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Influence of Compositional Modifications on the Corrosion of Iron Aluminides by Molten Nitrate Salts

P. F. Tortorelli
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DEPARTMENT OF ENERGY

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INFLUENCES OF COMPOSITIONAL MODIFICATIONS ON THE CORROSION OF IRON ALUMINDES BY MOLTEN NITRATE SALTS

P. F. Tortorelli and P. S. Bishop

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INFLUENCES OF COMPOSITIONAL MODIFICATIONS ON THE CORROSION OF IRON ALUMINIDES BY MOLTEN NITRATE SALTS*

ABSTRACT

The corrosion of iron-aluminum alloys by molten nitrate salt as a function of aluminum, chromium, and other minor elements has been studied as part of an alloy design effort aimed at the development of a strong, ductile, corrosion-resistant FeAl type of aluminide. Short-term weight change data were used to examine the compositional dependence of the corrosion processes that occurred upon exposure of iron aluminides to highly oxidizing nitrate salts at 650°C. Corrosion resistance was found to increase with increasing aluminum concentration of the alloy up to approximately 30 at. % Al. Chromium additions to the aluminide were not detrimental and may have improved the corrosion behavior for certain aluminum concentrations. No effects of minor alloying additions (C, B, Ti, and Zr) could be determined. The best overall corrosion resistance as measured by weight change results were obtained for an Fe-35.8 at. % Al aluminide containing some chromium. Based on linear weight loss kinetics, the weight change measurements for the most resistant compositions predict corrosion rates of 300 μm/year or less at 650°C. These rates are substantially better than typical nickel-based alloys and stainless steels.

From a consideration of the weight changes; the microstructural, thermodynamic, and X-ray diffraction data; and the salt analyses, corrosion of iron aluminides by the molten nitrate salt appears to be controlled by oxidation of base metal components and a slow release of material from an aluminum-rich product layer into the salt. The rate of release was substantially lower than that previously found for iron and iron-based alloys. This would imply that corrosion of iron aluminides could be minimized by maximizing the surface coverage of this aluminum-rich layer either by alloying or by an appropriate preoxidation treatment.

INTRODUCTION

Some of the uses of molten nitrate salts include removing or providing heat for chemical reactions,[1] heat treatment baths,[1] and coolant and thermal energy storage materials for solar power towers.[2] In addition, the chemical properties of nitrate salt can be exploited

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to produce high-purity oxygen and nitrogen gases at a substantial energy savings compared to cryogenic production of oxygen.[3] Because of these types of applications and the scientific interest in these melts, a number of investigators have conducted electrochemical and corrosion studies with molten sodium and sodium-potassium nitrate salts (see, for example, refs. 4-15). Recently, a study of the corrosion of a variety of materials by a highly oxidizing molten nitrate salt showed that iron aluminides had relatively good resistance when compared to standard nickel- and iron-based alloys.[15] Therefore, as part of an effort to develop Fe-Al alloys with greater than 30 at. % Al, the corrosion behavior of such materials in nitrate salt was investigated. The resulting corrosion data for Fe-Al alloys as a function of aluminum, chromium, and other minor elements are presented and discussed below.

EXPERIMENTAL PROCEDURES

A cross-sectional view of the crucible assembly used for the exposures of the specimens to the molten nitrate salt is shown in Fig. 1. Continuous sparging of the melt with dry air was maintained during specimen exposures using gas bubbler tubes. The crucibles and tubes were alumina and were replaced after each melt cycle. A specimen was suspended on the end of a wire (usually 0.5-mm-diam zirconium) in the salt. Above the salt-gas interface, the other end of the wire was attached to a 6.4-mm-diam nickel rod, which was supported by the compression of an O-ring in one of the end flange fittings. Specimens were normally positioned approximately halfway down into the salt. Details of the experimental equipment and procedures are given elsewhere.[15]

Two different nitrate salt base compositions were used in the present study. Salt composition 1A consisted of 49 mol % $NaNO_3$ (44.7% by weight) and 51 mol % KNO_3 (55.3%). Additions of 1 mol % Na_2O_2 (0.85%) were made to obtain the 1B composition. Salt 2A was 100% $NaNO_3$; 0.4 mol % Na_2O_2 (0.4%) was added to 2A to yield 2B. All of the salt components were reagent grade. They were weighed on a 0.0- to 311.0-g mechanical balance. To facilitate additions to the molten salt during operation, Na_2O_2 was measured into individual containers in an inert atmosphere (to reduce moisture absorption). Single portions were then added to the melt through the crucible vent. The salt charge in an individual crucible was kept molten between specimen exposures and was only periodically replaced.

With one exception (described below), all the iron aluminides used in this study were prepared from arc-melted 500-g heats. Table 1 shows the compositions of the iron aluminides examined in this study. To prepare specimens, material was sheared or cut as

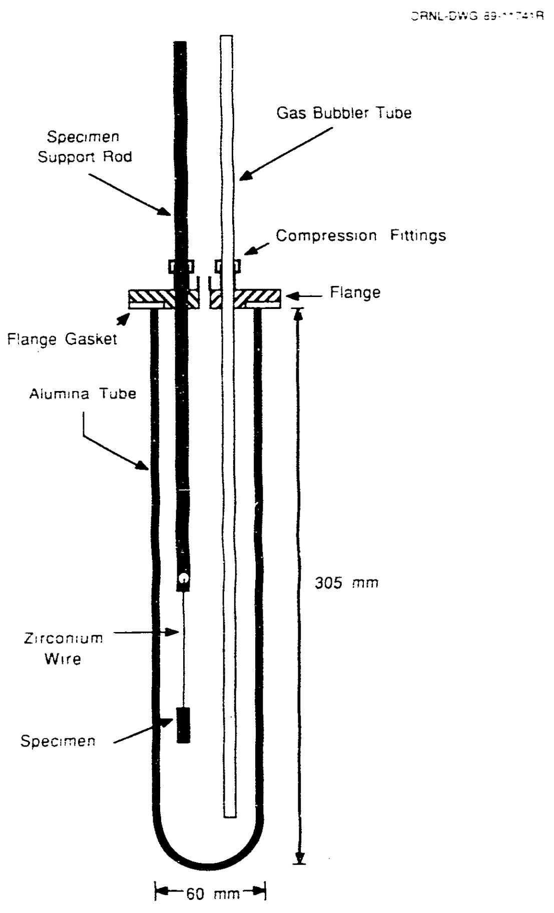

Fig. 1. Schematic drawing of sparged molten nitrate salt crucible assembly.

Table 1. Compositions of iron aluminides used in molten nitrate salt corrosion studies

Heat No. (FA-)	Concentration (at. %)		
	Al	Cr	Other
38	26	0	Ti, B
61	28	0	
39	30	0	Ti, B
76B	35	0	Ti, B
104	28	4	Zr, B, Y
118	30	2	Nb, Mo, Zr, B, Y
119	30	10	Nb, Mo, Zr
300	40	0	Zr, B
301	40	4	Zr, B
303	40	8	Zr, B
307	38	0	Zr, B
308	36.5	0	Zr, B
310	36.5	2	Zr, B
311	36.5	4	Zr, B
312	36.5	6	Zr, B
324	35.8	0	
325	35.8	0	B
326	35.8	0	Zr
328	35.8	5	Zr, B
350	35.8	0	Zr, B
353	35.8	5	Zr, B
6040*	40	0	
6040Nb*	40	0	Nb

*Not part of FA series.

close to the nominal dimensions, 5.1 × 2.5 × 19 mm, as possible. In some cases, a 1.5-mm-diam hole was drilled at one end of the specimen. Rough edges were removed by grinding and sanding. Selected specimens were lightly etched to remove surface films and then annealed at 850°C in air for 1 h. Final preparation involved surface cleaning using compatible solvents. All dimensional measurements were made with a dial caliper and all weights were measured with an electronic balance.

Upon removal from the salt, specimens were visually inspected, and any unusual observations were noted. Specimens were allowed to cool in air and rinsed in hot water. The wire was removed and the specimen was again rinsed. After air drying, the specimen weights and dimensions were recorded. The corroded surfaces and polished cross sections of selected specimens were analyzed by optical and scanning electron microscopy and energy dispersive X-ray (EDX) analysis. A few corroded and rinsed surfaces were examined by X-ray diffraction. Salt extracted from crucible melts was analyzed for selected elements by atomic absorption.

RESULTS

WEIGHT CHANGES

Table 2 shows weight change averages after 24-h exposures for over 20 heats of various iron aluminides used in crucible experiments with nitrate salts at 650°C. The number of duplicate experiments with a particular alloy ranged from 0 to 20.

Data from a series of exposures conducted in one particular salt charge (nominally 1B) were not included in these averages because weight changes from experiments with this salt mixture were consistently higher than the average of weight losses from all the other batches of salt in the 1B condition. This is clearly illustrated in Fig. 2, which compares the average weight losses of different iron aluminides exposed to the atypical salt with those from exposures to all other 1B salt charges (denoted 1B). The former salt was significantly more corrosive toward all four iron-aluminide compositions exposed in this environment.

Table 3, a subset of the results shown in Table 2, is a comparison of the average weight changes of similar compositions (in terms of aluminum and chromium) that differed only in the amount of minor alloying elements. Although there are differences in average weight losses, the effects of small amounts of B, Zr, Nb, and C are indeterminate when standard deviations are considered. Any influence of these elements on weight change was not sufficiently significant to cause any averages to fall outside the uncertainty limits. Likewise, as shown in Table 4, there was essentially no or little effect of pretreatment

Table 2. Average weight losses for iron aluminides exposed to nitrate salts at 650°C for approximately 24 h

Heat No. (FA-)	Weight change (g/m^2) 1B	Weight change (g/m^2) 2B
104	-85.1 ± 97.9	
118	-18.3 ± 19.5	
119	-17.3 ± 19.7	
300	-31.3 ± 19.8	
301	-10.8 ± 2.7	
303	-7.8 ± 5.5	
307	-29.6 ± 41.7	
308		-77.3 ± 80.2
310		-24.4
311		-70.8 ± 67.6
312		-26.6 ± 4.3
324	-5.6 ± 5.6	-45.2 ± 59.3
325	-3.4 ± 0.9	-6.0 ± 9.7
326	-1.2	-5.3 ± 4.3
328	-4.3 ± 1.5	-2.4 ± 2.3
350	-9.0 ± 13.6	-21.6 ± 18.1
353	-2.6 ± 0.3	-6.3 ± 7.7
38	-196	-240.1
39	-23.8 ± 30.6	-33.1 ± 22.4
61	-87.4 ± 45.9	
76B	-19.6 ± 10.8	-70.9 ± 28.3
6040*	-13.3 ± 8.2	
6060Nb*	-19.9 ± 17.6	

*Not part of FA series.

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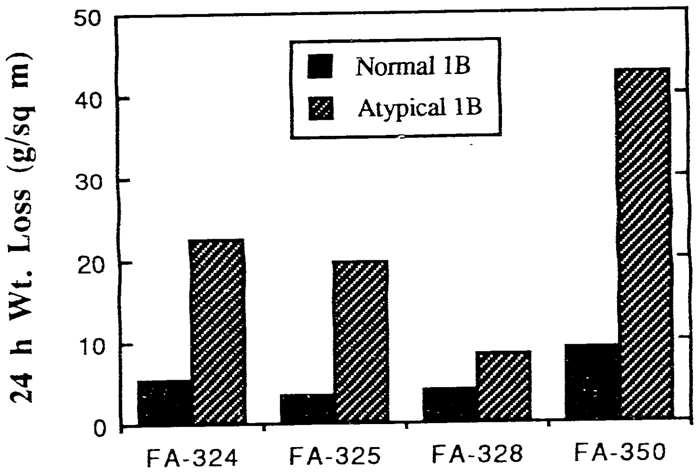

Fig. 2. Average weight change for different compositions of Fe-35.8 at. % Al exposed to $NaNO_3$-KNO_3-Na_2O_2 (salt 1B) at 650°C for approximately 24 h.

Table 3. Comparison of effects of minor alloying elements on 24-h corrosion losses of iron aluminides in $NaNO_3$-KNO_3-Na_2O_2 (salt 1B)

Al concentration (at. %)	Minor alloying element (at. %)	Weight change (g/m^2)
35.8	0	-5.6 ± 5.6
35.8	0.06B	-3.4 ± 0.9
35.8	0.1Zr	-0.8 ± 0.5
35.8	0.03B, 0.05Zr	-9.0 ± 13.6
35.8 + 5Cr	0.03B, 0.05Zr	-4.4 ± 4.2
35.8 + 5Cr	0.06B, 0.1Zr	-4.3 ± 1.5
40	0	-13.3 ± 8.2
40	0.2NbC	-19.9 ± 17.6

Table 4. Effects of preoxidiation in air (1 h at 850°C) on 24-h weight loss (g/m^2) in molten nitrate salt (salt 1B)

Al concentration (at. %)	Preoxidized	All conditions
30Al-2Cr	-27.1 ± 28.6	-18.3 ± 19.5
40Al	-34.5 ± 13.9	-31.3 ± 19.8
40-4Cr	-11.6 ± 3.5	-10.8 ± 2.7
40-8Cr	-11.8	-7.8 ± 5.5
35.8Al	-11.4 ± 16.4	-9.0 ± 13.6
35Al	-22.0 ± 13.2	-19.6 ± 10.8
40Al	-12.5 ± 8.5	-13.3 ± 8.2

(oxidation from the 850°C annealing treatment in air) on the weight change results. Selected specimens of Fe-35.8Al were mechanically polished to a metallographic surface finish to check for effects of surface roughness or preparation on the weight change measurements and none were found. Based on these results, effects of variations in the concentrations of aluminum and chromium on short-term weight change results can be compared without regard to the exact specimen preparation or concentration of minor alloying additions.

A series of Fe-Al binary alloys (with only very small, if any, concentrations of minor alloying elements) were used to examine the effect of aluminum concentration on corrosion by the nitrate salts over a range of 26 to 40 at. % Al. The weight losses of the iron aluminides decreased with increasing aluminum concentration up to 30 at. % Al, but were not sensitive to aluminum content at higher concentrations. This trend was observed at 650°C for both the $NaNO_3$-KNO_3-Na_2O_2 [salt 1B, Fig. 3(*a*)] and the $NaNO_3$-Na_2O_2 [salt 2B, Fig. 3(*b*)] environments. A binary Fe-35.8Al alloy composition (FA-350) was used to determine possible effects on corrosion resistance resulting from scaling up iron-aluminide heats from 500 g to 10 kg. The average 24-h weight loss for material from the 10 kg FA-350 (4.4 ± 4.2 g/m^2) was less than for the 500-g heat (11.4 ± 16.4 g/m^2), but the standard deviations overlap. Thus, no significant effect of scale-up on corrosion in the nitrate salt can be established from the present data.

Specific experiments to investigate the influence of chromium on corrosion by the molten salt were also conducted. The results for salt condition 1B are shown in Fig. 4, which indicates no discernible influence of chromium on 24-h weight changes for exposures to $NaNO_3$-KNO_3-Na_2O_2, outside the limits set by the scatter in the measurements (see Table 2). A similar conclusion was obtained from consideration of data from experiments in Salt 2 (Table 5); no major trend was apparent and any possible small effect was masked by the relatively large standard deviations in certain cases. The presence of chromium in the iron aluminides did not seem to affect the dependence of the weight loss on aluminum concentration (see above): the average weight loss for Fe-Al-4Cr was significantly higher for a 28 at. % Al composition (FA-118, 85.1 g/m^2) than for one with 40 at. % Al (FA-301, 10.1 g/m^2).

A limited number of experiments were conducted at longer exposure times and at different temperatures using the Fe-35Al heat (FA-76B). As reported previously,[15] the influence of the peroxide addition on corrosion is relatively short-lived; most of the weight change occurs within the first 24 h unless the $NaNO_2$ is periodically refreshed (see Fig. 5). This type of behavior was also observed in more recent experiments, which are described by the results shown in Table 6. For a given composition and salt condition, 48-h weight

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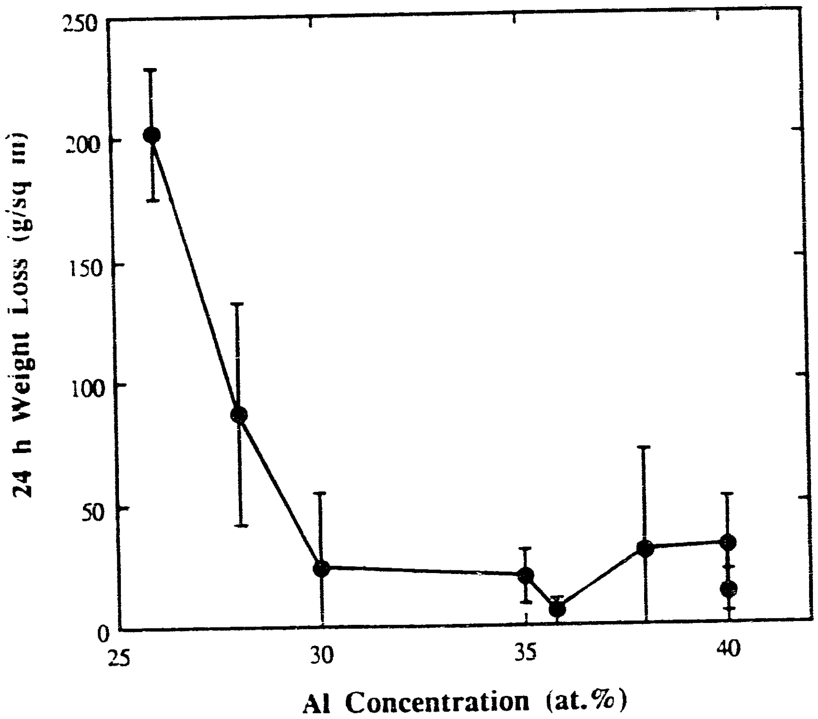

Fig. 3. Average weight loss as a function of aluminum concentration for iron aluminides exposed at 650°C for 20-30 h: (*a*) $NaNO_3$-KNO_3-Na_2O_2 (salt 1B) and (*b*) $NaNO_3$-Na_2O_2 (salt 2B).

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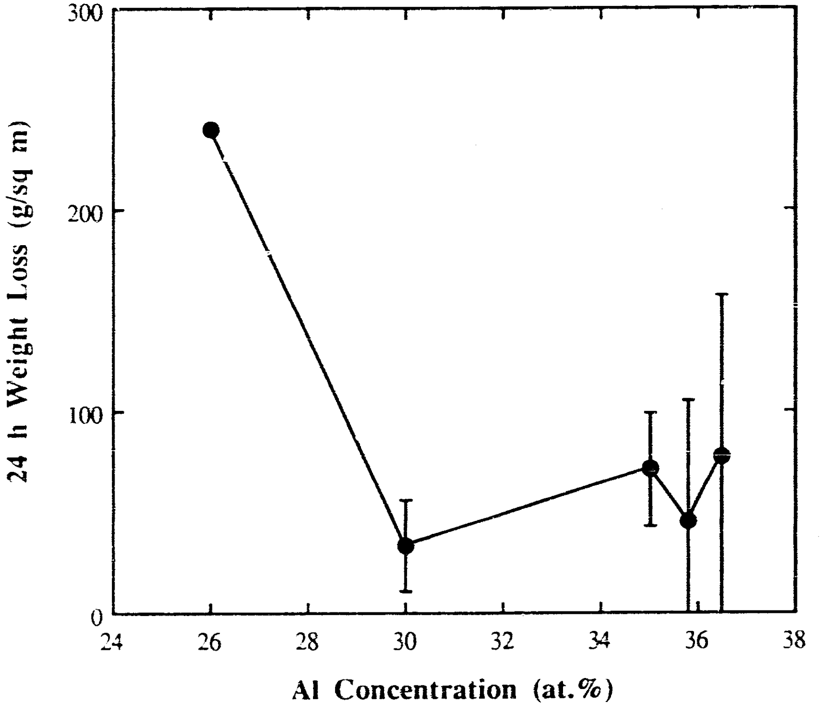

Fig. 4. Average 24-h weight loss for iron aluminides exposed to $NaNO_3$-KNO_3-Na_2O_2 at 650°C as a function of chromium and aluminum concentrations (in at. %).

Table 5. Influence of chromium concentration on 24-h weight loss in nitrate salts

Al concentration (at.%)	Cr concentration (at.%)	Heat No. (FA-)	Weight change (g/m^2) for 2B
35.8	0	325	-6.0 ± 9.7
35.8	0	326	-5.3 ± 4.3
35.8	5	328	-2.4 ± 2.3
36.5	0	308	-77.3 ± 80.2
36.5	2	310	-24.4
36.5	4	311	-70.8 ± 67.6
36.5	6	312	-26.6 ± 4.3

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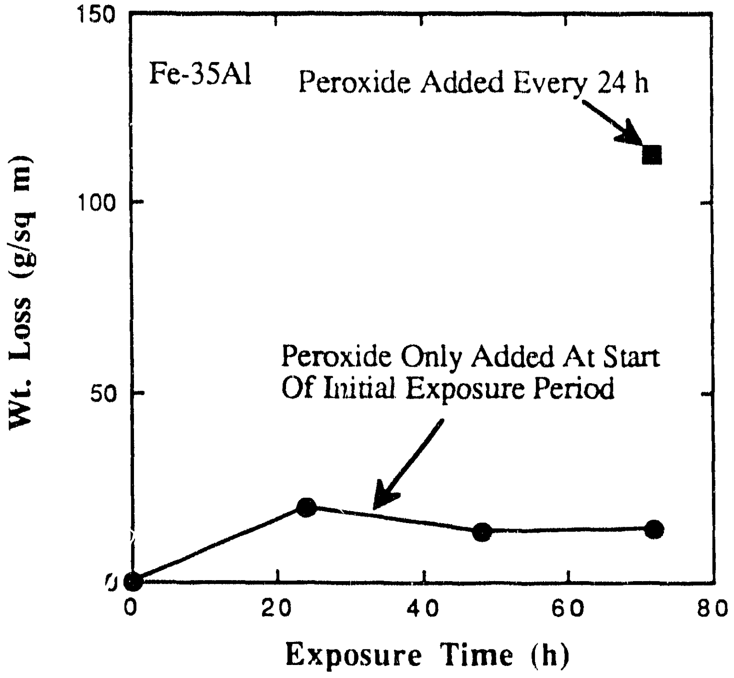

Fig. 5. Weight loss versus exposure time for Fe-35 at. % Al exposed to $NaNO_3$-KNO_3-Na_2O_2 at 650°C.

Table 6. Effect of peroxide additions as a function of exposure time for iron aluminides in nitrate salts

Heat No. (FA-)	Al concentration (at.%)	Cr concentration (at. %)	Exposure time (h)	Salt condition	Weight change (g/m^2)
350	35.8	0	24	1B	-9.0 ± 13.6
350	35.8	0	48	1B	-3.3
350	35.8	0	48	2B	-40.4
324	35.8	0	24	2B	-45.2 ± 59.3
353	35.8	5	24	1B	-2.6 ± 0.3
353	35.8	5	48	1B	-6.7
353	35.8	5	24	2B	-6.3 ± 7.7
353	35.8	5	48	2B	-3.8
104	28	0	24	1B	-85.1 ± 97.9
104	28	0	72	1B	-68.5
326	35.8	0	24	2B	-5.3 ± 4.3
326	35.8	0	70	2B	-9.0

changes were the same as those measured after 24 h. When peroxide is added every 24 h, the increase in weight loss with time is approximately linear. This type of behavior, described originally in a prior report,[15] is shown in Fig. 6, which plots cumulative weight loss (obtained from successive exposure of the same specimen) versus exposure time under salt condition 1B where 1 mol % Na_2O_2 was added to the melt at the beginning of each exposure segment. Note the sensitive dependence of the weight losses and corrosion rates (obtained from slopes of the fitted lines in Fig. 6) on temperature.

MICROSTRUCTURAL ANALYSES

Polished cross sections of selected specimens from alloys FA-324, -325, -326, and -328 were examined by optical metallography. Microstructural observations for other FA-76B specimens are described elsewhere.[15] Representative micrographs are shown in Fig. 7, which also includes the weight changes measured for the respective specimens. As can be seen from the figure, no evidence of a corrosion product layer was found. Likewise, X-ray diffraction of several specimen surfaces after exposure and cleaning did not detect anything but the base material. Only isolated areas of a possible sodium-containing product

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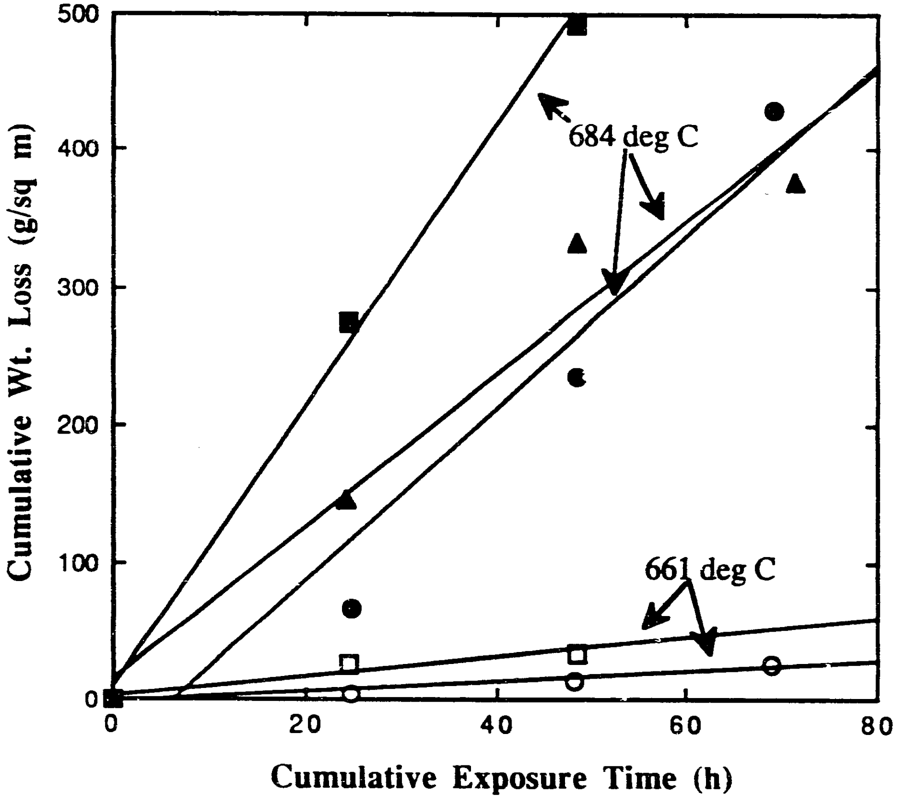

Fig. 6. Cumulative weight loss versus time for Fe-35 at. % Al exposed to $NaNO_3$-KNO_3-Na_2O_2 at 661 and 684°C. Peroxide was refreshed during each exposure segment.

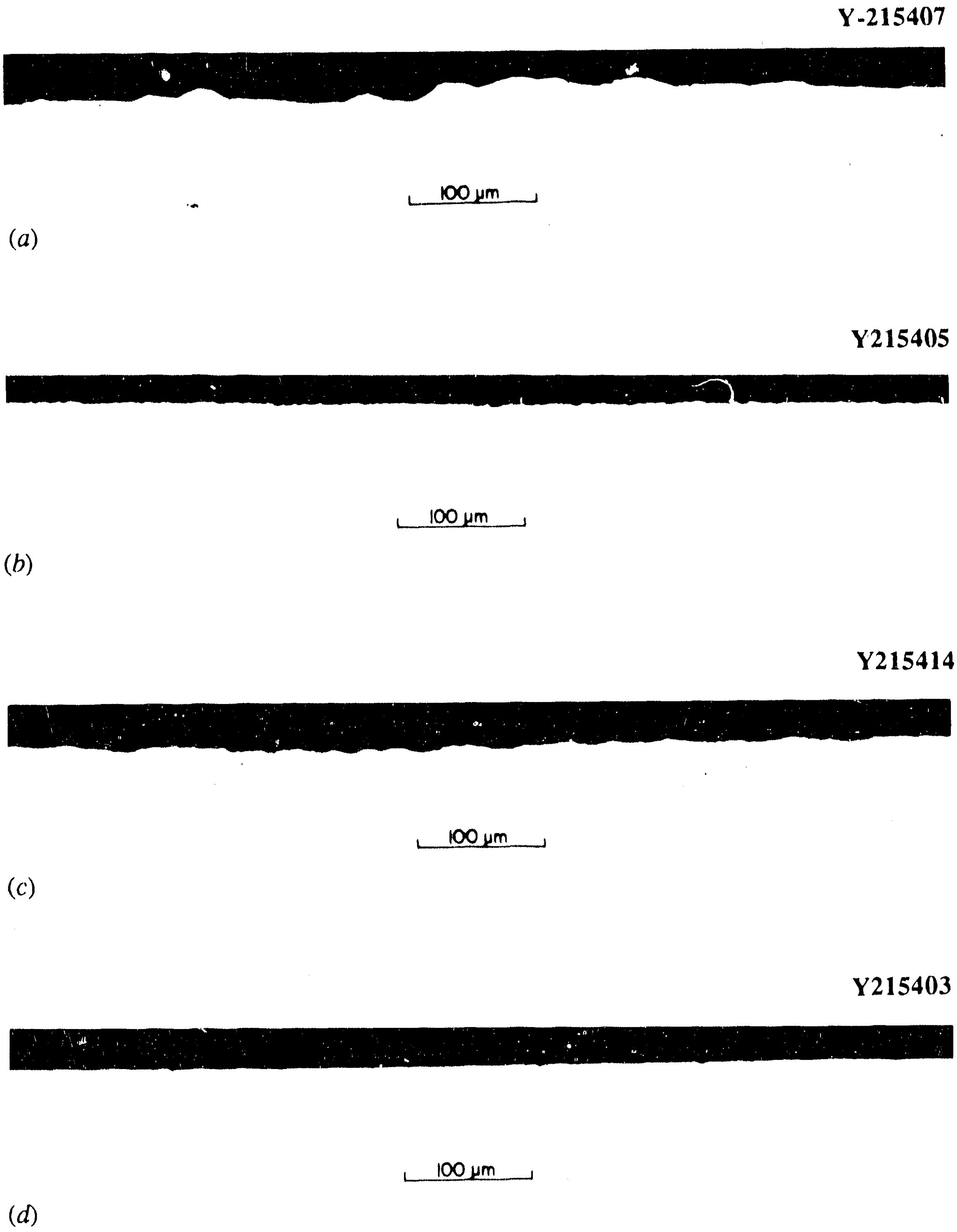

Fig. 7. Polished cross sections of iron aluminides exposed at 650°C: (*a*) FA-325, $NaNO_3$-Na_2O_2 (salt 1B), weight loss of 4.4 g/m^2; (*b*) FA-328, $NaNO_3$-KNO_3-Na_2O_2 (salt 1B), weight loss of 5.9 g/m^2; (*c*) FA-325, $NaNO_3$-Na_2O_2 (salt 2B), weight loss of 5.1 g/m^2; and (*d*) FA-328, $NaNO_3$-Na_2O_2 (salt 2B), weight loss of 0.7 g/m^2.

layer (area marked A in Fig. 8) were detected by scanning electron microscopy and EDX spectroscopy even when the salt was not removed from the surface (see Fig. 8).[15]

The surface roughness evident in the micrographs comprising Fig. 7 was not necessarily a result of the corrosion process. Specimens that were not mechanically polished prior to exposure to the salt (most were not) had varying degrees of surface roughness depending on the particular heat of material. Examples of cross sections of starting surfaces of specimens that were lightly etched and annealed are shown in Fig. 9. (Examination of the surfaces of as-received material revealed that such roughness was not due to the surface preparation treatments.) Furthermore, there was no correlation between the measured weight losses and the surface roughness of the specimens exposed to the salt. In fact, comparison of two FA-325 specimens that suffered weight losses of 40.0 and 4.4 g/m^2, respectively, show just the opposite trend (Fig. 10), as did FA-326 (Fig. 11). In these instances, the corrosion process seemed to result in smoother surfaces. In both Figs. 10 and 11, the specimen shown in (*a*) was exposed to the atypical salt described above.

SALT ANALYSES

Results of salt analyses as obtained by use of atomic absorption spectroscopy are shown in Table 7, which includes data for salts that contained only iron-aluminide specimens. Random samples of salt were taken after exposures of multiple specimens. Salt 3-2 was the one in which the trend of significantly higher weight losses was discovered (see above). Note that, for this salt, the concentrations of Fe, Al, and Cr were all higher compared to the other salt samples and that it contained a relatively high amount of silicon.

DISCUSSION

It has already been shown[14,15] that weight change can differentiate the relative corrosion resistance of materials exposed to this aggressive nitrate salt where significant variances in susceptibilities exist and that the superior behavior of FeAl in this environment can be demonstrated with the use of such measurements. However, the relatively large uncertainty limits typically associated with weight change measurements of specimens exposed to this type of salt (see Table 2 and ref. 15) makes detection of minor effects of understanding of the corrosion process. Other techniques employing electrochemical measurements or thermogravimetry (using thin layers of salt on the specimens) possibly could be quite helpful in this regard. Nevertheless, as discussed below, when the major

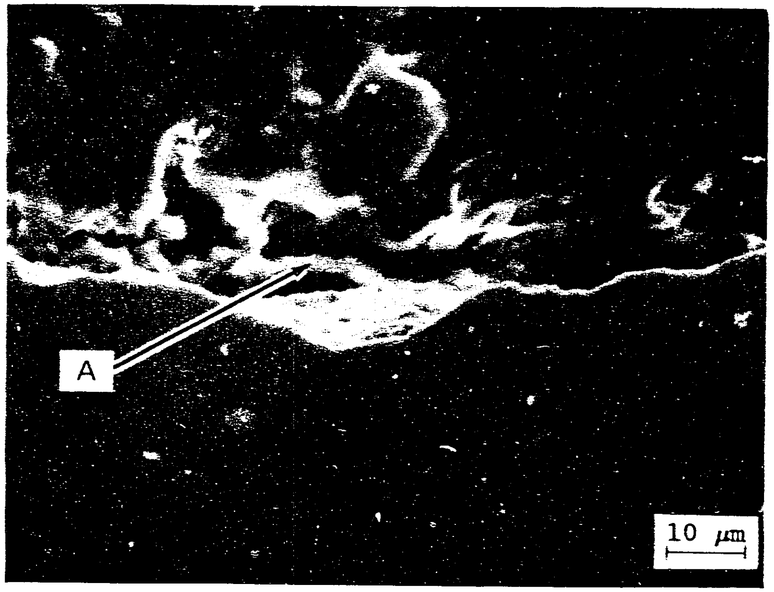

Fig. 8. Polished cross section of Fe-35 at. % Al exposed to $NaNO_3$-KNO_3-Na_2O_2 (salt 1B) for 24 h at 650°C. Energy dispersive X-ray analysis indicated presence of a sodium-containing product at A.

Fig. 9. Polished cross sections of unexposed iron aluminides (starting surface roughness): (*a*) FA-325 and (*b*) FA-328.

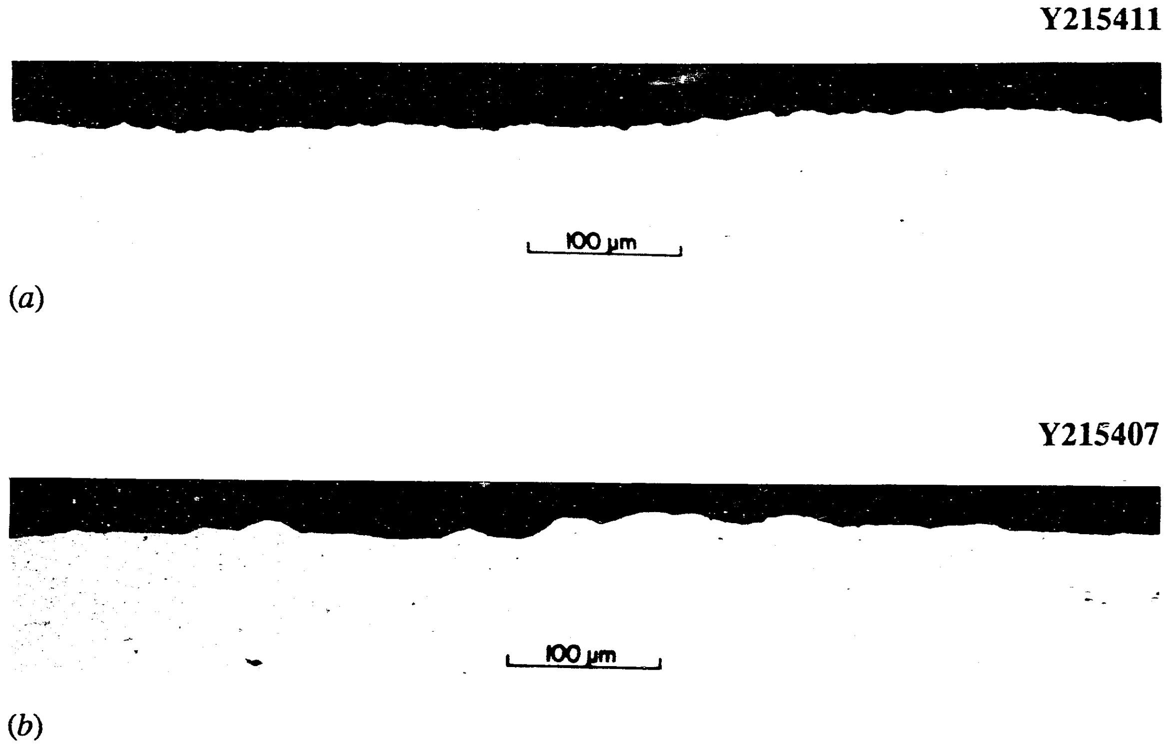

Fig. 10. Polished cross sections of FA-325 exposed at 650°C to $NaNO_3$-KNO_3-Na_2O2 (salt 1B): (*a*) weight loss of 40.0 g/m^2 and (*b*) weight loss of 4.4 g/m^2.

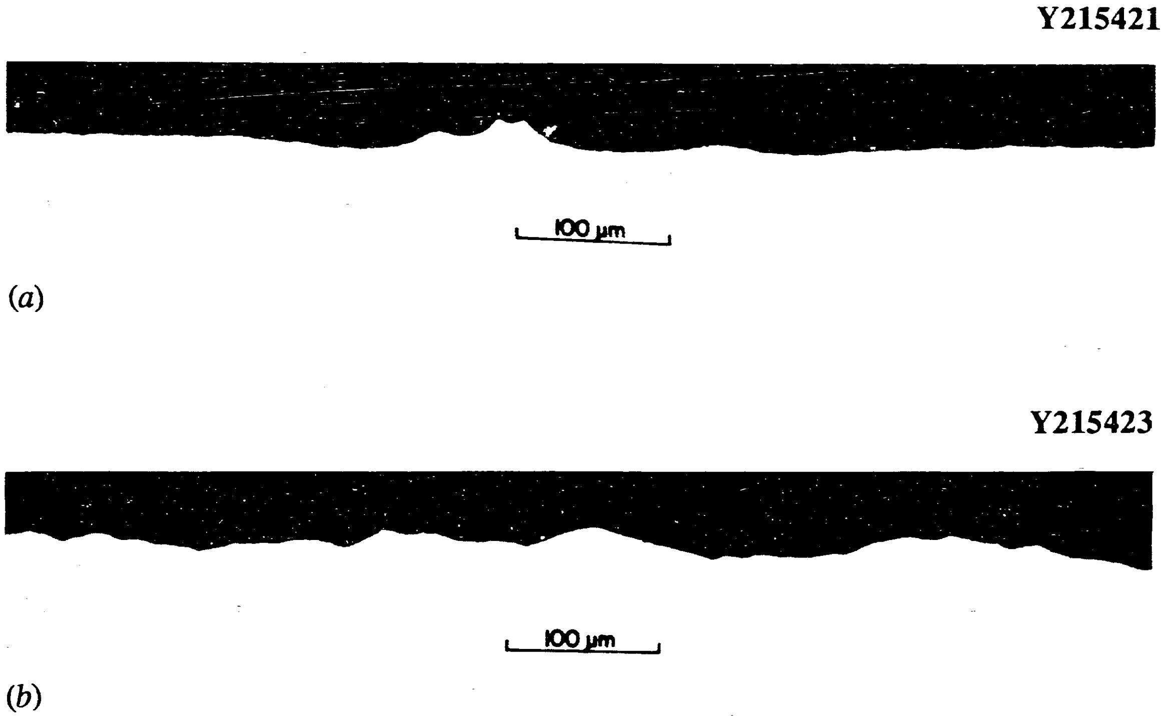

Fig. 11. Polished cross sections of FA-326 exposed at 650°C to $NaNO_3$-Na_2O_2 (salt 2B): (*a*) weight loss of 118.7 g/m^2 and (*b*) weight loss of 5.2 g/m^2.

Table 7. Analysis of salt samples from crucibles (by atomic absorption)

Element	Concentration (wt ppm)			
	2-2[a]	3-2[b]	3-3	4-2[c]
Al	<9.3	31	3.1	<10.0
Cr	3.1	4.9	<0.2	1.6
Fe	3.8	170	<0.5	<2.1
Si	<37	130	13	54
Zr	<3.7	<9.0	<1	<4.1

[a] First digit = crucible number, second digit = sequence number; crucibles 2 and 3 contained salt 1.

[b] Atypical salt.

[c] Salt 2.

contributions to weight changes are considered and these measurements are combined with thermodynamic information, salt analyses, and microstructural observations, a qualitative process by which FeAl type of alloys corrode at a relatively slow rate in high-temperature $NaNO_3(-KNO_3)-Na_2O_2$ can be postulated.

As demonstrated previously,[5-7] corrosion in molten nitrate salts can result both from solid-state oxidation of the base metal components and from solubility-driven dissolution reactions. Therefore, the significance of net weight change measurements must always be carefully considered in the analysis of results. The measured weight change, w, can be expressed as

$$w = w_o - w_s - w_d - w_l, \tag{1}$$

where w_s is the absolute value of the weight loss due to dissolution of metal ions into the salt, w_o is the gain due to the presence of oxygen in the specimen or its adhering scale, and w_d and w_l are the weight changes caused by removal of any (or all) of the surface reaction product when rinsing the salt from the specimen after exposure (descaling, w_d) and/or by mechanical scale detachment at temperature or during cooling (spallation, w_l). Weight losses (negative w) indicate corrosive attack, but, by themselves, do not distinguish between that caused by dissolution (weight loss during exposure) or scaling (weight gain followed by loss due to descaling and/or spallation). Significant weight gains are indicative of corrosion susceptibility that can be associated with oxidation and scale formation. However, relatively small weight changes do not necessarily mean a particular material is resistant to corrosion. If both dissolution and oxidation are occurring at rates such that oxygen uptake is approximately offset by metal ion dissolution, the net weight change would be small while the actual overall corrosion rate is substantial. However, in the present case of iron aluminides, there was no evidence for residual corrosion product on, or internal oxidation of, specimens exposed to the nitrate salts (see Figs. 7, 8, 10, and 11). Therefore, w_o must be very small, so that, in many cases,

$$w \approx -(w_s + w_d + w_l) \,. \tag{2}$$

The finding that $w_o \approx 0$ does not mean that the iron aluminides exhibiting weight losses have not been oxidized. Since weight losses were normally higher when the salt was periodically refreshed with highly oxidizing Na_2O_2, it probably indicates that any corrosion product that formed dissolved into the salt and/or became detached during or after exposure. Thermodynamic calculations and microstructural analyses have shown that Fe_2O_3, $NaFeO_2$, and $NaAlO_2$ should or can be stable in one or more of these nitrate salt mixtures.[8,9,13,15] In the absence of peroxide, exposure of iron to nitrate salt (1A, see above) led to weight gains,[9-12,15] thereby indicating that a corrosion product had formed. (In contrast, weight losses were measured upon exposure to salt 1B.[15]) The observation that weight changes for bulk alumina were negative (but relatively small) after exposures to 1B and 2B[15] may indicate that $NaAlO_2$ forms but dissolves (albeit rather slowly) into the salt. (There is some indirect microstructural evidence for this product based on work with Fe-Ni-Cr systems.[15])

When the aforementioned results for alumina are compared with relevant data from Table 2, it can be noted that the lowest weight losses of the alloys (that is, those near the minima of the respective scatterbands) agree with those measured for alumina (see Table 8). This correlation, when considered with the other evidence discussed above, would seem to indicate that a slowly dissolving product layer involving aluminum forms on iron aluminides exposed to the aggressive nitrate salts and produces lower weight losses compared to iron and other iron-based alloys. The decreasing weight losses with increasing aluminum concentration of iron aluminide (see Fig. 3) indicate that the Fe_2O_3 is either more soluble or less mechanically compatible than Al_2O_3 over the basicity range of these experiments. This is consistent with the findings regarding pure iron and Al_2O_3 exposures[15] discussed above. By being more corrosion resistant than the iron oxide products, this slowly thickening (small w_d, w_l) and/or dissolving (small w_s) aluminum-containing layer thereby imparts a beneficial effect.

Table 8. Comparison of weight changes of iron aluminides and bulk alumina exposed to molten nitrate salt at 650°C for approximately 24 h

Material	Weight change (g/m^2)	
	Salt 1B	Salt 2B
Al_2O_3	-2.0[a]	-8.2 ± 2.7[b]
Al_2O_3[c]		-11.0
FA-76B	-19.6 ± 10.8	-70.9 ± 28.3
FA-324	-5.6 ± 5.6	-45.2 ± 59.3
FA-350	-9.0 ± 13.6	-21.6 ± 18.1

[a] Single crystal.

[b] Includes data for gelcast alumina with 0 and 0.05 wt % MgO.

[c] Fine grained (~2 μm) alumina with 0.1 wt % MgO.

When Na_2O_2 was added to the salt at the beginning of an extended exposure period and not replenished, weight losses did not increase after about 24 h (Table 6). Such observations illustrate the importance of the peroxide in reactions associated with the formation and loss of, presumably, $NaAlO_2$ and $NaFeO_2/Fe_2O_3$ (for iron aluminide). This is not unexpected; solubilities of oxides in the nitrate salt should be highly dependent on salt basicity,[13,16] and it is reasonable that the addition of Na_2O_2 should significantly increase the solubility of these oxides in the basic dissolution regime.[15,16] Alternatively, or additionally, the increased basicity due to the introduction of the peroxide can change the stable species (for example, Fe_2O_3 to $NaFeO_2$) and lead to increased dissolution and/or growth of a product that is less mechanically compatible with the base material (causing delamination). The observation that pure $NaNO_3$ is more corrosive than the mixed $(Na,K)NO_3$ salt (see Table 2 and ref. 14) is consistent with a reaction process involving the formation of a sodium-containing product. The transitory effect of the peroxide additions (that is, the higher corrosion rate is only maintained if Na_2O_2 is refreshed) indicates that the additions are consumed in the corrosion process and that they directly participate in, rather than simply accelerate, the corrosion reaction.

The salt analyses shown in Table 7 are also consistent with a model of a slowly dissolving $NaAlO_2$ layer that is controlling the overall corrosion process. Note that the relative concentration of aluminum ions was about the same for the three normal salt charges from which samples were taken; but for the atypical, more corrosive salt, iron was in greater abundance. While the reason for the greater corrosivity of this salt is unknown (possibly related to contamination from an O-ring), the end result was the inability of an aluminum-containing surface layer to hinder the movement of iron into the salt, and correspondingly higher weight losses were measured (Fig. 2). The increased concentrations of all the principal components of the exposed iron aluminides for the highest weight losses is what is to be expected; but the finding is very significant in that, assuming no change in corrosion mechanism between the typical and atypical salts, it is indicative of a process(es) in which solid material is lost into the salt (by dissolution or in-salt spallation) rather than during the specimen removal from the crucible (by spallation) or during the cleaning process after exposure (descaling). In terms of Eq. (2), it is thus possible to assume $w_d \approx 0$ and that the principal contribution to w_l (if any) is from loss of scale into the salt.

There is a relatively high solubility of chromate and dichromate in the nitrate salt[5] and materials with significant amounts of chromium perform poorly in these salts at 625°C and above.[5,15] Therefore, it is interesting that the addition of chromium to iron aluminides had no large effect on their corrosion resistance (see Fig. 4 and Table 5) and, therefore, this element can be used as an alloying addition without a significant negative impact on corrosion. In fact, the average 1B 24-h weight loss for a Fe-35.8Al-5Cr heat (FA-328) was among the lowest of those measured and this same composition did exhibit the lowest average weight losses in the 2B salt environment (see Table 5). Additionally, FA-328 also was the most resistant of the Fe-35.8Al alloys in the atypical salt and showed the least sensitivity to changes in salt corrosivity (see Fig. 2). However, it must also be noted that the FA-328 alloy tended to have less initial surface roughness than several other iron aluminides (see Fig. 9) and thus should suffer a slightly less weight loss in terms of mass per unit surface area. Nevertheless, while the present data cannot be used to determine whether chromium has a definite beneficial effect on corrosion resistance of iron aluminide exposed to molten nitrate salt, it can be speculated that this element may promote the development of a thinner, more uniform aluminum-containing layer that is more effective in preventing the iron oxide from extensive reaction. This can possibly be accomplished by initial formation of a chromia-rich scale (that preferentially forms with respect to iron oxide) that is then dissolved and replaced with the slower growing aluminum surface product. Chromium has been observed to reduce the oxidation rate of Ni_3Al at 605°C[17] and to affect scale morphology.[18]

Under salt conditions where there was continued (refreshed) high concentrations of peroxide that somewhat simulate the MOLTOX™ chemical air separation process,[3,14] weight losses were linearly dependent on time (Fig. 6); thus, it is valid (and conservative) to use 24-h weight change data to determine corrosion rates. Such rates are listed in Table 9 for the various iron aluminides. Note that, for this aggressive type of environment, the best alloys have very low corrosion rates (<300 μm/year), which are substantially better than typical nickel-based alloys and stainless steel.[15] (Rates less than 800 μm/year are probably acceptable for the MOLTOX™ process since they are within the range controllable by economical process changes.)

There are several implications of this study for development of iron aluminides that are corrosion resistant in such nitrate salts. Based on a model of a thin, slowly dissolving

Table 9. Average corrosion rates based on linear weight loss kinetics

Heat No. (FA-)	Al concentration[a] (at. %)	Corrosion rate (μm/year)	
		1B	2B
76B	35	1200	4300
300	30	2000	
301	40	680	
303	40	500	
307	38	1800	
308	36.5		4700
310	36.5		1500
311	36.5		4300
312	36.5		1600
324	35.8	340	2700
325	35.8	210	360
326	35.8	70	320
328	35.8	260	150
350	35.8	540	1300
353	35.8	160	380
6040	40	830	

[a] See Table 1 for other constituents of alloy, if any.

$NaAlO_2$ that controls the corrosion process at a rate lower than that associated with iron species, minimum corrosion losses would be obtained when the surface coverage by this layer is maximized. This possibly could be accomplished by adding more aluminum or including an element (perhaps chromium) that promotes a faster or more uniform nucleation and growth of $NaAlO_2$ and/or improves the adherence of this layer to the underlying metal. A high-temperature preoxidation treatment in an oxidizing gaseous environment that results in the formation of an adherent Al_2O_3 scale on the iron aluminide should aid corrosion resistance under such conditions. However, alumina scales on Fe_3Al tend to spall[19] and alloying additions that promote sound, compact, more adherent oxide scales under high-temperature environments should also help in improving the corrosion resistance of iron aluminides in nitrate salt. Although the current results indicate that aluminum concentrations over 30 at. % do not result in greater corrosion resistance, it does appear prudent for an excess of aluminum to be available in case of localized breakdown of the $NaAlO_2$. Indeed, it would seem that the relatively large standard deviations associated with the determination of average weight changes may be associated with a tendency for nonuniform attack promoted by incomplete surface coverage of $NaAlO_2$. (Some pitting was observed on certain specimens and seemed to be related to galvanic effects due to the presence of the zirconium wire used to suspend the specimens in the salts. This may be a cause of the large standard deviations, but there was no strong correlation between the occurrence of pits on a specimen and its net weight loss.)

SUMMARY

The corrosion of iron-aluminum alloys by molten nitrate salt as a function of aluminum, chromium, and other minor elements was studied as part of an alloy design effort aimed at the development of a strong, ductile, corrosion-resistant FeAl type of aluminide. Short-term weight change data were used to examine the compositional dependence of corrosion upon exposure of iron aluminides to highly oxidizing nitrate salts at 650°C. Corrosion resistance was found to increase with increasing aluminum concentration of the alloy up to approximately 30 at. % Al. Chromium additions to the aluminide were not detrimental and may actually improve the corrosion behavior for certain aluminum concentrations. No effects of minor alloying additions (C, B, Ti, and Zr) could be determined. The best weight change results were obtained for a Fe-35.8 at. % Al aluminide containing some chromium. Based on linear weight loss kinetics, the weight change measurements for the most resistant compositions predict 650°C corrosion rates of 300 μm/year or less, which are substantially better than typical nickel-based alloys and stainless steels.

While weight change results are valuable in assessing relative corrosion resistance of different materials, such data do not directly lead to a mechanistic understanding of the corrosion process(es). Nevertheless, from a consideration of the weight changes; the microstructural, thermodynamic, and X-ray diffraction data; and the salt analyses, it appeared that the corrosion of iron aluminides by the molten nitrate salt proceeded by oxidation and a slow release of an aluminum-rich product into the salt. This rate is lower than that previously associated with iron and iron-based alloys. Such a controlling process would imply that corrosion losses of iron aluminides could be minimized by maximizing the surface coverage of this layer by alloying additions or by a preoxidation treatment.

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