**EFFECTS OF HIGH TEMPERATURE SURFACE OXIDES
ON ROOM TEMPERATURE AQUEOUS CORROSION
AND ENVIRONMENTAL EMBRITTLEMENT OF IRON ALUMINIDES**

September, 1996

Report Prepared by

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EFFECTS OF HIGH TEMPERATURE SURFACE OXIDES ON ROOM TEMPERATURE
AQUEOUS CORROSION AND ENVIRONMENTAL EMBRITTLEMENT
OF IRON ALUMINIDES*

R. A. Buchanan and R. L. Perrin

ABSTRACT

Studies were conducted to determine the effects of high-temperature surface oxides, produced during thermomechanical processing, heat treatment (750 °C in air, one hour) or simulated in-service-type oxidation (1000 °C in air, 24 hours) on the room-temperature aqueous-corrosion and environmental-embrittlement characteristics of iron aluminides. Materials evaluated included the Fe₃Al-based iron aluminides, FA-84, FA-129, FAL and FAL-Mo, a FeAl-based iron aluminide, FA-385, and a disordered low-aluminum Fe-Al alloy, FAPY. Tests were performed in a mild acid-chloride solution to simulate aggressive atmospheric corrosion.

Cyclic-anodic-polarization tests were employed to evaluate resistances to localized aqueous corrosion. The high-temperature oxide surfaces consistently produced detrimental results relative to mechanically or chemically cleaned surfaces. Specifically, the pitting corrosion resistances were much lower for the as-processed and 750 °C surfaces, relative to the cleaned surfaces, for FA-84, FA-129, FAL-Mo, FA-385 and FAPY. Furthermore, the pitting corrosion resistances were much lower for the 1000 °C surfaces, relative to cleaned surfaces, for FA-129, FAL and FAL-Mo.

U-bend and slow-strain-rate stress-corrosion-cracking tests were employed to evaluate environmental embrittlement characteristics. In this case, the high-temperature oxide surfaces consistently produced beneficial results relative to mechanically cleaned surfaces. Specifically, in 200-hour U-bend tests of FA-84, FA-129, FAL-Mo and FAPY, specimens with 750 °C oxide surfaces demonstrated lower tendencies for cracking than specimens with cleaned surfaces. Furthermore, slow-strain-rate tests of FA-129, FAL and FAL-Mo at free-corrosion and cathodic-hydrogen-charging potentials showed consistently higher ductilities for 1000 °C oxide surfaces as compared to cleaned surfaces.

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INTRODUCTION

The overall objective of this project was to study the effects of retained high-temperature surface oxides on the aqueous corrosion and environmental embrittlement characteristics of several Fe_3Al -based iron aluminides, an FeAl-based iron aluminide, and a low-aluminum Fe-Al alloy. Previous aqueous-corrosion studies of these materials by the present authors and colleagues have examined specimens only with cleaned surfaces (i.e., mechanically ground or polished surfaces).¹⁻⁴ The high-temperature surface oxides of the present study are not likely to have the same chemistry nor the same morphology (e.g., thermal cracking) as the passive films formed naturally on a clean surface in an aqueous environment. It was hypothesized that the presence of the high-temperature oxides could modify both the resistance to localized corrosion and the resistance to environmental embrittlement. To address these issues, the project involved cyclic-anodic-polarization testing to evaluate aqueous-corrosion behavior and either U-bend or slow-strain-rate testing in corrosive environments to evaluate environmental-embrittlement behavior.

The project consisted of two stages. Stage I involved characterizations of the corrosion and embrittlement effects of high-temperature surface oxides produced by thermomechanical processing (as-processed surfaces) and heat treatment in air at 750 °C for one hour (750 °C surfaces). In Stage II, the focus changed from surfaces produced by processing and heat treatment to surfaces produced by simulated in-service-type oxidation, accomplished by exposure in air at 1000 °C for 24 hours (1000 °C surfaces). In this report, the experimental procedures, results and conclusions will be given separately for Stages I and II of the project, then a final section summarizing the overall results and conclusions will be given.

The principal electrolyte employed in this study was a mild acid-chloride solution designed to simulate aggressive atmospheric corrosion. In this solution, the alloys evaluated are susceptible, to varying degrees, to pitting corrosion. An excellent method for evaluating the relative pitting corrosion susceptibility involves producing and analyzing cyclic anodic polarization curves. Since the reader may not be familiar with this electrochemical method, a brief review will be provided. A schematic illustration of a cyclic anodic polarization curve is shown in Figure 1, where certain electrochemical parameters are identified.

Fig. 1. Schematic illustration of cyclic anodic polarization behavior.

To produce the curve, the corrosion sample first is allowed to stabilize in the electrolyte such that a steady-state, open-circuit, corrosion potential, E_{corr} , is established. Next, with a potentiostatic circuit, the potential of the sample, E , is forced in the positive direction at a given rate while the current density, i , is continuously measured. After the potential has moved away from E_{corr} by a relatively small amount (≈ 50 mV), the current density is directly proportional to the corrosion rate. The potential scan is continued in the positive direction until a predetermined reversing current density, i_r , is reached. At this point, the potential scan direction is reversed, moving in the negative direction, and continued until a very low current density is achieved. In Figure 1, the nearly vertical region in the up-scan portion of the curve, characterized by an average passive current density, i_p , is reflective of passivation, where the material has developed a thin protective oxide/hydroxide passive film, and, therefore, corrodes at a very low rate over a range of potentials. At the critical pitting potential, E_{pit} , pitting corrosion initiates, with a sharp increase in current density. The pitting corrosion accelerates to i_r , and continues during the down-scan portion of the curve until the protection (or repassivation) potential, E_p , is reached (which corresponds to the cross-over point for the up-scan and down-scan curves). With reference to Figure 1, two of the parameters often used to characterize relative resistance to pitting corrosion are: (1) the magnitude of E_{pit} , with higher values corresponding to greater pitting resistance, and (2) the difference between E_{pit} and E_{corr} , i.e., $(E_{\text{pit}} - E_{\text{corr}})$, with higher values corresponding to greater pitting resistance.

STAGE I PROCEDURES: AS-PROCESSED AND 750 °C SURFACES

The materials studied were: three Fe₃Al-based iron aluminides, FA-84, FA-129 and FAL-Mo; a disordered low-aluminum Fe-Al alloy, FAPY; and an FeAl-based iron aluminide, FA-385. The chemical compositions are given in Table 1. The FA-84, FA-129, FAL-Mo and FAPY materials, in as-processed sheet form, were kindly supplied by Dr. Vinod Sikka of the Oak Ridge National Laboratory (ORNL); the FA-385 material, in rod form with a cladding, was kindly supplied by Dr. Philip Maziasz of ORNL.

Table 1: Chemical compositions of Stage I materials (atomic percent).

	Fe	Al	Cr	Mo	B	Zr	Nb	C	Y
FA-84	69.9	28.0	2.0	-	0.05	-	-	-	-
FA-129	66.3	28.0	5.0	-	-	-	0.5	0.2	-
FAL-Mo	65.9	28.0	5.0	1.0	0.04	0.08	-	-	-
FAPY	77.2	16.1	5.4	1.1	-	0.1	-	-	0.06
FA-385	63.9	36.7	-	0.2	-	0.05	-	0.11	-

For the FA-84, FA-129, FAL-Mo and FAPY materials, the surface conditions evaluated were: (1) as-processed, (2) 750 °C oxide, (3) mechanically cleaned, and (4) chemically cleaned. The thermomechanical processing procedures (accomplished at ORNL) for the as-processed condition were: hot forged 50% at 1000 °C, hot rolled 50% at 800 °C, and warm rolled 70% at 650 °C to a finish thickness of 0.75 mm. The heat treatment procedure for the 750 °C oxide surface condition consisted of annealing mechanically-cleaned as-processed specimens for one hour at 750 °C in air, followed by oil quenching. The mechanically cleaned condition was accomplished by grinding the as-processed materials through 600-grit SiC to remove all high-temperature oxides. The chemically cleaned condition was accomplished by immersing the as-processed materials in an acid solution until all high-temperature oxides were removed (approximately 30 minutes), then immersing in 10% HNO₃ at 43 °C for one hour as a passivation treatment. The acid solutions varied with the material, as follows: FA-84, 10% HNO₃ at 43 °C; FA-129, 10% HNO₃ plus 2% HF at room temperature; FAL-Mo, 10% HNO₃ plus 2% HF at 43 °C; and FAPY, no HNO₃/HF/temperature combination was found to satisfactorily remove the oxide and produce a smooth surface.

Because of the form of the as-received FA-385 material (rods with protective claddings), it did not have the as-processed high-temperature oxides. To create the 750 °C oxide surface condition, the FA-385 samples were annealed in air at 750 °C for one hour, then air cooled. The mechanically-cleaned condition was accomplished in the same manner as described for the other materials.

Cyclic anodic polarization tests were performed in a mild acid-chloride solution (pH = 4 (H₂SO₄), 200 ppm Cl⁻ (NaCl)). A standard polarization cell and an EG&G Model 273 potentiostat were employed. Each sample was mounted in epoxy, attached and electrically connected to a sample holder, and placed in a polarization cell containing the mild acid-chloride solution. Stabilization was allowed to occur for at least one hour, after which E_{corr} was measured. The cyclic polarization curve was then generated at a scan rate of 600 mV/h. The reversing current density was 1000 $\mu\text{A}/\text{cm}^2$.

U-bend stress corrosion cracking tests were performed on the above alloys (except FA-385). Sheet material was cut into 1 cm by 11.9 cm strips and then the thermomechanical oxide was mechanically removed (600-grit SiC). The strips were heat treated in air at 750°C for one hour and then quenched in oil. Half of the strips were mechanically cleaned once again to remove the furnace oxide. The strips were bent to a radius of 15.9 mm, thus forming the U-bend specimen geometry illustrated in Figure 2. It is noted that in the as-processed condition, the materials were generally too brittle to be deformed into the U-bend geometry. Each specimen was fastened into the U-bend shape by means of a polyvinyl chloride bolt so as to eliminate galvanic corrosion effects. The total strain in the bend was determined by the relation (ASTM G 30), $e = T / 2R$, where T is the specimen thickness and R the bend radius. The total strain placed on the U-bend specimens ranged from 0.024 to 0.037 cm/cm. Due to an inability to reliably produce chemically-cleaned surfaces for all alloys, this surface condition was not evaluated. Testing of the U-bend specimens was conducted in the same mild acid-chloride solution employed in the polarization tests.

One series of U-bend tests was conducted at the open-circuit corrosion potential, E_{corr} , which was monitored continuously for 200 hours. A second series of tests was conducted at a hydrogen-charging potential of -1500 mV(SHE) to enhance the production of hydrogen at the specimen surfaces. In this series of tests, the cathodic current density was monitored continuously.

Fig. 2: Schematic geometry of U-bend test specimen.

STAGE I RESULTS: AS-PROCESSED AND 750 °C SURFACES

Aqueous Corrosion

The cyclic anodic polarization behaviors of FA-84 (28Al-2Cr) in the as-processed, 750 °C oxide, mechanically cleaned, and chemically cleaned surface conditions are shown in Figure 3. The as-processed and 750 °C oxide conditions, relative to the mechanically cleaned and chemically cleaned conditions, produced lower pitting potentials (E_{pit}) and lower values of ($E_{\text{pit}} - E_{\text{corr}}$). Both of these results indicated lower resistance to pitting corrosion for the as-processed and 750 °C oxide surface conditions. The 750 °C oxide surface condition showed essentially no ability to undergo passivation. The very low passive current density, i_p , for the chemically cleaned surface suggested that the chemical cleaning process enhanced the properties of the passive film.

Similar results were obtained for FA-129 (28Al-5Cr), as seen in Figure 4. The as-processed and 750 °C oxide conditions, relative to the mechanically cleaned and chemically cleaned conditions, produced lower pitting potentials (E_{pit}) and lower values of ($E_{\text{pit}} - E_{\text{corr}}$), thus indicating lower resistance to pitting corrosion for the high-temperature oxide surfaces. The 750 °C oxide surface again showed essentially no ability to undergo passivation.

As seen in Figure 5 for FAL-Mo (28Al-5Cr-1Mo), E_{pit} and ($E_{\text{pit}} - E_{\text{corr}}$) again were lower for the as-processed and 750 °C oxide surface conditions relative to the mechanically and chemically cleaned surface conditions. Again, these results indicate lower pitting corrosion resistance for the high-temperature surface oxide conditions. It also is noted that the high-temperature oxide conditions did not produce stable values for the passive current densities, i_p , i.e., current-density fluctuations are obvious. The lower i_p value for the chemically cleaned condition suggested that there was either a thickening of the passive film or possibly a Cr enrichment of the passive film caused by the chemical cleaning process.

The cyclic anodic polarization behaviors of the disordered alloy, FAPY (16Al-5Cr-1Mo), are shown in Figure 6. Once again, the as-processed and 750 °C oxide surfaces performed poorly in comparison to the mechanically and chemically cleaned surfaces. The lower E_{pit} and $(E_{\text{pit}} - E_{\text{corr}})$ values for the high-temperature oxide surfaces indicated reduced resistance to pitting corrosion. Indeed, the as-processed and 750 °C oxide surfaces showed little indication of passivation.

As seen in Figure 7 for the FeAl-based iron aluminide, FA-385 (36Al-0.2Mo), the 750 °C oxide surface produced lower E_{pit} and $(E_{\text{pit}} - E_{\text{corr}})$ values, indicating lower resistance to pitting corrosion. On the beneficial side, however, the 750 °C oxide surface produced a considerably lower passive current density, i_p , although the passive current density was unstable.

Fig. 3. Cyclic anodic polarization behaviors of FA-84 with as-processed, 750 °C oxide, mechanically-cleaned, and chemically-cleaned surfaces.

Fig 4. Cyclic anodic polarization behaviors of FA-129 with as-processed, 750 °C oxide, mechanically-cleaned, and chemically-cleaned surfaces.

Fig 5. Cyclic anodic polarization behaviors of FAL-Mo with as-processed, 750 °C oxide, mechanically-cleaned, and chemically-cleaned surfaces.

Fig 6. Cyclic anodic polarization behaviors of FAPY with as-processed, 750 °C oxide, mechanically-cleaned, and chemically-cleaned surfaces.

Fig 7. Cyclic anodic polarization behaviors of FA-385 with 750 °C oxide and mechanically-cleaned surfaces.

The overall polarization results are summarized in Figure 8, where the resistance to localized corrosion in the mild acid chloride solution (in terms of the measured parameter ($E_{pit} - E_{corr}$)) is shown for all materials and all surface conditions. It is apparent that the mechanically and chemically cleaned surfaces for all materials demonstrated superior localized corrosion resistance relative to the high-temperature oxide surfaces of the as-processed and 750 °C heat treatment conditions.

Fig. 8. Effects of as-processed, 750 °C oxide, mechanically cleaned, and chemically cleaned surfaces on localized corrosion resistance in the mild acid chloride solution.

An observation that did not manifest itself directly in the polarization behavior was the appearance of the surfaces of the samples after chemical cleaning. The high-temperature as-processed oxide surfaces of FA-84 and FA-129 were cleaned rather easily in the 10% HNO_3 , 2% HF solution but also could be cleaned in a solution containing only 10% HNO_3 .

However, the oxide surfaces of FAL-Mo and FAPY were slow to clean in the 10% HNO₃, 2% HF solution, and could not be cleaned at all in the 10% HNO₃ solution. Furthermore, after chemical cleaning, the FA-129 and FA-84 samples had shiny smooth surfaces, whereas the FAL-Mo and FAPY samples had shiny rough surfaces. For the FAL-Mo and FAPY samples, the attack of the 10% HNO₃, 2% HF solution was more prominent in areas of exposed base material, i.e., cut faces or where the oxide had flaked off. The difficulties experienced in removal of the high-temperature oxides apparently can be attributed to the presence of molybdenum in the FAL-Mo and FAPY alloys, which also is partly responsible for the greater aqueous-corrosion resistance exhibited by these alloys.³

Environmental Embrittlement

With regard to the 200-hour U-bend stress-corrosion-cracking tests, the overall results in terms of cracking behavior are shown in Table 2. For the tests conducted at the corrosion potential, E_{corr} , none of the 750 ° oxide nor mechanically cleaned U-bend specimens underwent cracking. For the tests conducted at the hydrogen-charging potential, -1500 mV(SHE), none of the 750 ° oxide specimens underwent cracking. However, mechanically cleaned specimens of FAL-Mo, FA-129, and FA-84 did undergo cracking. These results indicate that under severe hydrogen-producing conditions, the presence of the 750 °C heat treatment oxides are beneficial in improving the resistance of the materials to environmental embrittlement.

Table 2. 200-hour U-bend-test results for 750 °C oxide and mechanically cleaned surfaces.

Alloy	Corrosion Potential, E_{corr}		Hydrogen-Charging Potential, -1500 mV SHE	
	750 °C Oxide Surfaces	Mechanically Cleaned Surfaces	750 °C Oxide Surfaces	Mechanically Cleaned Surfaces
FA-84	No Cracking	No Cracking	No Cracking	Cracked (~190 h)
FA-129	No Cracking	No Cracking	No Cracking	Cracked (~23 h)
FAL-Mo	No Cracking	No Cracking	No Cracking	Cracked (~190 h)
FAPY	No Cracking	No Cracking	No Cracking	No Cracking

For the U-bend tests conducted at the hydrogen-charging potential, the resultant cathodic current densities for the alloys in the two surface conditions are shown in Table 3 for time periods of 20 and 200 hours. The current densities are directly proportional to the electrochemical reduction rate at the specimen surfaces. At this very negative cathodic potential, the dominant reduction reactions are the reduction of hydrogen ions and water to hydrogen atoms which, with time, combine to form hydrogen molecules, then hydrogen vapor bubbles, i.e., $2\text{H}^+ + 2\text{e} \rightarrow 2\text{H}(\text{H}_2)$ and $2\text{H}_2\text{O} + 2\text{e} \rightarrow 2\text{H}(\text{H}_2) + 2\text{OH}^-$. It is the diffusion of atomic hydrogen (H) into the metal substrate which, of course, can cause hydrogen embrittlement. Another possible reduction reaction, which could contribute to the measured cathodic current densities, especially for the high-temperature oxide surfaces, is the reduction of the oxides to metallic form. Thermodynamically, the iron-oxide constituents should undergo reduction at the -1500 mV(SHE) potential. It was anticipated that the cathodic current densities for the oxide specimens would be considerably lower than those for the mechanically cleaned specimens, simply because of the higher electrical resistivity of the oxide (i.e., greater resistance to electron flow). However, as seen in Table 3, the current densities for the 750 °C oxide surfaces and the mechanically cleaned surfaces were, generally, quite similar. The relatively high current densities for the 750 °C oxide specimens could have been due to cracked/spalled oxide regions exposing the metal substrate on the strained U-bend specimens, to the porous or non-adherent nature of the oxides, or to a contribution associated with the electrochemical reduction of the oxides. If the reduction of the oxides was a significant factor for the 750 °C oxide surfaces, and since roughly comparable current densities were observed for the 750 °C oxide and mechanically cleaned specimens, the implication is that less hydrogen was being produced on the 750 °C oxide surfaces. This possibility would explain why the 750 °C oxide specimens did not undergo cracking at the hydrogen-charging potential whereas several of the mechanically cleaned specimens did undergo cracking.

Table 3. Cathodic current densities ($\mu\text{A}/\text{cm}^2$) during hydrogen charging for 750 °C oxide and mechanically cleaned surfaces.

Alloy	At 20 Hours		At 200 Hours	
	750 °C Oxide Surfaces	Mechanically Cleaned Surfaces	750 °C Oxide Surfaces	Mechanically Cleaned Surfaces
FA-84	780	375	836	430
FA-129	351	445	435	425
FAL-Mo	217	306	405	321
FAPY	323	327	499	346

STAGE I CONCLUSIONS: AS-PROCESSED AND 750 °C SURFACES

Electrochemical aqueous-corrosion studies showed that retained high-temperature surface oxides on iron aluminides (FA-84, FA-129, FAL-Mo, and FA-385) and a related alloy (FAPY), produced during thermomechanical processing or heat treatment (750 °C in air for one hour), caused major reductions in pitting corrosion resistance in a mild acid-chloride solution designed to simulate aggressive atmospheric corrosion. It is hypothesized that cracks or flaws in the high-temperature oxides created crevice geometries which enhanced the susceptibilities of the materials to localized corrosion. Removal of the oxides by mechanical grinding or chemical cleaning restored the corrosion resistance. Standard pickling solutions employed for stainless steels, based on nitric plus hydrofluoric acids, worked well for the FA-84 and FA-129 iron aluminides, which do not contain molybdenum, producing clean, smooth surfaces. For the FAL-Mo and FAPY alloys, which do contain molybdenum, the solutions were effective in removing the oxides, but not effective in producing acceptably smooth surfaces.

U-bend stress-corrosion-cracking tests conducted in the mild acid-chloride solution on FA-84, FA-129, FAL-Mo, and FAPY showed that under electrochemical cathodic hydrogen-charging conditions, specimens with 750 °C heat treatment oxide surfaces did not undergo cracking, whereas specimens with mechanically-cleaned surfaces did undergo cracking. The results suggest that the 750 °C oxide retards penetration of hydrogen into the metal substrate and, consequently, is beneficial in terms of improving resistance to environmental embrittlement.

STAGE II PROCEDURES: 1000 °C SURFACES

Stage II of this project involved a study of the effects of in-service-type high-temperature oxides on the room-temperature aqueous corrosion and environmental embrittlement properties of iron aluminides. The study was designed to be consistent with past and on-going research by Tortorelli and DeVan at ORNL. These investigators have evaluated the high-temperature oxidation resistances of Fe₃Al-based iron aluminides (FA-129 and FAL) in air to simulate combustion products.⁵ Equally importantly, Tortorelli and Alexander are conducting studies to characterize the chemical, morphological, and mechanical properties of the oxide scales created by these exposures after cool-down to room temperature.⁶ The characteristics of these scales, and in particular the cracking/spallation characteristics, will have a strong influence on the aqueous-corrosion and hydrogen-embrittlement behaviors of the iron aluminides during shut-down periods at room temperature.

In terms of materials, FA-129 and FAL iron aluminides were selected to be consistent with Tortorelli and DeVan's work. In addition, the iron aluminide FAL-Mo was included. The chemical compositions are given in Table 4. FAL-Mo is very similar to FAL, but with the addition of 1.0 % Mo. Due to the Mo addition, it has been shown to have superior aqueous-corrosion resistance in acid-chloride and sulfur-bearing electrolytes.¹⁻³

Table 4. Chemical compositions of Stage II iron aluminides (atomic percent).

Alloy	Fe	Al	Cr	Mo	B	Zr	Nb	C	Y
FA-129	66.3	28.0	5.0	-	-	-	0.5	0.2	-
FAL	66.9	28.0	5.0	-	-	0.1	-	-	-
FAL-Mo	65.9	28.0	5.0	1.0	0.04	0.08	-	-	-

With regard to the high-temperature exposures, based on recommendations by Dr. Peter Tortorelli at ORNL, specimens of all alloys were exposed to air at 1000 °C for 24 hours, then furnace cooled. Aqueous corrosion and environmental embrittlement tests were conducted on specimens with, and for comparison, without the 1000 °C oxide. The 1000 °C oxide specimens were first ground through 600-grit SiC, then exposed to the 1000 °C heat treatment. The specimens without the 1000 °C oxide, i.e., the mechanically cleaned specimens, were first ground through 600-grit SiC, then exposed to the 1000 °C heat

treatment, then ground again through 600-grit SiC to remove the high-temperature oxides. Specimens were contained in an alumina crucible during all heat treatments.

The aqueous corrosion characterizations involved room-temperature cyclic anodic polarization tests, using the same procedures as described earlier in this report (Stage I Procedures). Polarization tests were conducted on FA-129, FAL and FAL-Mo in the mild acid-chloride solution [$\text{pH} = 4$ (H_2SO_4), 200 ppm Cl^- (NaCl)] to simulate aggressive atmospheric corrosion. In addition, tests were conducted on FAL in 0.001 M sodium tetrathionate solution ($\text{Na}_2\text{S}_4\text{O}_6$) to simulate an aqueous solution contaminated by sulfur-bearing combustion products.

The environmental embrittlement characterizations involved room-temperature slow-strain-rate tests (SSRTs) in the mild acid-chloride solution (FA-129, FAL and FAL-Mo) and the sodium tetrathionate solution (FAL). In this test, specimens are continuously strained in tension at a very slow rate to enhance corrosion and hydrogen embrittlement effects.

SSRT specimens were punched out of sheet stock of FAL, FAL-Mo, and FA-129 at ORNL and then machined to the final geometry, shown in Figure 9, at the University of Tennessee. A titanium wire was spot welded near the end of each specimen to provide electrical contact. The gripping sections of each specimen, as well as the titanium wire and its contact area, were coated with epoxy, thus allowing only the specimen gage section to be exposed to the solution. The tests were conducted at a strain rate of $1.2 \times 10^{-6} \text{ s}^{-1}$ in a load frame which contained the corrosion cell. The cell consisted of a glass vessel with polyvinyl-chloride top and bottom. The top section contained access ports for the titanium wire, a platinum counter electrode, and a salt bridge for use with the reference electrode. The top and bottom sections of polyvinyl chloride had compression fittings that allowed them to be tightened to the glass cell, independent of the load frame, which provided a suitable seal for the solution in the cell. The test specimen was secured to the load frame by iron aluminide pins that were coated with epoxy. The epoxy was used to eliminate galvanic effects, and iron aluminide was chosen as the pin material in case the epoxy cracked, thereby further minimizing galvanic effects. The SSRTs were conducted at the free corrosion potential, E_{corr} , and at the cathodic hydrogen-charging potential of -1500 mV(SHE).

Fig. 9. Slow-strain-rate test specimen geometry.

STAGE II RESULTS: 1000 °C SURFACES

Aqueous Corrosion

The cyclic anodic polarization behaviors of FA-129 (28Al-5Cr) in the 1000 °C oxide and mechanically cleaned conditions, as evaluated in the mild acid chloride solution, are shown in Figure 10. The 1000 °C oxide condition essentially showed no evidence of passivation. However, the mechanically cleaned condition remained passivated until the pitting potential, E_{pit} , of 530 mV(SHE) was exceeded. These results indicated that the mechanically cleaned condition is much more resistant to pitting corrosion in this solution.

The cyclic anodic polarization results in the mild acid chloride solution for FAL (28Al-5Cr-0.1Zr) in the 1000°C oxide and mechanically cleaned conditions are shown in Figure 11. The 1000 °C oxide condition, relative to the mechanically cleaned condition, produced a lower pitting potential (E_{pit}) and a lower value of ($E_{pit} - E_{corr}$). Both of these results indicated lower resistance to pitting corrosion for the 1000 °C oxide surface condition. However, the corrosion differences between the two surface conditions of FAL were not as dramatic as between the two surface conditions of FA-129, as seen by comparison of Figures 10 and 11.

The polarization results for FAL-Mo (28Al-5Cr-1Mo-0.08Zr) in the mild acid chloride solution are shown in Figure 12. As with FA-129 (Figure 10), significant differences existed in the polarization behaviors for FAL-Mo with the 1000°C oxide and mechanically cleaned surface conditions. The 1000 °C oxide condition essentially showed no evidence of

passivation. However, the mechanically cleaned condition remained passivated until a pitting potential, E_{pit} , of 600 mV(SHE) was exceeded. These results indicated that the mechanically cleaned condition is much more resistant to pitting corrosion than the 1000°C oxide condition.

The polarization results for FAL-Mo in the 0.001 M sodium tetrathionate solution are shown in Figure 13. This solution proved to be very aggressive toward both the 1000°C oxide and mechanically cleaned conditions. In fact, passivation occurred for neither surface condition, only active corrosion.

The overall polarization results in the mild acid chloride solution are summarized in Figure 14, where the resistance to localized corrosion (in terms of the measured parameter ($E_{\text{pit}} - E_{\text{corr}}$)) is shown for all materials in both surface conditions. In cases where passivation was not obvious, E_{pit} was taken as the potential corresponding to a current density of $10 \mu\text{A}/\text{cm}^2$. It is apparent that the mechanically cleaned surfaces for all materials demonstrated superior localized corrosion resistance relative to the 1000 °C oxide surfaces.

Fig. 10. Cyclic anodic polarization behaviors of FA-129 with 1000 °C oxide and mechanically-cleaned surfaces in the mild acid chloride solution.

Fig. 11. Cyclic anodic polarization behaviors of FAL with 1000 °C oxide and mechanically-cleaned surfaces in the mild acid chloride solution.

Fig. 12. Cyclic anodic polarization behaviors of FAL-Mo with 1000 °C oxide and mechanically-cleaned surfaces in the mild acid chloride solution.

Fig. 13. Cyclic anodic polarization behaviors of FAL with 1000 °C oxide and mechanically-cleaned surfaces in the 0.001 M sodium tetrathionate solution.

Fig. 14. Effects of 1000 °C oxide and mechanically cleaned surfaces on localized corrosion resistance in the mild acid chloride solution.

Environmental Embrittlement

The slow-strain-rate test results for replicate specimens of FA-129, FAL and FAL-Mo in the mild acid chloride solution, in terms of percent total plastic elongation and fracture stress values, are presented in Table 5. The average percent elongation results at the free corrosion potential, E_{corr} , and at the hydrogen-charging potential, -1500 mV(SHE) , are summarized in graphical form in Figure 15(a) and (b), respectively. It is seen that the 1000°C oxide surfaces produced higher ductilities than the mechanically cleaned surfaces for all materials at both potentials. At E_{corr} , the average increase in ductility was 39 %, and at -1500 mV(SHE) , 52 %. At the hydrogen-charging potential, the FAL and FAL-Mo 1000°C oxides were noticeably more effective in improving the ductilities. These results imply that the 1000°C oxides retard hydrogen penetration into the metal substrates and, therefore, improve the resistances of the materials to environmental embrittlement. Under the conditions of this study, the 1000°C oxides produced beneficial effects, but not dramatically beneficial effects.

The slow-strain-rate test results for FAL in the 0.001 M sodium tetrathionate solution, in terms of percent total plastic elongation and fracture stress values, are presented in Table 6. It is seen that the 1000°C oxide and mechanically cleaned surfaces produced approximately equal ductilities at each of the potentials. Therefore, in this solution, which was considerably more aggressive than the mild acid chloride solution, no beneficial effects were observed for the 1000°C oxide surfaces in terms of resistance to environmental embrittlement.

Table 5. Results of slow strain-rate tests on iron aluminides in the mild acid chloride solution.

Material	Potential	Surface Condition	Fracture Stress		Percent Total Plastic Elongation
			(ksi)	(MPa)	
FAL	E_{corr}	Mechanically Cleaned	22	150	2.23
		Mechanically Cleaned	27	183	3.13
		1000°C Oxide	24	163	3.32
		1000°C Oxide	26	177	3.76
	-1500 mV SHE	Mechanically Cleaned	21	143	1.42
		Mechanically Cleaned	25	170	1.72
		1000°C Oxide	19	129	2.23
		1000°C Oxide	23	156	2.49
FAL-Mo	E_{corr}	Mechanically Cleaned	30	204	1.09
		Mechanically Cleaned	38	258	1.27
		1000°C Oxide	24	163	1.52
		1000°C Oxide	30	198	1.62
	-1500 mV SHE	Mechanically Cleaned	40	272	1.92
		Mechanically Cleaned	48	326	2.02
		1000°C Oxide	34	231	3.87
		1000°C Oxide	38	254	3.95
FA-129	E_{corr}	Mechanically Cleaned	35	238	1.71
		Mechanically Cleaned	39	261	1.83
		1000°C Oxide	31	211	2.72
		1000°C Oxide	37	251	2.80
	-1500 mV SHE	Mechanically Cleaned	29	197	1.12
		Mechanically Cleaned	35	239	1.24
		1000°C Oxide	23	156	1.23
		1000°C Oxide	25	170	1.29

(a)

(b)

Fig. 15. Slow-strain-rate test results for iron aluminides in the mild acid chloride solution at (a) the free corrosion potential, E_{corr} , and (b) the hydrogen-charging potential, -1500 mV(SHE).

Table 6. Results of slow strain-rate tests on FAL iron aluminide in 0.001 M sodium tetrathionate solution.

Material	Potential	Surface Condition	Fracture Stress		Percent Total Plastic Elongation
			(ksi)	(MPa)	
FAL	E_{corr}	Mechanically Cleaned	23	158	1.2
		1000°C Oxide	23	158	1.2
	-1500 mV SHE	Mechanically Cleaned	32	220	1.4
		1000°C Oxide	26	179	1.6

For the slow-strain-rate tests conducted at the hydrogen-charging potential, the resultant cathodic current densities for the alloys in the two surface conditions are shown in Figure 16 for the mild acid chloride solution and in Figure 17 for the sodium tetrathionate solution. As discussed earlier, these cathodic current densities are directly proportional to the electrochemical reduction rate at the specimen surfaces. At this very negative cathodic potential, the dominant reduction reactions are the reduction of hydrogen ions and water to hydrogen atoms which, with time, combine to form hydrogen molecules, then hydrogen vapor bubbles, i.e., $2\text{H}^+ + 2\text{e} \rightarrow 2\text{H}(\text{H}_2)$ and $2\text{H}_2\text{O} + 2\text{e} \rightarrow 2\text{H}(\text{H}_2) + 2\text{OH}^-$. It is the diffusion of atomic hydrogen (H) into the metal substrate which, of course, can cause hydrogen embrittlement. Another possible reduction reaction, which could contribute to the measured cathodic current densities is the reduction of the oxides to metallic form. However, for the 1000 °C oxides, it is believed that the dominant oxide is alumina, which thermodynamically should not undergo reduction at -1500 mV(SHE). As with the U-bend tests in Stage I of this project, it was anticipated that the cathodic current densities for the oxide specimens would be considerably lower than those for the mechanically cleaned specimens, simply because of the higher electrical resistivity of the oxide. This result was observed for FAL in the chloride and tetrathionate solutions, but not for FA-129 and FAL-Mo in the chloride solution. Reasons for these results are not clear at the present time. It is noted that the current densities observed in the dynamic slow-strain-rate tests (Figures 16 and 17) were considerably higher than those observed in the static U-bend tests (Table 3) -- a result possibly due to continuous disruption of the oxides and passive films by dislocation movement to the surfaces during the slow-strain-rate tests.

Fig. 16. Cathodic current densities in the mild acid chloride solution during hydrogen charging for 1000 °C oxide and mechanically cleaned surfaces.

Fig. 17. Cathodic current densities in the 0.001 M sodium tetrathionate solution during hydrogen charging for 1000 °C oxide and mechanically cleaned surfaces.

In addition to the data collected during the slow strain-rate tests, it was desirable to determine the mode of cracking that led to failure. Therefore, fractography by scanning electron microscopy was performed. A typical fractograph for all of the specimens is shown in Figure 18. The fracture surface is indicative of intergranular brittle fracture which is consistent with hydrogen embrittlement of the slow-strain-rate specimens. A typical microstructure for the specimens after annealing at 1000 °C for 24 hours is shown in Figure 19. It is seen that the fracture surface features (Figure 18) were very similar in shape and size to the grain structure (Figure 19).

STAGE II CONCLUSIONS: 1000 °C SURFACES

Electrochemical aqueous-corrosion studies showed that retained in-service-type high-temperature surface oxides (1000 °C for 24 hours) on FA-129, FAL and FAL-Mo iron aluminides caused major reductions in pitting corrosion resistance in a mild acid-chloride solution designed to simulate aggressive atmospheric corrosion. Removal of the oxides by mechanical grinding restored the corrosion resistance. In a more aggressive solution of sodium tetrathionate, designed to simulate an aqueous environment contaminated by sulfur-bearing combustion products, only active corrosion was produced by both the 1000 °C oxide and mechanically cleaned surfaces of FAL.

Slow-strain-rate tests on FA-129, FAL and FAL-Mo at free-corrosion and hydrogen-charging potentials in the mild acid chloride solution showed somewhat higher ductilities (on the order of 50 %) for the 1000 °C oxide surfaces as compared to the mechanically cleaned surfaces. These results indicate that the 1000 °C oxide retards the penetration of hydrogen into the metal substrate and, consequently, is beneficial in terms of improving resistance to environmental embrittlement. For FAL in the aggressive sodium tetrathionate solution, no differences were observed in the ductilities produced by the 1000 °C oxide and mechanically cleaned surfaces.

Fig. 18. Fracture surface of FAL slow-strain-rate 1000 °C oxide specimen tested at -1500 mV SHE.

Fig. 19. Annealed microstructure of FAL slow-strain-rate specimen.

OVERALL RESULTS AND CONCLUSIONS

In terms of room-temperature aqueous corrosion behavior in the mild acid chloride solution, designed to simulate aggressive atmospheric corrosion, the retained high-temperature surface oxides, including as-processed oxides, 750 °C oxides (reflective of heat treatment) and 1000 °C oxides (reflective of in-service oxidation), consistently produced detrimental results relative to mechanically or chemically cleaned surfaces. Specifically, the pitting corrosion resistances were much lower for the as-processed and 750 °C surfaces, relative to the cleaned surfaces, for FA-84, FA-129 and FAL-Mo Fe₃Al-based iron aluminides, FA-385 FeAl-based iron aluminide and disordered FAPY. Furthermore, the pitting corrosion resistances were much lower for the 1000 °C surfaces, relative to the cleaned surfaces, for FA-129, FAL and FAL-Mo Fe₃Al-based iron aluminides. It is believed that the detrimental corrosion effects associated with the high-temperature oxides are related to flaws in the oxides which serve as crevice corrosion sites, thus increasing the susceptibility to localized corrosion.

In terms of room-temperature environmental embrittlement behavior in the mild acid chloride solution, the retained high-temperature surface oxides consistently produced beneficial results relative to mechanically cleaned surfaces. Specifically, in 200-hour U-bend stress-corrosion-cracking tests of FA-84, FA-129, FAL-Mo and FAPY, specimens with 750 °C oxide surfaces demonstrated lower tendencies for cracking than specimens with cleaned surfaces. Furthermore, slow-strain-rate tests of FA-129, FAL and FAL-Mo at free-corrosion and cathodic-hydrogen-charging potentials showed consistently higher ductilities for 1000 °C oxide surfaces as compared to cleaned surfaces. It is believed that the beneficial effects associated with the high-temperature oxides, i.e., greater resistance to environmental embrittlement, are due to the oxides serving as barriers to hydrogen penetration of the metal substrates in those surface regions where the oxides maintain adherency with the substrates.

As described above, the high-temperature oxides produce opposite effects with regard to localized corrosion (a detrimental effect) and hydrogen embrittlement (a somewhat beneficial effect). These opposite effects can be further rationalized by considering in more detail the proposed mechanisms in each case. With regard to localized corrosion, it is postulated that cracks in the high-temperature oxides provide crevice geometries which enhance the susceptibility to corrosion at the bottom of the cracks (e.g., $\text{Al} \rightarrow \text{Al}^{3+} + 3\text{e}$). The enhanced corrosion susceptibility at these crevice locations is caused by the higher chloride concentrations and lower pH values due to metal-ion hydrolysis. With regard to hydrogen

embrittlement of the high-temperature oxide specimens at the free-corrosion potential, it is proposed that the hydrogen is produced at oxide/solution interface areas adjacent to the bottoms of the cracks by reduction of hydrogen ions and water (i.e., $H^+ + e \rightarrow H$ and $H_2O + e \rightarrow H + OH^-$). For this hydrogen to produce hydrogen embrittlement, it must diffuse through the oxide and into the metal substrate. At the hydrogen-charging potential, hydrogen is produced over virtually the entire oxide/solution interface area; and again, to produce embrittlement it must diffuse through the oxide and into the metal substrate. The same considerations apply to the cleaned specimen surfaces, except in this case the barrier to hydrogen diffusion into the metal substrate is the passive film, which is much thinner than the high-temperature oxide. Relative to these proposed mechanisms, the results of the present study indicate that the high-temperature oxides, in those surface regions where they are retained, function as better barriers to hydrogen diffusion into the metal substrates than the much-thinner passive films.

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