

**Effects of Surface Chromium Depletion on the Localized
Corrosion Alloy 825 High-Level Waste Container Material**

**EFFECTS OF SURFACE CHROMIUM DEPLETION ON
THE LOCALIZED CORROSION OF ALLOY 825
AS A HIGH-LEVEL NUCLEAR WASTE
CONTAINER MATERIAL**

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ABSTRACT

The effects of the chromium-depleted, mill-finished surface on the localized corrosion resistance of alloy 825 were investigated. Tests conducted in solutions based on the groundwater at Yucca Mountain, but with a higher concentration of chloride, indicated that the breakdown and repassivation potentials for the mill-finished surfaces were more active than those for polished surfaces. Potentiodynamic polarization tests indicated that pits can be initiated on the Cr-depleted surface at potentials of 220 mV_{SCE} in a solution containing 1,000 ppm chloride at 95 °C. Potentiostatic tests identified a similar pit initiation potential for the mill-finished surface. However, under long-term potentiostatic tests, a higher potential of 300 mV_{SCE} was needed to sustain stable pit growth beyond the Cr-depleted layer. An increase in the surface roughness was also observed to decrease the localized corrosion resistance of the material.

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INTRODUCTION

The high-level nuclear waste (HLW) packages intended for geological disposal are required by the U.S. Code of Federal Regulations (10 CFR Part 60) to provide substantially complete containment of the radionuclides for a period of at least 300 to 1,000 yr. One perceived threat to compliance with this regulation is the possibility of localized corrosion of the HLW containers. As a means of minimizing this possible mode of failure under the unsaturated conditions anticipated at the proposed repository at Yucca Mountain (YM), it has been suggested to increase the thermal load of the repository in order to create a dry-out area in the geologic surroundings adjacent to the containers for an extended period. However, previous studies have demonstrated that remote condensation of evaporated groundwater and backflow to the containers is possible (Patrick, 1986; Ramirez, 1991; Zimmerman et al., 1986). In addition, the concentration of aggressive anions such as Cl^- can increase with the number of evaporation cycles (Beavers et al., 1992). Therefore, the environment surrounding the waste packages may be expected to vary from hot and dry to conditions in which the waste containers become wetted by groundwater with varying concentrations of aggressive anions.

Recent studies undertaken to predict the service life of nuclear waste containers have proposed the use of the repassivation potential, E_{rp} , first suggested by Pourbaix et al. (1963) as a conservative lower bound parameter to predict long-term container performance (Sridhar et al., 1992, 1993; Sridhar and Cragolino, 1993; Tsujikawa and Hisamatsu, 1984; Tsujikawa and Kojima, 1991; Nakayama et al., 1993). These investigations have used carefully prepared specimens having a polished surface which removes any surface imperfections or near-surface alloy concentration variations. Many mill-finished surfaces on stainless steels (SSs) and Ni-base alloys have been shown to be depleted in chromium (Trax and Holzwarth, 1960; Sydberger, 1981; Grubb, 1991; Shone et al., 1988). For many applications, it is assumed that this layer is not detrimental to the overall performance of the material because it is often removed shortly after being placed in the service environment. Trax and Holzwarth were among the first investigators to identify the origin of the Cr-depleted layer on SS (Trax and Holzwarth, 1960). During the final anneal in a high speed continuous furnace in an oxidizing atmosphere, a Cr-rich oxide scale is formed on the metal surface. This leaves the underlying metal deficient in chromium. Subsequent mill processing such as blasting and pickling often remove only the oxide scale, and, as a result, a chromium-deficient layer is left on the material surface. For many Ni-base alloys with higher alloying additions, the etch rate of this depleted layer during the pickling operations is very low (Grubb, 1991). The resulting Cr-depleted, mill-finished surface layer has been shown to decrease the materials resistance to localized corrosion (Grubb, 1991; Shone et al., 1988). Therefore, previous studies with ideally prepared specimens may not accurately represent the surface of the waste package exposed to the repository environment.

The objective of this investigation was to determine the effect of the Cr-depleted, mill-finished surface of alloy 825 (UNS N08825) on the localized corrosion resistance of this proposed nuclear waste container material. Both potentiodynamic and potentiostatic tests were conducted on mill-finished surfaces to determine the breakdown and repassivation potentials of the Cr-depleted layer in a simulated repository environment.

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EXPERIMENTAL PROCEDURES

All experiments were conducted on Ni-based alloy 825 received in the form of a 12.5 mm plate with mill-finished surfaces. The bulk alloy and surface compositions are given in Table 1. Specimens having dimensions of 12.5×12.7×17.8 mm were cut from the plate. Care was taken to preserve the mill-finished surfaces. All other surfaces were polished to either a 60 or 600 grit finish using silicon carbide (SiC) paper. Prior to testing, the specimens were cleaned ultrasonically in detergent, rinsed in deionized (DI) water, ultrasonically cleaned in acetone, and dried. The weights of all specimens were then measured. Testing was carried out by first connecting the specimens to an ASTM G-5 (American Society for Testing and Materials, 1992) specimen holder and then partially immersing them to prevent exposing the gasket-specimen interface to the test solution. Test solutions representative of the groundwater at YM, but with a higher chloride concentration to simulate the effects of evaporation cycles, were prepared using analytical grade reagents and high-purity water. The solutions consisted of 1,000 ppm Cl⁻, 85 ppm HCO₃⁻, 20 ppm SO₄²⁻, 10 ppm NO₃⁻, and 2 ppm F⁻ all as Na salts. All tests were done at 95 ± 2 °C, which is just below the boiling point of water at YM. Solutions were deaerated with high-purity argon for the cyclic polarization tests or nitrogen for the potentiostatic tests.

Cyclic potentiodynamic polarization (CPP) experiments were conducted in an ASTM G-5-type five-neck flask equipped with a calibrated thermometer and a platinum counterelectrode. A Luggin probe with a porous silica tip placed 4–5 mm from the specimen was filled with 0.5 M NaCl to provide electrical contact to a remote saturated calomel reference electrode (SCE) maintained at room temperature. Argon was bubbled into the test solution at approximately one atmosphere pressure for a minimum of 1 hour prior to the start and throughout the duration of the tests. Electrochemical measurements were made with a computer-controlled potentiostat. The scan rate used in all CPP tests was 0.17 mV/s. Scans were started from a steady-state corrosion potential, E_{corr}, obtained 1 hour after immersing the specimens in solution. The scans on polished specimens were reversed when a current density of 5 mA/cm² was reached. Multiple scans, reversed at a current density of 1 mA/cm², were used to characterize Cr-depleted, mill-finished surfaces. The additional scans were carried out in fresh solution and started from the new measured value of E_{corr}. Specimens were examined after each scan. No additional scans were performed after pits were identified on the specimens.

Potentiostatic polarization tests were performed in an ASTM G-31 (American Society for Testing and Materials, 1992) one-liter reaction kettle (approximately 900 mL solution) fitted with a glass frit bubbler, a platinum counterelectrode, and a Luggin probe with a porous silica tip. The Luggin probe was filled with the test solution and attached to a water-cooled condenser. This ensured a reproducible thermal liquid junction potential and prevented deterioration of the SCE. Electrochemical measurements were made with a multichannel potentiostat controlled with a computer. Details of the experimental setup have been previously published (Sridhar and Cragolino, 1993). Following 1 hour of immersion, the E_{corr} was recorded and the specimen was polarized. Experiments were terminated after 7 days or after pitting of the specimens was apparent.

Following the completion of the experiments, the specimens were rinsed in DI water and ultrasonically cleaned in a mixture of 4 mL 2-Butyne-1,4-diol (35 percent aq. solution) + 3 mL concentrated HCl + 50 mL H₂O to remove the attached corrosion products (Tromans and Frederik, 1984). Tests on control samples indicated that this cleaning procedure does not result in measurable corrosion of the substrate. The specimens were then reweighed and examined using a 70 × stereoscope. Compositional analysis of the specimen surfaces was performed with a scanning electron microscope using energy dispersive spectroscopy (SEM-EDS).

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Table 1. Bulk and surface chemical composition of alloy 825 in wt. percent

Analysis	Fe	Ni	Cr	Ti	Cu	Mo	Si	Al
Bulk (vendor analysis)	30.4	41.1	22.1	0.8	1.8	3.2	0.2	0.1
Bulk (independent analysis)	28.1	41.8	23.0	0.9	1.8	3.6	0.1	0.1
Bulk (EDS)	29.0	42.0	22.7	0.9	1.5	3.2	0.5	0.2
Mill Surface (EDS)	43.4	35.3	15.7	0.4	1.6	2.1	0.9	0.5

The surface roughness of the mill-finished and polished surfaces was measured with a surface profilometer using a stylus with a 5 μm radius. The surface profiles obtained for the various samples were recorded with a computer and then converted to an ASCII format for analysis.

RESULTS

The results of single CPP scans of mill-finished and polished alloy 825 specimens are shown in Figure 1. It may be seen from this figure that the polished specimen had a breakdown potential, E_p , of 690 mV_{SCE} and a E_{tp} of 160 mV_{SCE} . A significant hysteresis indicative of pitting corrosion was also observed. Post-test observation with a 70 \times stereoscope confirmed the presence of pits. In contrast, the CPP scan for the mill-finished specimen had a E_p of 20 mV_{SCE} and a E_{tp} of -250 mV_{SCE} , significantly more active than the polished, bulk composition specimen. However, very little hysteresis was observed for the mill-finished specimen, and the post-test examination did not reveal any pits. Representative multiple CPP scans for this surface are displayed in Figure 2. These are CPP scans conducted successively, on a single specimen, with fresh solution being used for each scan. Once again, very low E_p and E_{tp} values were obtained in the first and second scans of Cr-depleted surfaces, but only uniform corrosion was observed. Subsequent scans, however, exhibited large hysteresis, indicating the occurrence of localized corrosion. Examination of the specimens using a 70 \times stereoscope revealed that the mill-finished surface pitted after 3 or 4 scans. On the other hand, the adjacent surfaces, having the bulk composition through grinding to either a 60 or 600 grit finish, did not show any evidence of pitting.

For the mill-finished surfaces, the E_{tp} was found to increase with cumulative charge density, or total amount of charge passed per unit area, contrary to previous results obtained for polished specimens (Sridhar et al., 1992). Analysis of the mill-finished specimens using SEM-EDS revealed that the chromium content at the surface increases with the amount of charge passed. Figure 3 plots the results of the CPP tests in terms of E_p and E_{tp} as a function of the chromium content measured on nonpitted regions at the conclusion of successive scans. This figure was generated using the results of both mill-finished surfaces having less than the bulk concentration of chromium and polished specimens having 22 percent chromium. The observed variation of E_p and E_{tp} for the polished surfaces in three tests was approximately 50 mV. Larger variations, observed with the mill-finished specimens are, most likely, the result of fluctuations in the surface composition and surface roughness of the Cr-depleted layer. However, it is evident from this plot that E_p and E_{tp} are strongly dependent on the concentration of chromium at the surface. Below a surface chromium concentration of 17 percent, uniform corrosion predominated.

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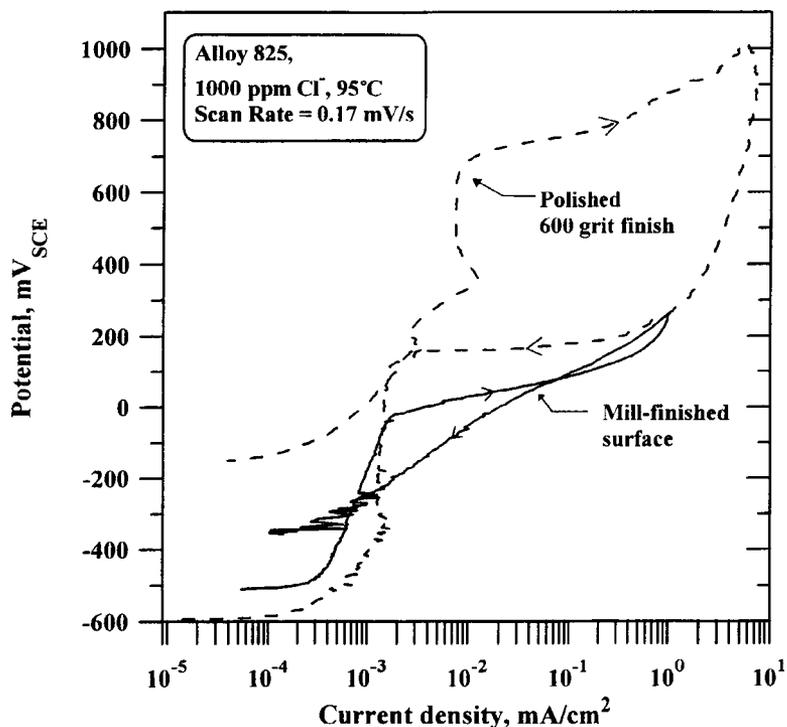


Figure 1. Cyclic potentiodynamic polarization scans for polished and mill-finished alloy 825

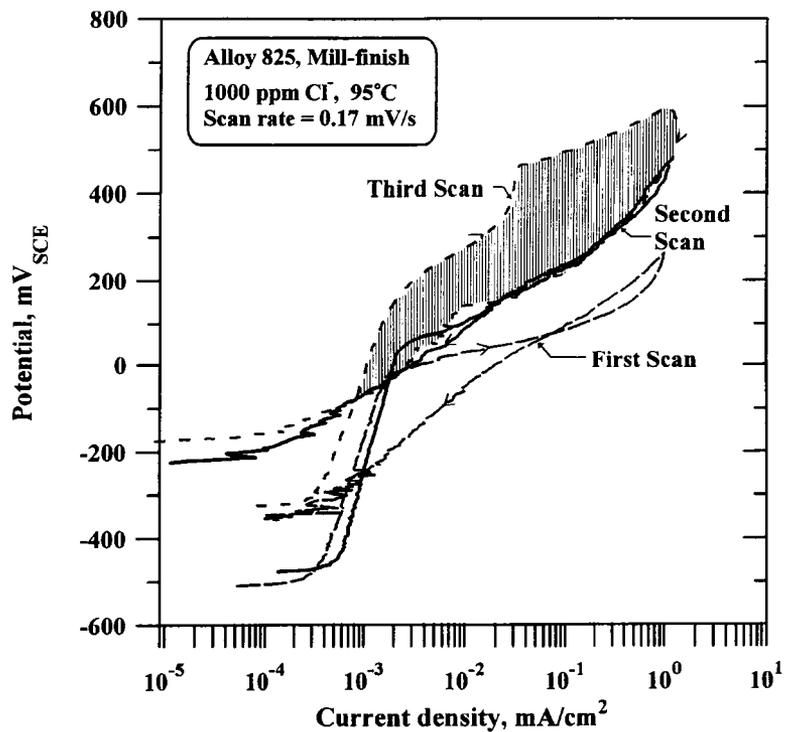


Figure 2. Successive cyclic potentiodynamic polarization scans for mill-finished alloy 825

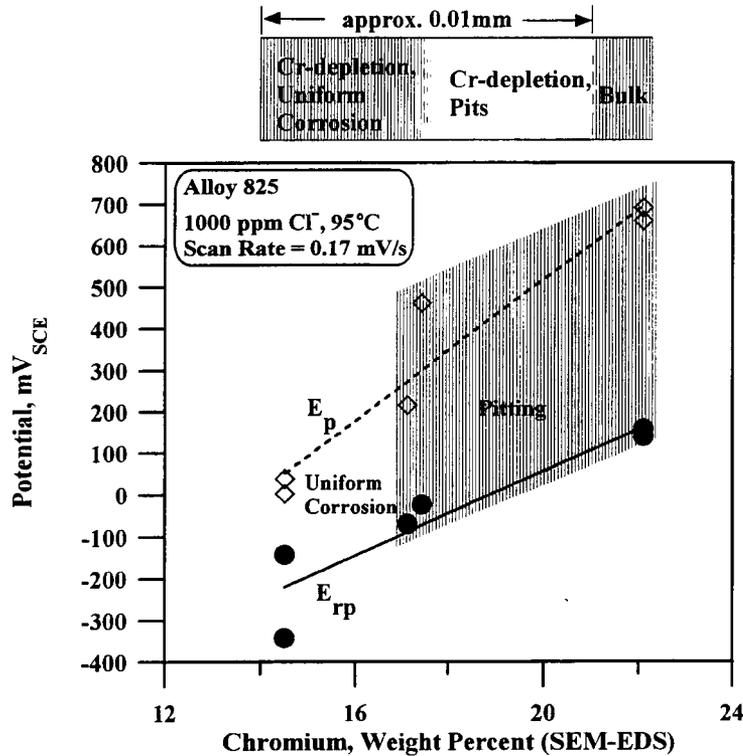


Figure 3. Breakdown and repassivation potential versus weight percent chromium

Localized corrosion occurred at surface chromium concentrations higher than 17 percent. Pitting of the Cr-depleted surfaces was initiated at potentials as low as 220 mV_{SCE}. In addition, the E_{rp} of these surfaces, following the initiation of pitting, was found to be much more active than that of the bulk material.

The results of potentiostatic tests on mill-finished specimens of alloy 825 are shown in Figures 4 and 5. Pits were initiated on specimens polished to a 600 grit finish at a potential of 600 mV_{SCE}. Below this potential, the alloy corrodes at a very low rate corresponding to the passive current density. This is shown in Figure 4 for the 600 grit specimen held at 500 mV_{SCE}. The absolute value of current density is plotted because at low current densities observed on this specimen there are brief bursts of cathodic current. In contrast, the mill-finished surface with a lower chromium content exhibits a very high corrosion rate at 450 mV_{SCE}. Post-test examination of the specimen revealed the presence of deep pits on the mill-finished surfaces. Adjacent surfaces polished to a 600 grit finish showed no indication of pitting. Figure 5 shows the results of additional potentiostatic tests on mill-finished specimens. These results indicate that pitting of the Cr-depleted surface is easily possible at potentials greater than 300 mV_{SCE}. Pits only occurred on the mill-finished surfaces, and no preferential attack at the solution/vapor interface was observed. At 275 mV_{SCE}, the specimen initially exhibited a high current density which then decreased to a value corresponding to the passive current density. Post-test examination of this specimen did not reveal any indication of pitting. The near-surface chromium concentrations measured by SEM-EDS are shown in Figure 6. For unpitted specimens, potentiostatically polarized at potentials of 200 mV_{SCE} to 275 mV_{SCE}, the concentration of chromium tends to increase with applied potential. Analysis of the specimen held at 300 mV_{SCE} in a region where pits were not present indicated the chromium concentration was 17 percent. Specimens polarized at higher potentials had lower

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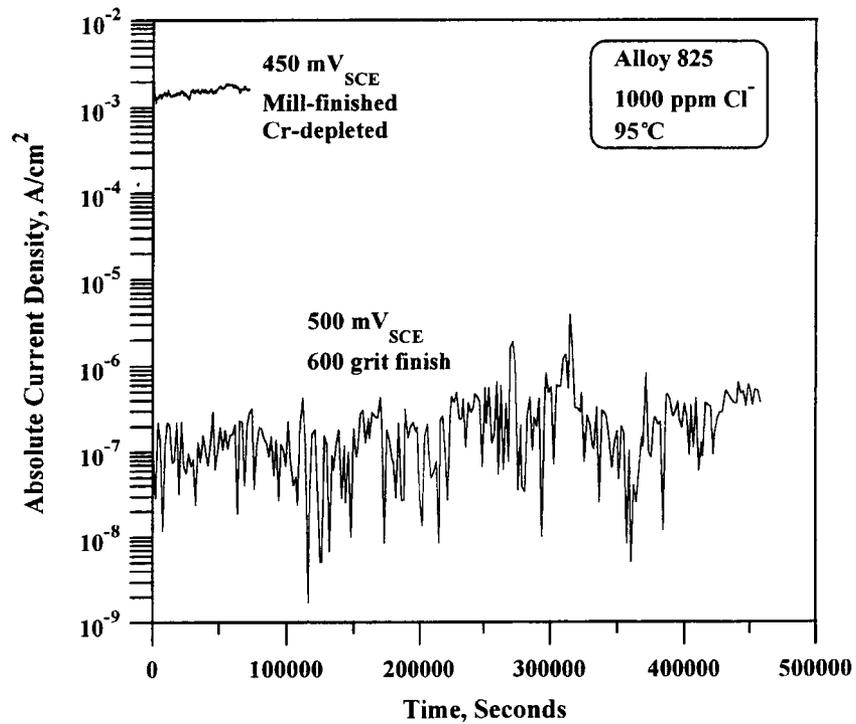


Figure 4. Potentiostatic polarization of Cr-depleted, mill-finished, and polished (600 grit finish) alloy 825

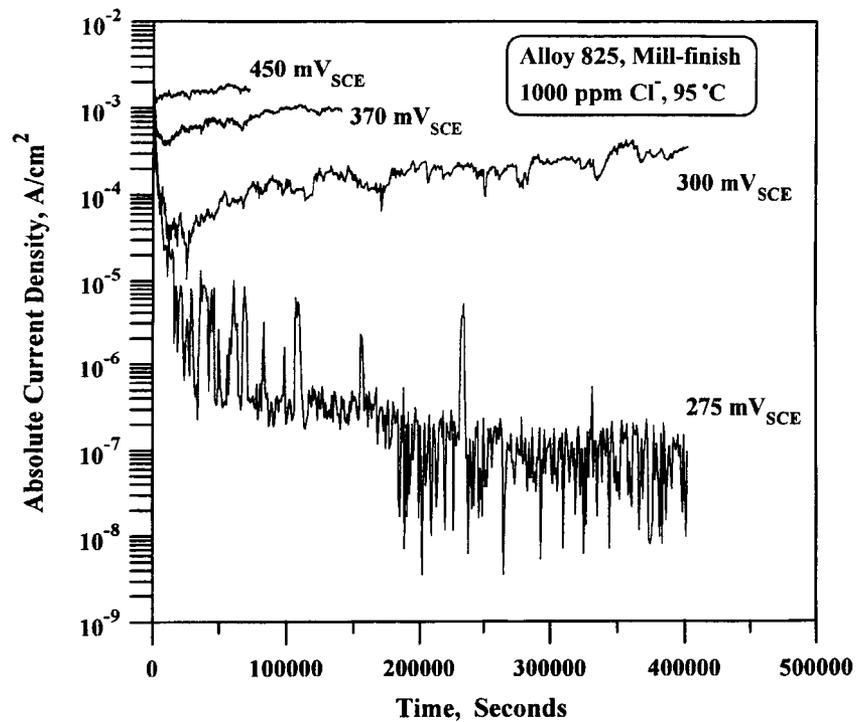


Figure 5. Potentiostatic polarization of mill-finished alloy 825 at several potentials

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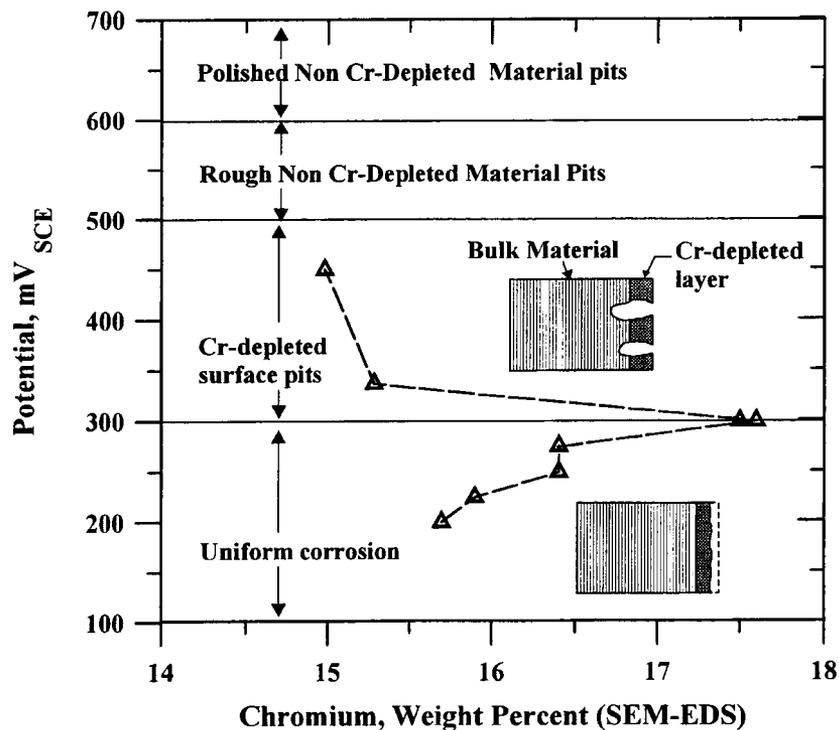


Figure 6. Weight percent chromium measured on unpitted regions of alloy 825 following potentiostatic polarization in 1,000 ppm Cl⁻ at 95 °C

concentrations of chromium at the surface, indicating the degree of uniform corrosion was low and that localized attack was quickly initiated leaving some areas of the original surface free from corrosion.

Potentiostatic tests were also carried out on specimens having the same surface roughness as the mill-finished specimens but without the Cr-depleted surface layer. The current density versus time plots (shown in Figure 7) indicate that at 500 mV_{SCE}, pits were initiated but a longer initiation time, compared to the mill-finished surfaces, was observed. The specimen held at 300 mV_{SCE} was observed to have a passive current density throughout the duration of the test, and no pits were observed during subsequent examination. The surface roughness profiles for the mill-finished, 36 grit, and 600 grit alloy 825 surfaces are shown in Figure 8.

DISCUSSION

The results of the CPP experiments comparing the mill-finished surface with the polished surface demonstrate the decreased corrosion resistance of the Cr-depleted surface layer. In addition to having a E_p and a E_{np} considerably more active than the bulk material, the passive current density of the mill-finished surface was found to be as high as 2.2 μA/cm², or 50 percent greater than that for the polished specimen. The mill-finished surfaces were observed to have E_{np} values that increased with charge density. This can be explained by the progression of the corrosion front through the Cr-depleted surface layer. The fact that several scans were needed to form pits indicates that uniform corrosion initially occurs on the Cr-depleted surface. After the initial surface layers are removed, the new surface composition has a higher concentration of chromium and becomes more resistant to corrosion. Pitting of

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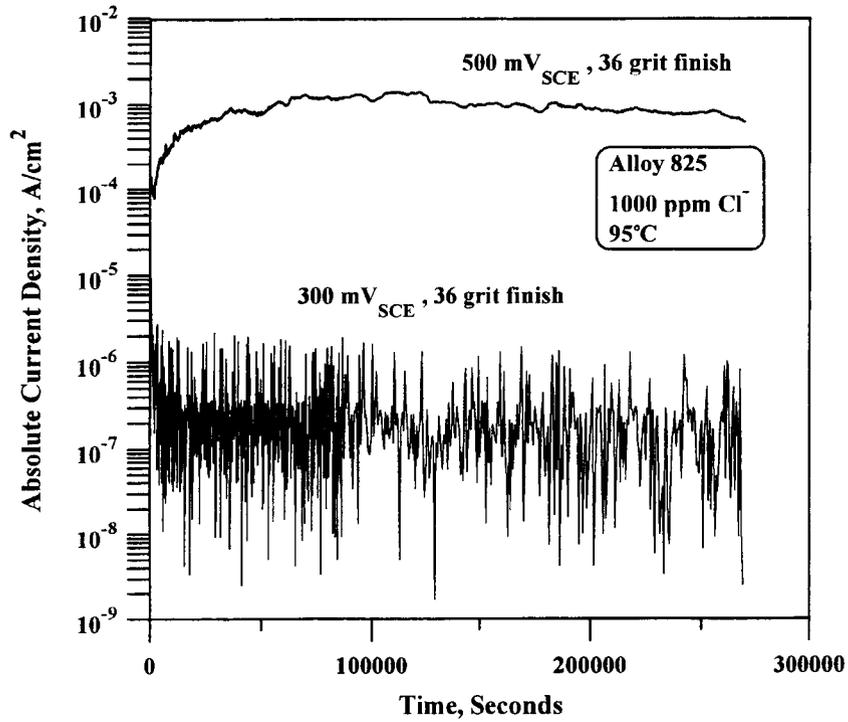


Figure 7. Potentiostatic polarization of bulk composition alloy 825 with a 36 grit finish

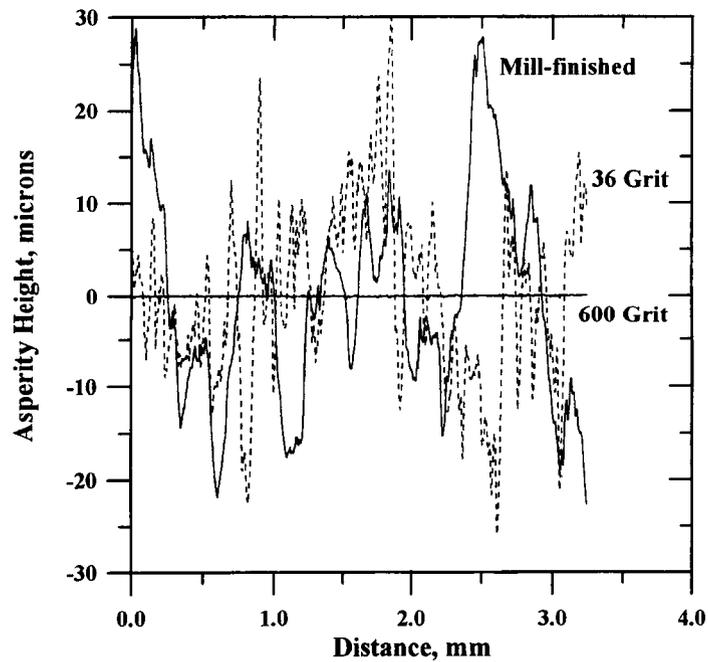


Figure 8. Surface roughness profiles of alloy 825 specimens

the surface can then occur at potentials more noble than the E_p for the initial surface layer, but much more active than the E_p for the bulk material. For many SS and Ni-base alloys, it is well known that an increased chromium concentration shifts the pitting potential to more noble values (Horvath and Uhlig, 1968). The E_{rp} for the pitted Cr-depleted surface was also considerably less than for the bulk material. Increasing the maximum current density attained prior to reversing the scan direction has been shown to result in lower repassivation potentials (Wilde, 1974; Wilde and Williams, 1972). This effect can be attributed to an increase in the charge density and, therefore, greater pit depths as the maximum current density is increased (Sridhar et al., 1993). In this investigation, the charge densities for polished specimens during CPP tests reversed at 5 mA/cm² were typically 23 to 25 coulombs/cm². For the mill-finished surfaces, the total charge density for multiple scans was always less than 10 coulombs/cm². Thus, the lower value of the repassivation potential for the mill-finished surfaces compared to the polished surfaces cannot be attributed to differences in test technique. The Cr-depleted layer on some Ni-based alloys has been measured to be 10-12 μ m deep (Grubb, 1991; Shone et al., 1988). Deeper pits, penetrating through the initial Cr-depleted surface layer, would be expected to have E_{rp} values similar to that of the bulk material.

Potentiostatic tests resulted in the mill-finished surfaces being severely pitted at potentials of 300 mV_{SCE} or greater. At 275 mV_{SCE}, the current density is initially high but then decreases to a passive value. This result indicates that at 275 mV_{SCE}, pits are initiated but then repassivate after penetrating into subsurface layers containing higher concentrations of chromium. This potential is higher than the E_{rp} measured for deep pits on polished specimens. However, pits which just penetrate the Cr-depleted layer are quite shallow. Previous results on polished specimens indicated that E_{rp} decreases rapidly with increasing pit depths and then attains a constant value for deep pits (Sridhar and Cragnolino, 1993). Since 275 mV_{SCE} is lower than the E_{rp} for very shallow pits, it is not surprising that at this potential, pits on Cr-depleted surfaces repassivated after initially growing partially through this layer. The potentiostatic test of the bulk composition surface prepared with 36 grit SiC paper indicates that the surface finish is also an important factor in the initiation of localized corrosion. Increasing the surface roughness of the bulk composition specimens resulted in a 100 mV decrease in the potentiostatic pit initiation potential. This result may be expected since increased surface roughness has previously been shown to decrease the corrosion resistance of a material by creating deep surface irregularities that may act as crevices or pit nuclei (Williams et al., 1983).

The SEM-EDS results for both CPP specimens and potentiostatic specimens indicate that a chromium concentration of 17 percent is necessary for the alloy to undergo localized corrosion. The CPP results have shown that at a surface composition of 17 percent chromium, pitting can be initiated at potentials as low as 220 mV_{SCE}. However, potentiostatic tests indicate that the mill-finished alloy 825 surface corrodes uniformly at potentials less than 300 mV_{SCE} even after a surface chromium concentration of 17 percent is reached. The differences in the results may be explained by observing that during the CPP tests, pits were initiated at potentials around 220 mV_{SCE}, but in the course of the scan, the potential increased to over 300 mV_{SCE}. Therefore, pits that were initiated at 220 mV_{SCE} were not allowed to repassivate due to an increasing applied potential even though they penetrated into regions containing greater amounts of chromium. For the potentiostatic tests, any pits initiated at potentials less than 300 mV_{SCE} repassivated after a very shallow penetration. The corrosion rate of the material in this case would be expected to be very low corresponding to the passive current density. At 300 mV_{SCE}, the material first undergoes uniform corrosion down to a subsurface layer containing 17 percent chromium. Pitting corrosion was then observed to penetrate deep into the specimen. Specimens held at potentials 370 mV_{SCE} and 450 mV_{SCE} were determined to have surface chromium compositions of 15.5 percent and 14 percent, respectively. This indicates that at progressively higher potentials the degree of uniform corrosion decreases, perhaps due to a shorter initiation time for pitting attack.

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Previous investigations by the authors have shown that in similar solutions the E_{corr} for alloy 825 depends on both the redox potential of the environment and the thermal history of the oxide film (Dunn et al., 1993). In deaerated environments, the E_{corr} was typically $-550 \text{ mV}_{\text{SCE}}$, which is much lower than the E_{p} . The addition of dissolved oxygen, by bubbling air into the solution, increased the E_{corr} by 300 mV. In solutions with 0.5 mM hydrogen peroxide (H_2O_2), added to simulate the effects of radiolysis, the E_{corr} of the mill-finished surface was as high as $20 \text{ mV}_{\text{SCE}}$. Specimens with polished surfaces were consistently observed to have a higher E_{corr} . However, it should be noted that the long-term time-dependent changes in the E_{corr} have not yet been fully investigated.

CONCLUSIONS

- The breakdown potential of Cr-depleted, mill-finished alloy 825 is strongly dependent on the surface composition of the material. Pits can be initiated on the mill-finished alloy 825 surface at potentials as low as $220 \text{ mV}_{\text{SCE}}$. A potential of $300 \text{ mV}_{\text{SCE}}$ is required for the pits to penetrate through the Cr-depleted layer into the bulk material.
- The pit initiation potential for bulk composition specimens was observed to decrease with an increase in the surface roughness of the material. Potentiostatic tests indicated that the pit initiation potential decreased from $600 \text{ mV}_{\text{SCE}}$ for specimens polished to a 600 grit finish, to $500 \text{ mV}_{\text{SCE}}$ for specimens with a 36 grit finish.
- Shallow pits in the mill-finished surface were determined to have a repassivation potential dependent on the chromium composition of the Cr-depleted layer. As pits propagate into the mill-finished layer, the repassivation potential increases as the chromium concentration increases. Pits penetrating through the Cr-depleted layer can be expected to have a repassivation potential similar to that for deep pits on polished bulk composition specimens.
- Long-term prediction of the localized corrosion resistance of alloy 825 HLW containers using the pit initiation potential for polished surfaces may not be representative of actual repository conditions. The presence of a mill-finished surface, or unintentional abrasion of the waste package during handling, can drastically alter the localized corrosion resistance of the material. The repassivation potential for deep pits, which is unaffected by surface imperfections, is a more conservative parameter for lifetime prediction.

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