31	0.023	0.24	0.17	0.008	0.001	0.003
C	58.5	20.73	14.13	3.15	3.05	0.09	0.060	0.24	0.01	0.007	0.001	0.006

Flat specimens, machined from Heat A plate, were thermally treated at 870 °C over times ranging from 5 to 30 min to determine the effect of thermal aging on localized corrosion. Rather than the time at 870 °C, the treatment times indicate the total time during which the specimens were inside the furnace. This distinction is particularly important for treatment times shorter than 30 min because the time required for the specimens to reach 870 °C after placement in the furnace was determined to be about 2 min.

Tests were performed at temperatures ranging from 80 to 150 °C in nitrogen-deaerated solutions containing 0.1 mM to 4.0 M chloride, 1.24 mM  $\text{HCO}_3^-$ , 0.20 mM  $\text{SO}_4^{2-}$ , 0.16 mM to 2.0 M nitrate, and 0.1 mM  $\text{F}^-$ , with  $\text{Na}^+$  as the single cation. These anions are the predominant groundwater anionic species in the vicinity of the proposed repository site [7].

Cyclic potentiodynamic polarization (CPP) tests were conducted in PTFE-lined autoclaves to measure the repassivation potential for crevice corrosion ( $E_{\text{rrev}}$ ) using a potential scan rate of 0.167 mV/s, as described elsewhere [4, 5]. The autoclaves were used to conduct

measurements at temperatures above the boiling point of water or dilute solutions at ambient atmospheric pressure. Additional  $E_{\text{rcrev}}$  measurements were conducted in a glass cell using a modified test technique in which crevice corrosion was initiated at an applied potential lower than the potential for the onset of transpassive dissolution. The potential was then scanned back at a rate of 0.167 mV/s until repassivation occurred. By avoiding transpassive dissolution, the data obtained in the glass cells were found to be in agreement with values obtained in autoclaves because localized corrosion was consistently initiated. In previous measurements [3,5] in which the potential attained high values in the forward scan and transpassive dissolution occurred because only a maximum current limit was set in the CPP tests, localized corrosion in glass cells was not consistently observed under equivalent environmental conditions.

## RESULTS

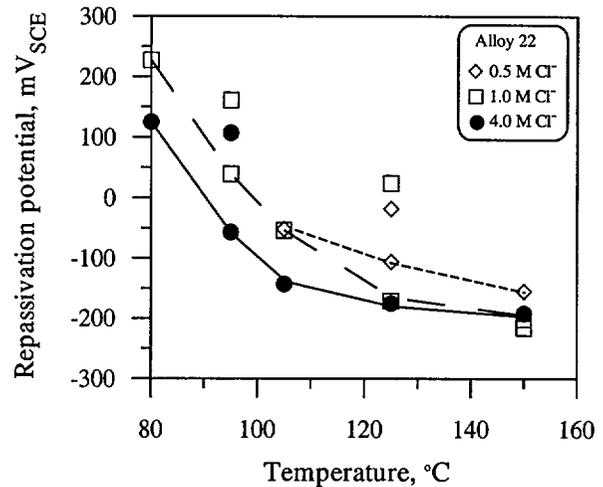
The minimum  $\text{Cl}^-$  concentration and the potential required for crevice corrosion to be initiated at a given temperature (e.g., 95 °C) are both higher for Alloy 22 than for the other alloys Ni-Cr-Mo alloys such as Alloys 825 and 625, as well as for type 316 L stainless steel [7,8]. Also, at a given  $\text{Cl}^-$  concentration, the lowest temperature at which crevice corrosion can occur is higher for Alloy 22 than for the other Ni-Cr-Mo alloys. A similar trend is observed in the values of  $E_{\text{rcrev}}$  for the various Ni-Cr-Mo alloys. A plot of  $E_{\text{rcrev}}$  for the mill annealed Alloy 22 as a function of temperature is shown in Figure 1 for a range of  $\text{Cl}^-$  concentrations [6]. A significant decrease in the  $E_{\text{rcrev}}$  with increasing temperature from 80 to 105 °C can be observed for the three  $\text{Cl}^-$  concentrations, followed by a leveling off at higher temperatures. It should be noted that no crevice corrosion was observed in the 0.5 M  $\text{Cl}^-$  solution at 80 and 95 °C. When the  $E_{\text{rcrev}}$  values (using only those that represent a lower bound) are replotted as a function of the logarithm of the  $\text{Cl}^-$  concentration as shown in Figure 2, a linear dependence is satisfied up to 150 °C can be observed for the three  $\text{Cl}^-$  concentrations, according to the following expression [6]

$$E_{\text{rcrev}} = E_{\text{rcrev}}^0(T) + B(T)\log[\text{Cl}^-] \quad (1)$$

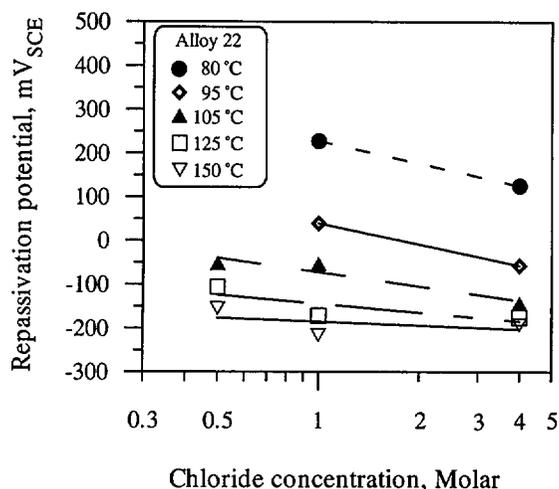
where both  $E_{\text{rcrev}}$  and  $B$  are parameters that, within the 80 to 105 °C range, can be expressed through a data regression as Eq. (2).

$$E_{\text{rcrev}}^0(T) = 1,300 - 13.1T; \quad B(T) = -362.7 + 2.3T \quad (2)$$

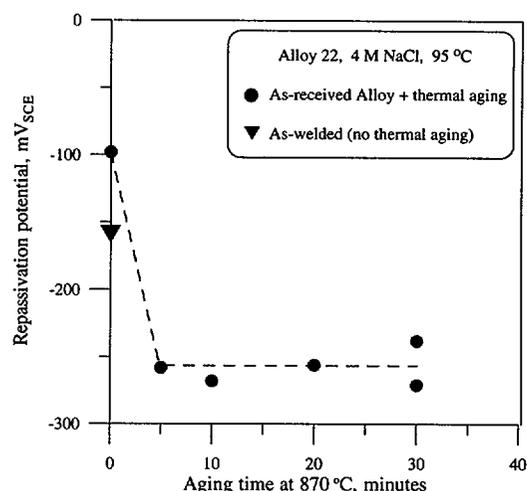
where  $T$  is temperature in °C and  $E_{\text{rcrev}}^0$  is in mV<sub>SCE</sub>. It is apparent from the value of the



**Figure 1.** Effect of temperature on the crevice corrosion repassivation potential for mill annealed Alloy 22 in  $\text{Cl}^-$  solutions.



**Figure 2.** Crevice corrosion repassivation potential for mill annealed Alloy 22 as a function of  $\text{Cl}^-$  concentration at various temperatures.



**Figure 3.** Effect of thermal aging time at 870 °C compared to mill-annealed and welded material on the crevice corrosion repassivation potential for Alloy 22 in 4 M  $\text{Cl}^-$  solution at 95 °C.

coefficients in Eq. (2) that  $E_{\text{rcrev}}$  is strongly dependent on temperature 100 °C and the dependence on  $\text{Cl}^-$  concentration decreases with increasing temperature.

The possible detrimental effects of fabrication processes on WP performance can be investigated by measuring the  $E_{\text{rcrev}}$  for both welded and thermally treated specimens and comparing the results to  $E_{\text{rcrev}}$  values for the mill annealed specimens. Because mill annealed Alloy 22 was found to be resistant to crevice corrosion at 95 °C in  $\text{Cl}^-$  solutions at concentrations equal to and lower than 0.5 M, a concentration of 4M  $\text{Cl}^-$  was used. Figure 3 shows the  $E_{\text{rcrev}}$  for both welded and thermally treated conditions in comparison with mill annealed Alloy 22 in 4M  $\text{Cl}^-$  solution at 95 °C. The  $E_{\text{rcrev}}$  for the welded material is approximately 60 mV lower than that of the mill annealed. The most pronounced effect on  $E_{\text{rcrev}}$ , however, is exhibited by the thermal aged material. Only 5 minutes of thermal treatment at 870 °C (note that the exposure time at that temperature is even shorter) are sufficient to induce a decrease of more than 150 mV in  $E_{\text{rcrev}}$ . As shown in Figure 3, no further decrease in  $E_{\text{rcrev}}$  is observed with thermal aging times up to 30 minutes. Results previously reported [5], indicated that the values of  $E_{\text{rcrev}}$  also ranged from -240 to -260 mV<sub>SCE</sub> for specimens thermally aged at 870 °C for 30 minutes to 24 hours.

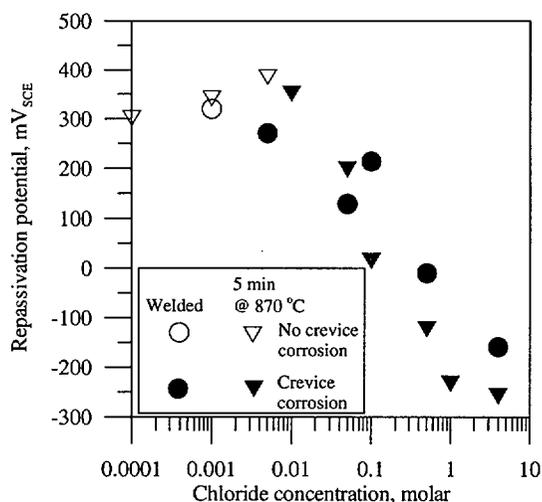
In contrast to the results obtained for the mill annealed material, localized corrosion of welded Alloy 22 was observed at a much lower critical  $\text{Cl}^-$  concentration. As shown in Figure 4, the substantial decrease of  $E_{\text{rcrev}}$  above 0.005 M  $\text{Cl}^-$  at 95 °C undoubtedly indicates that the welded material is significantly more susceptible to localized corrosion than the mill annealed alloy which exhibits a critical  $\text{Cl}^-$  concentration of about 0.5 M at the same temperature. Optical examination after testing revealed that preferential localized attack occurred in the weld and in the heat-affected zone (HAZ). Similar results as those for the welded specimens were obtained for as-received Alloy 22 after thermal aging for 5 min at 870 °C, as also shown in Figure 4. Post-test optical examination revealed the occurrence of intergranular corrosion in the creviced areas of the specimens. The morphology of the grain boundary attack in a specimen heat treated

at 870 °C for 5 min after a test conducted in 0.01 M  $\text{Cl}^-$  solution at 95 °C is shown in a scanning electron micrograph in Figure 5. In this case,  $E_{\text{rcrev}}$  was equal to 350 mV<sub>SCE</sub> but only 3 out of the 24 crevice sites in the specimen exhibited this severe grain boundary attack accompanied by some attack at twin boundaries. The number of intergranularly corroded crevice sites and the severity of the attack increased with increasing thermal treatment time. As can be noted for the specimens tested in 4M  $\text{Cl}^-$  solution at 95 °C, despite of the increase in the severity of the attack with increasing thermal treatment time, the value of  $E_{\text{rcrev}}$  as shown in Figure 3 was found to be practically constant, independent of the aging time.

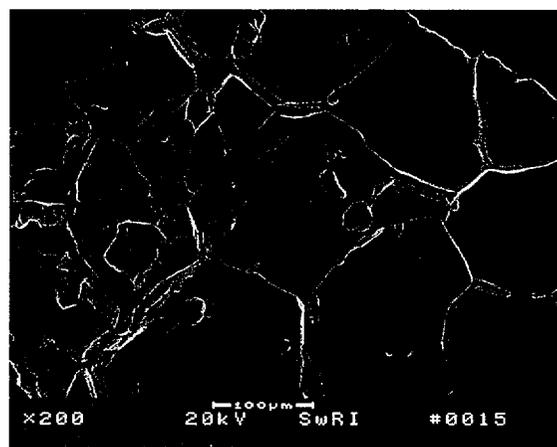
The chemistry of the waste package environment can have a significant effect on the localized corrosion resistance of the container material. Of particular interest is the possible role of  $\text{NO}_3^-$  as an inhibitor of crevice corrosion induced by  $\text{Cl}^-$ . The effect of the  $\text{NO}_3^-$  to  $\text{Cl}^-$  concentration ratio on the  $E_{\text{rcrev}}$  of welded Alloy 22 is shown in Figure 6. In pure 0.5 M  $\text{Cl}^-$  solution at 95 °C, the  $E_{\text{rcrev}}$  of welded Alloy 22 is 0 mV<sub>SCE</sub>. The  $E_{\text{rcrev}}$  of welded Alloy 22 is not significantly altered in 0.5 M  $\text{Cl}^-$  with the addition of 0.05 M  $\text{NO}_3^-$ . However, with  $\text{NO}_3^-$  concentrations equal to 0.1 M or greater, no localized corrosion was initiated in the short-term tests used for measuring  $E_{\text{rcrev}}$ . An additional test was conducted to determine the effect of  $\text{NO}_3^-$  on actively growing localized corrosion by initiating crevice corrosion in a solution containing only 0.5 M  $\text{Cl}^-$  and measuring the  $E_{\text{rcrev}}$  after the addition of  $\text{NO}_3^-$  to the solution. As shown in Figure 6, the addition of  $\text{NO}_3^-$  increased the  $E_{\text{rcrev}}$  of welded Alloy 22 to 250 mV<sub>SCE</sub>, indicating that  $\text{NO}_3^-$  has not only a significant effect on the inhibition of localized corrosion initiation but also promotes repassivation of an actively growing localized corrosion front.

## DISCUSSION

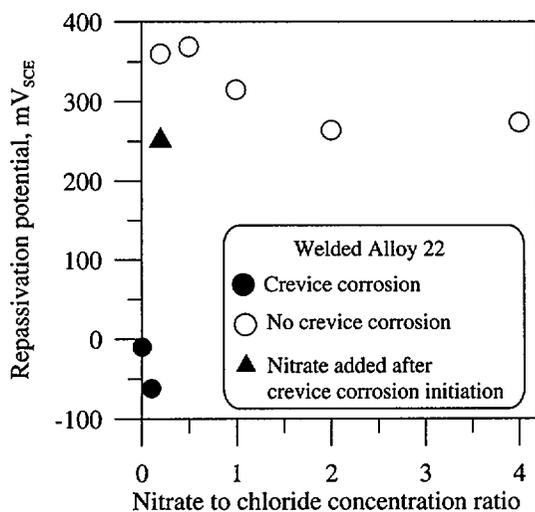
As reported previously, Alloy 22 in the as-received, mill annealed condition is extremely resistant to crevice corrosion over a wide range of  $\text{Cl}^-$  concentrations and temperatures. Crevice corrosion can only be expected at temperatures above 80 °C and  $\text{Cl}^-$  concentrations higher than



**Figure 4.** Crevice corrosion repassivation potentials for both welded and thermally aged Alloy 22 in  $\text{Cl}^-$  solutions at 95 °C.



**Figure 5.** Morphology of the intergranular attack in the crevice area of Alloy 22 heat treated at 870 °C for 5 min after a test conducted in 0.01 M  $\text{Cl}^-$  at 95 °C.



**Figure 6.** Crevice corrosion repassivation potential as a function of the  $\text{NO}_3^-$  to  $\text{Cl}^-$  ratio for welded Alloy 22 in 0.5 M  $\text{Cl}^-$  solution at 95 °C.

a temperature which is within the time/temperature domain in which topologically close-packed (TCP) phases precipitate, exhibits significantly lower  $E_{\text{rcrev}}$  values than the mill annealed material. It should be noted that only 5 minutes, or even less, of exposure to that temperature are sufficient to render the material susceptible to crevice corrosion at a substantially lower potential or at a much lower  $\text{Cl}^-$  concentration than that for the mill annealed material, as shown in Figure 4. Although the microstructure of the thermally treated specimens has not been characterized, TCP phases precipitated at grain boundaries seem to be the cause of the intergranular attack. Heubner et al. [10] reported the precipitation of TCP phases at grain boundaries after 15 minutes of treatment (the shortest time investigated) at temperatures ranging from 800 to 900 °C, but they did not detect intergranular corrosion using the ASTM G28A test. They only observed intergranular corrosion after longer aging times (> 1 hour).

The most plausible explanation for the intergranular attack in the crevice areas arises from the fact that TCP phases, such P,  $\sigma$ , and  $\mu$ -phases, are rich in Mo. As a result, incorporation of Mo in the TCP phases preferentially precipitated at grain boundaries induces the depletion of Mo in the matrix immediately around the precipitate. The occurrence of intergranular corrosion is, hence, facilitated because there is not sufficient Mo available to inhibit the initiation of the localized attack promoted by  $\text{Cl}^-$  anions in the occluded chemical environment of the crevice. Thus, the passivity range of the thermally treated alloy is reduced by the occurrence of intergranular corrosion inside crevices, which leads to significantly low values of  $E_{\text{rcrev}}$  with respect to that of the mill annealed material. Under certain fabrication or post-weld treatment conditions (e.g., slow cooling of thick sections) Alloy 22 could be exposed to temperatures around 800 to 900 °C over a time sufficient to induce precipitation of TCP phases at grain boundaries. As a result, intergranular corrosion of the alloy may occur in the presence of a crevice above a critical temperature and  $\text{Cl}^-$  concentration at relatively low potentials within the passive range.

The effect of welding on  $E_{\text{rcrev}}$  is comparable to that of thermal aging over a wide range

0.5 M if  $E_{\text{corr}}$  is higher than  $E_{\text{rcrev}}$ . Values of  $E_{\text{corr}}$  after 2-month exposure range from -190 to -140 mV<sub>SCE</sub> in air-saturated 0.028 M  $\text{Cl}^-$  solution (pH = 8.0) at 95 °C. Preoxidized specimens exhibit  $E_{\text{corr}}$  values approximately 100 mV higher. Within the temperature range of 80 to 105 °C  $E_{\text{rcrev}}$  exhibits a linear dependence on temperature and on the logarithm of  $\text{Cl}^-$  concentration similar to that found for other Cr-Ni-Mo alloys [2]. As noted before [6], the  $E_{\text{rcrev}}$  calculated by using Eq. (2) can be used as a lower bound for assessing the crevice corrosion susceptibility of the alloy in the mill annealed condition. This is the approach used in modeling the corrosion of Alloy 22 in the U.S. Nuclear Regulatory Commission/CNWRA Total-system Performance Assessment (TPA) code, as discussed by Dunn et al.[9].

The material thermally treated at 870 °C,

of  $\text{Cl}^-$  concentrations as shown in Figure 4. The detrimental effect appears to be more pronounced than that previously reported [5,6], even at 95 °C, because at  $\text{Cl}^-$  concentrations above 0.005 M a substantial decrease of  $E_{\text{rcrev}}$  is observed. At least a  $\text{Cl}^-$  concentration two orders of magnitude greater seems to be needed to have a similar effect on the mill annealed alloy. The preferential attack observed in the multipass welded region of the specimens, accompanied by intergranular corrosion in the HAZ could be attributed also to the precipitation of TCP phases. Cieslak et al. [11, 12] observed the presence of P-phase,  $\sigma$ -phase, and only traces of  $\mu$ -phase in the interdendritic regions of the as-welded microstructure, with concurrent enrichment in Mo and depletion in Ni in the precipitates. As in the case of the thermal aged specimens, depletion of Mo (and presumably W) confined to the regions around the precipitates promotes the localized attack even in relatively dilute  $\text{Cl}^-$  solutions ions when the feet of the crevice former are located on the weld.

One of the important observations in this study is the beneficial role of  $\text{NO}_3^-$  as inhibitor of crevice corrosion promoted by  $\text{Cl}^-$ . Kehler et al. [13] reported the beneficial  $\text{NO}_3^-$  plus  $\text{SO}_4^{2-}$  had on the stabilization and repassivation potentials measured in 5 M LiCl solution at 95 °C. However, in their study, conducted using Alloy 22 in the mill annealed condition, they covered a limited range of  $\text{NO}_3^- + \text{SO}_4^{2-}$  to  $\text{Cl}^-$  concentration ratios. As clearly shown in Figure 6,  $E_{\text{rcrev}}$  increased by more than 300 mV when the  $\text{NO}_3^-$  to  $\text{Cl}^-$  concentration ratio was increased just above 0.2 indicating the  $\text{NO}_3^-$  has a very strong inhibiting effect on the initiation of localized corrosion. It is also shown in Figure 6 that at about the same  $\text{NO}_3^-$  to  $\text{Cl}^-$  ratio,  $\text{NO}_3^-$  can arrest the propagation of a localized corrosion front. Although it is well established in the literature that  $\text{NO}_3^-$  is a strong inhibitor for the pitting corrosion of austenitic stainless steels in  $\text{Cl}^-$  solutions, higher  $\text{NO}_3^-$  to  $\text{Cl}^-$  ratios have been reported as needed to avoid pit nucleation [14]. There is no agreement in the literature regarding a mechanistic interpretation of the beneficial effect of  $\text{NO}_3^-$  as a localized corrosion inhibitor. In neutral solutions and at low concentrations,  $\text{NO}_3^-$  is a very mild oxidizing species, but it is generally considered a passivating anion as opposed to pitting promoter anions such as the halides. It is possible that its role as inhibitor can be explained in terms of competitive transport with  $\text{Cl}^-$  by electromigration and/or diffusion, thereby interfering in the build-up of high, local concentration of this aggressive anion. Saturated solutions of mixed salts (i.e. NaCl, NaNO<sub>3</sub> and KNO<sub>3</sub>) can be formed by absorption of water vapor at very low values of the relative humidity or at temperatures well above the boiling point of water [7, 15]. Therefore, concentrated solutions containing  $\text{NO}_3^-$  and  $\text{Cl}^-$  anions may exist in contact with the waste package over a wide range of temperatures. It will be necessary to determine the effect of the  $\text{NO}_3^-$  to  $\text{Cl}^-$  ratio on  $E_{\text{rcrev}}$  over a broader range of  $\text{Cl}^-$  concentrations and temperatures to reach a definitive conclusion about his beneficial role under the evolving environmental conditions in the repository.

## CONCLUSIONS

Alloy 22 in the as received mill annealed conditions can be susceptible to crevice corrosion in  $\text{Cl}^-$  containing solutions at  $\text{Cl}^-$  concentrations higher than 0.5 M and temperatures above 80 °C if  $E_{\text{corr}}$  is higher than  $E_{\text{rcrev}}$ . However,  $E_{\text{corr}}$  was found to be lower than  $E_{\text{rcrev}}$  in air-saturated solutions. Both welding and thermal treatment for a short time at temperatures at which precipitation of intermetallic Mo-rich phases occurs on Alloy 22 render the alloy susceptible to crevice corrosion in the form of severe interdendritic or grain boundary attack. The addition of  $\text{NO}_3^-$  to the  $\text{Cl}^-$  solutions even at a low concentration ratio inhibits crevice corrosion resulting in

a significant increase of  $E_{\text{crev}}$ . The beneficial effect of  $\text{NO}_3^-$  needs to be studied over a wide range of  $\text{Cl}^-$  concentrations and temperatures, as well as the combined effect of welding followed by thermal aging which may be more detrimental than the separate effect of these metallurgical processes.

## ACKNOWLEDGMENT

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