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High-Temperature Corrosion Performance of FeAl-Based Alloys Containing Carbon in Molten Salt

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Abstract: Corrosion behavior of FeAl-based alloys containing carbon produced through arc melting in argon atmosphere has been studied at 500 °C to 700 °C. The samples were tested in the aggressive environment of molten salts (80%V₂O₅/20%Na₂SO₄). The corrosion behavior was observed by weight change method and the layer products formed were examined by using X-ray diffraction (XRD), scanning electron microscopy (SEM) and energy-dispersive spectroscopy (EDS). The different phase components were observed in the surface layer after the test in Fe-22Al alloy. A protective Al₂O₃ layer was confirmed for Fe-22Al alloy containing carbon only. However, an additional TiO layer was also observed in Fe-22Al alloy containing carbon with Ti addition. The microstructural and XRD examinations revealed that this additional TiO layer protects better against penetration of corrosive media. The corrosion resistance behavior of FeAl-based alloys were addressed on the basis of microstructural evidence.

Keywords: FeAl intermetallic alloys; high-temperature corrosion; SEM with EDS; XRD



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1. Introduction

Many industrial components of various engineering systems such as fuel sources (oil and coal) used at high temperature, power generation devices, aircraft gas turbines, fuel cells, etc. fail at high temperatures. The degradation of components occurred by accelerated attack due to condensation of films composed of molten salts. Transition metal aluminides such as Fe, Ti, Ni, etc. are potentially more suitable alternatives to superalloys at high temperatures with applications in aggressive environmental conditions [1–3]. These materials show excellent resistance to corrosion, oxidation, sulfidation, and carburizing atmospheres, even at high temperatures, due to the formation of stable alumina layer [2,4].

Among these, iron aluminides with higher Al content such as Fe₃Al (DO₃) and FeAl (B2) due to their low cost, are being possible substitute materials for high-temperature applications up to 1000 °C in aggressive environment [5–9]. However, iron aluminides have been poorly studied in the environment composed of (Na₂SO₄-V₂O₅) molten salt. In the past, Baligidad et al. reported that carbon additions have significantly improved the strength of Fe₃Al-based alloys attributed to precipitation hardening due to carbide and interstitial solid solution strengthening [10]. In the similar context, carbon addition to FeAl-based alloys was tried but graphite precipitation limits further development of these alloys [11]. Recently, it was reported that Ti addition together with carbon additions to

FeAl-based alloys has successfully developed FeAl-based alloys with no graphite precipitation [12]. Addition of Ti to iron aluminides containing carbon leads to improvement in the mechanical properties [12,13], but it has been established that Ti could also improve the oxidation and corrosion resistance [14,15]. Li et al. also found that Ti addition to Fe-36.5Al alloy leads to improvement of oxidation resistance due to compactness of oxide scales on the surface which deteriorate the further penetration of corrosive media [14,15].

Iron aluminide due to their better oxidation resistance at elevated temperature, are also considered as replacements of ferritic/austenitic steels for applications up to about 500 °C. Among iron aluminides, FeAl-based alloys with large amounts of Al (~37 wt.% or above) are generally considered potential candidate for protection at elevated temperature applications [15–17]. Ti additions to FeAl alloys with carbon to enhance its mechanical properties offers an excellent opportunity to improve its corrosion resistance especially supporting oxide adherence [14].

Unfortunately, the literature on the effect of alloying addition on FeAl-based alloys on its corrosion/oxidation behavior is limited. In the recent past, FeAl-based alloys were successfully developed with carbon additions and studied their mechanical and tribological behavior. However, these alloys are not studied for its corrosive behavior at high temperatures. Since these alloys show good corrosion and sulfidation resistance and therefore, it is necessary to study the behavior of these alloys in corrosive environment. The present work aims to study the effect of Ti alloying element on the corrosion behavior of FeAl alloys at high temperature.

2. Materials and Methods

Iron aluminides containing carbon with and without Ti addition alloys were prepared by arc melting under argon using pure iron (99.0%), pure aluminum (99.99%) and pure titanium (99.9%) powders. Four different alloys with composition (in wt%) Fe-22Al-0.1C (C010T), Fe-22Al-1C (C1T0), Fe-22Al-0.1-1Ti (C01T1) and Fe-22Al-1C-5Ti (C1T5) were produced. All the alloys were homogenized, and small pieces were cut from each alloy and grounded with emery paper of different grades and finally polished with fine alumina powder. The polished specimens were etched with reagent having composition of 33% CH₃COOH + 33% HNO₃ + 33% H₂O + 1% HF by volume. Microstructural examination was done using electron microscope (FEI Quanta 200 F, FEI, The Netherlands) and elemental analysis was done by using energy dispersion spectroscopy (EDS, FEI Quanta 200 F, FEI, The Netherlands). Small sections with size of 10 × 10 × 20 mm³ were cut from the disc using wire cut EDM for corrosion testing. The specimens were then polished with different grade emery papers to finish. Before oxidation tests, the specimens were cleaned with acetone and dried.

For the hot corrosion testing, the specimens were coated with a thin film of 80%V₂O₅/20%Na₂SO₄ salt solution and then exposed to the environment at the desired temperature. The specimens covered with the salt were isothermally heated in furnace, followed by cooling down to room temperature. The corrosion tests were performed at temperatures of 500, 600, and 700 °C for 70 h. During the experiment, the specimens were first weighed, put into an alumina crucible and then finally placed in the furnace at the desired temperature. After the regular interval of time, the specimen was left to cool down outside the furnace and then immediately reweighed. The change in the weight of the tested specimens were measured in an electronic balance with an accuracy of 0.1 mg. After the completion of 70 h study, the cross-sections of the oxidized surfaces were examined under scanning electron microscope (SU8010 Series, Hitachi, Japan) to study the mechanism of oxide film. EDS analysis was performed to analyze the distribution of various elements across the oxide and substrate interface in oxidized specimens.

3. Results and Discussions

In Figure 1a, C01T0 exhibits the fine carbides particles at the grain boundaries with coarse grain structure. In C1T0 alloy, an uneven distribution of coarse Fe₃AlC_{0.5} particles

with few graphite flakes are observed (Figure 1b). These $\text{Fe}_3\text{AlC}_{0.5}$ carbides are distributed along the grain boundaries as well as inside the grains. With Ti additions in C01T1 and C1T5 alloys, both TiC and $\text{Fe}_3\text{AlC}_{0.5}$ carbide particles are observed as shown in Figure 1c,d, respectively. No graphite formation is found after Ti additions to FeAl-based alloys. Both carbides are randomly distributed in the FeAl matrix, and their fractions increase with carbon and Ti contents.

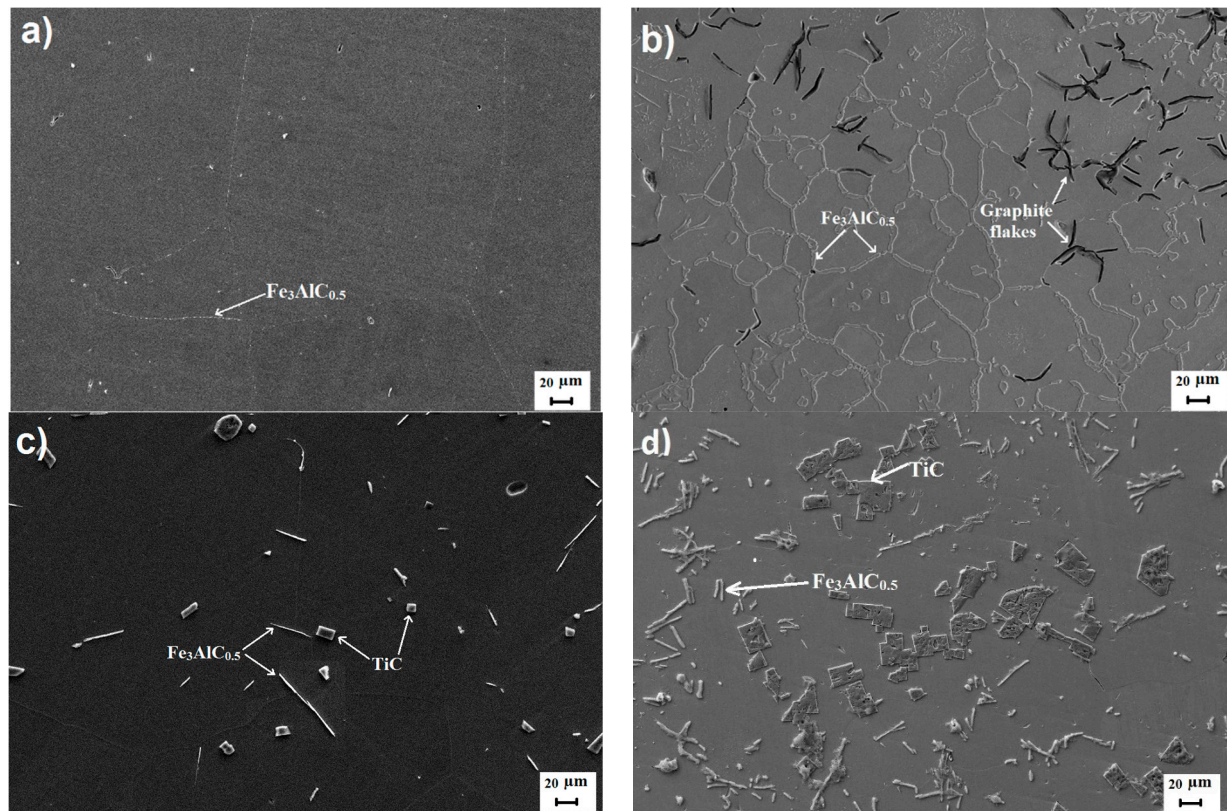


Figure 1. SEM images of FeAl-based alloys (a) C01T0 with fine $\text{Fe}_3\text{AlC}_{0.5}$, (b) C1T0 with graphite and coarse $\text{Fe}_3\text{AlC}_{0.5}$, (c) C01T1 with $\text{Fe}_3\text{AlC}_{0.5}$ and TiC and (d) C1T5 with $\text{Fe}_3\text{AlC}_{0.5}$ and TiC.

It has been found that carbon addition leads to $\text{Fe}_3\text{AlC}_{0.5}$ carbides and interstitial solid solution strengthening observed in EDS analysis (Figure 2a). Higher carbon addition causes graphite precipitation (Figure 1b). Incorporation of Ti eliminates the graphite precipitation as evident from the above microstructures (Figure 1d). Furthermore, Ti content was chosen such that the Ti:C ratios used for C01T1 and C1T5 were more than that for TiC formation. This is due to fact that some part of Ti dissolve in the FeAl matrix. EDS analysis of C1T5 alloy, confirms the presence of Ti in the matrix (Figure 2b). The confirmation of these carbide particles in C01T0 and C1T5 is done by EDS analysis and X-ray diffraction (XRD) (Panalytical's X'Pert Pro, Rigaku, Japan) which were reported elsewhere [12].

In quaternary Fe-Al-Ti-C system, TiC has large negative free energy of formation [18] and thus, TiC may be precipitate from the supersaturated solution. During solidification, both C and Ti may lose its solubility and therefore, Ti and C lead to formation of TiC particles. The reinforcing components in these alloys are formed through an in-situ process by solution precipitation or a solid-dissolving mechanism. It has been further observed that TiC bond strength is higher than C-C and Ti-Ti bonds. Thus, the graphite flakes in the alloy may reacts with Ti to form TiC and the residual C utilized to form $\text{Fe}_3\text{AlC}_{0.5}$ carbides. For higher Ti and C contents alloy (C1T5), TiC particles appear to be nucleated adjacent to existing TiC particles resulting agglomeration of TiC particles. This is called sympathetic nucleation and may lead to precipitate at the interphase boundary of already

existing precipitate with same phase. This is mainly attributed to lower activation energy barrier for precipitate–precipitate interface than precipitate–matrix interface [19].

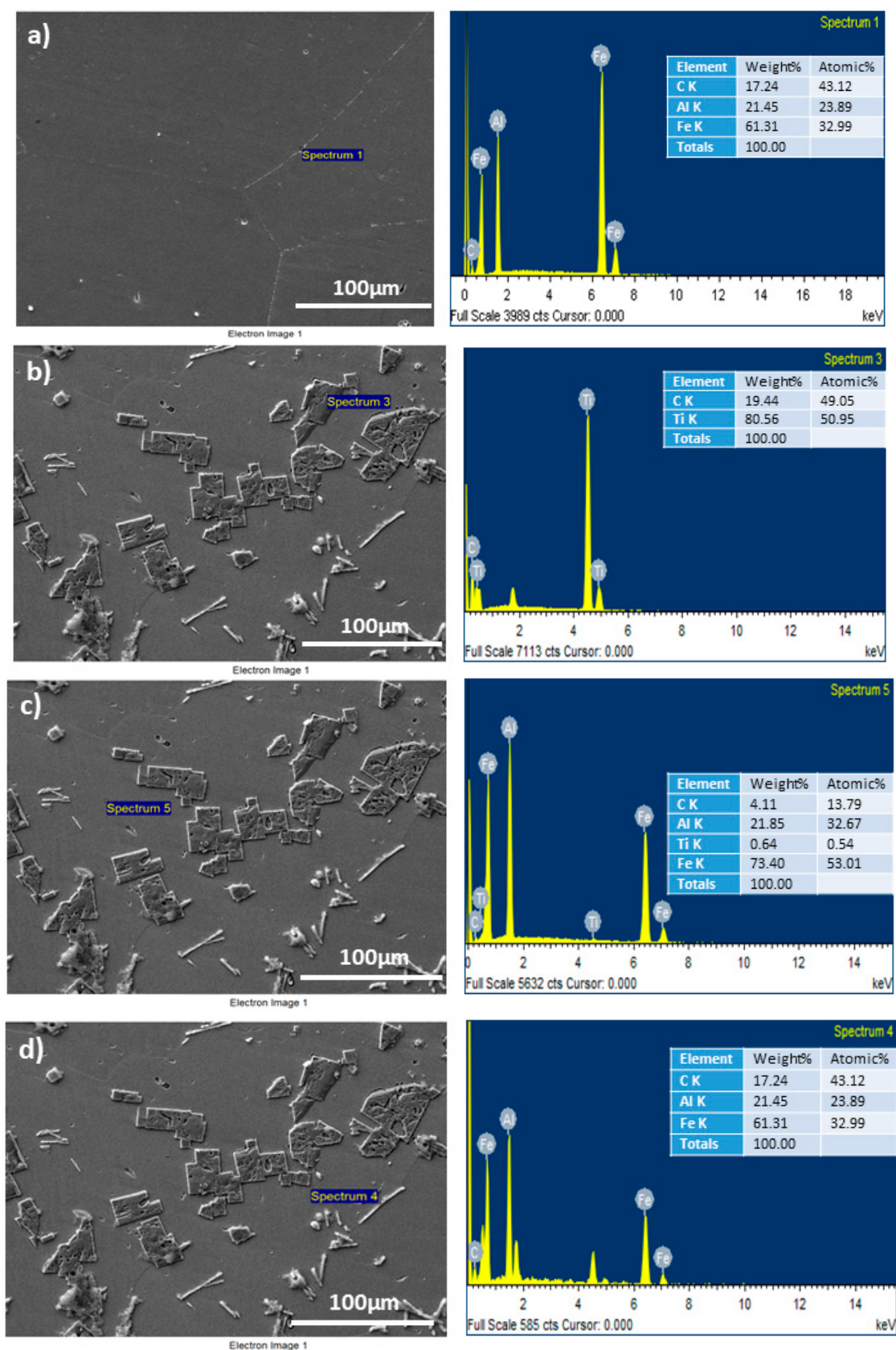


Figure 2. EDS analysis of (a) needle like $\text{Fe}_3\text{AlC}_{0.5}$ carbide in C01T0, (b) TiC, (c) matrix, and (d) needle like $\text{Fe}_3\text{AlC}_{0.5}$ carbide in C1T5.

The specific weight gains as a function of exposure time for FeAl alloys at temperatures of 500 °C, 600 °C, and 700 °C, respectively, are shown in Figure 3. From the figure, it is found that the mass gain increases parabolically with increasing exposure time for all temperatures. The deviations from the parabolic behavior initially corresponds to existence of growing alumina phase at the time during oxidation process. However, various types of behaviors such as exponential, linear, and parabolic behaviors have been noticed during the oxidation kinetics at high temperatures [20]. The parabolic nature of curve is the most commonly kinetics behavior of the layer products that can cover and shield the entire exposed surface of metallic counterpart [21]. The initial linear behavior of the kinetics due to the deposition of oxide layer product on the metallic surface and then reduced due to decrease in the exposed areas. The mass gain is significantly higher in C01T0 and C1T0 (without Ti) than that C01T1 and C1T5 (with Ti) at various temperatures. Among all of the alloys, C1T0 with $\text{Fe}_3\text{AlC}_{0.5}$ carbide shows highest weight gain. This is attributed to coarse and large number of $\text{Fe}_3\text{AlC}_{0.5}$ carbides as compared to C01T0. A. Raja et al. have also reported that the mass gain in iron aluminides was found to be increased with carbon content [22]. Isothermal mass gains were obtained at different temperatures and alloys acquired more mass gain at 700 °C than they did at other temperatures. However, both C01T1 and C1T5 (with Ti) showed lower weight gain than their corresponding alloys (C01T0 and C1T0). The increase in the slope was low for FeAl alloys containing Ti than their counterparts. This decrease in the value of slopes is mainly attributed to the present of small amount of Ti in the matrix. It resulted in the formation of additional TiO layer which causes the compactness of the oxidized layer and may decrease the further corrosion of the alloy. Based on the microstructural and compositional characterization of different alloys, the process of hot corrosion can be understood.

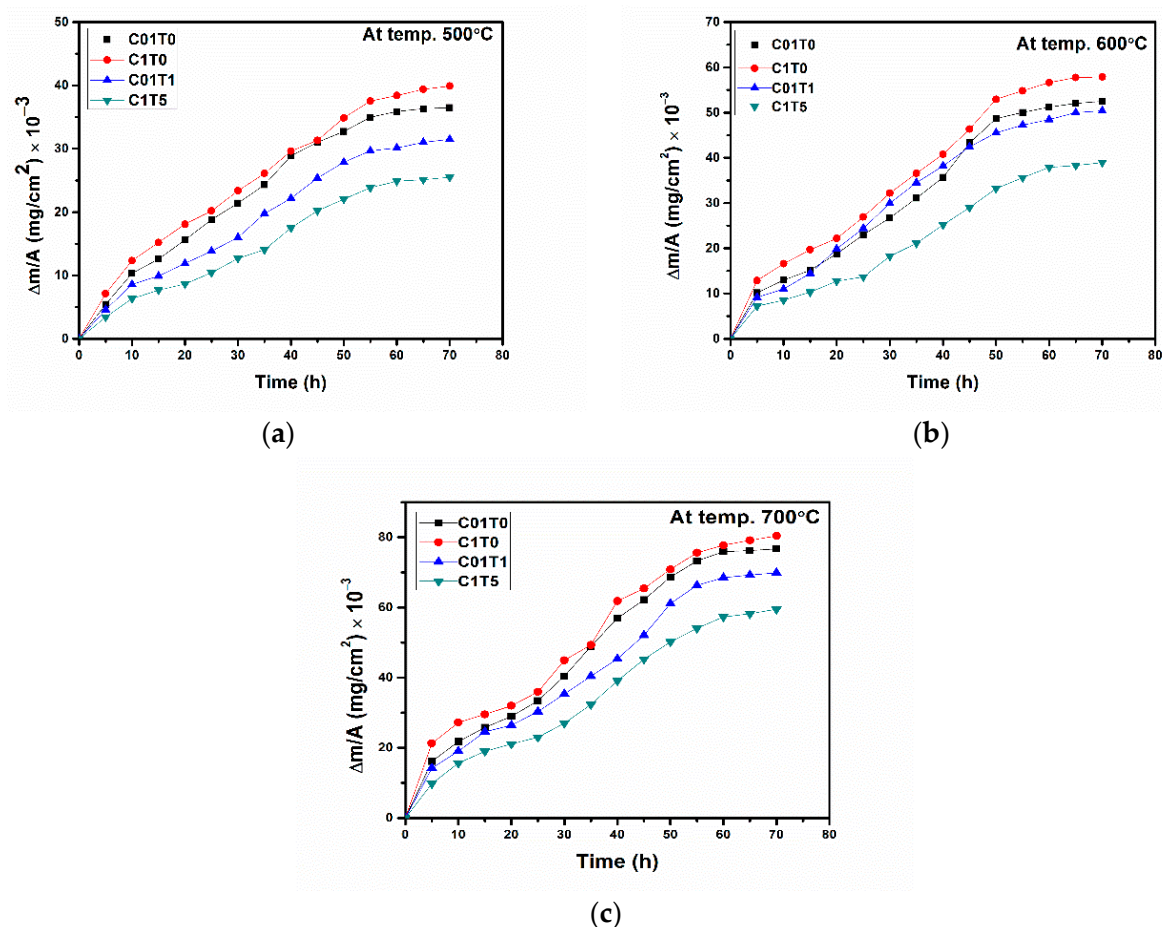


Figure 3. Variation of the weight gain of FeAl-based alloys with time at exposure of temperature (a) 500 °C, (b) 600 °C, and (c) 700 °C (lines joining the data points are for visual aid only).

Figure 4 shows the comparative weight gain of C01T0 and C1T5 at different temperatures. It is evident from the curve that the weight gain was found to be increased with increasing temperature. This increase in the weight gain of the alloys due to increase in the reaction rate and thus causes higher corrosion of the specimens. It can be seen that the corrosion rate increases with temperature, which indicates the diffusion rate is accelerated to produce the oxide-forming components. It has been found that the higher melting point of the salt may act as barrier to oxidation and limits the diffusion rate at lower temperature. This may lead to reduction of rate of reaction compared to that of metal with no Na_2SO_4 deposit [23]. In alloy C01T0, the oxide surface was composed of thin, adherent oxide-based Al_2O_3 layer, as evidenced by the cross-section view of the oxidized specimen. On the other hand, oxide surface of C1T5 alloy composed of dense adherent oxides with TiO in addition to Al_2O_3 layer. The formation of both Al_2O_3 and TiO oxides layers as oxidation products during hot corrosion, are in agreement with other hot corrosion studies of iron aluminides [14]. The better adhesion of complex oxide formed containing both aluminum and titanium (C1T5) may lead to higher oxidation resistance than pure Al_2O_3 layer as in case of C01T0 alloy. The cross-sectional view of the oxidized specimens further confirms that stable and adhered oxide layer was formed on the surface only. In C01T0 and C1T0, the layer was found to be Al-rich scale while in C01T1 and C1T5, an oxide layer of Ti-rich was formed in addition to Al-rich scale. This additional oxide layer containing Ti helps to improve the high-temperature oxidation resistance of C1T5 alloy. XRD analysis of oxide layer further confirms the formation of titanium oxide layer in C1T5 (Figure 5).

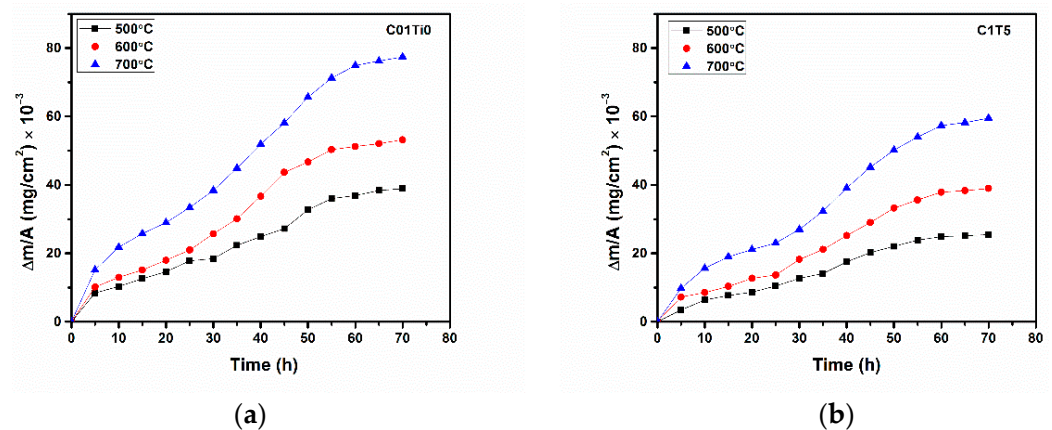


Figure 4. Comparative study showing the weight gain of (a) C01T0 and (b) C1T5 at different temperatures (Lines joining the data points are for visual aid only).

In molten salt (80% V_2O_5 /20% Na_2SO_4), the corrosion process takes place in two steps i.e., oxidation of the alloy, and then decomposition of the protective oxides deposits which can occur either by basic or acidic reactions by the molten salt [24]. The reaction occurs may result the formation of oxide film by consumption of oxide ions in the melt and may release the complex ions. Now, in alloy with no titanium, their hot corrosion resistance depends upon the formation of Al_2O_3 layer on the alloy surface only. However, with the presence of titanium in the alloy, there is an establishment of additional layer on the alloy surface. This would help to increase the compactness of the oxide layer and thus increases the corrosion resistance of the alloy. Further, more the aluminum (Al) or titanium (Ti) contents, thicker would be the oxide layer and thus, corrosion resistance of the alloy improved. For alloy without titanium i.e., C01T0 and C1T0 which forms only Al_2O_3 oxide layer, have lower compactness (Figure 6a) and may undergo dissolution of the oxide layer more rapidly at metal/melt interface and thus, leads to more weight gain. On the other hand, Ti-containing alloys more compact and adhere oxide layer is formed which reduces the dissolution rate and thus, decreases the rate of weight gain.

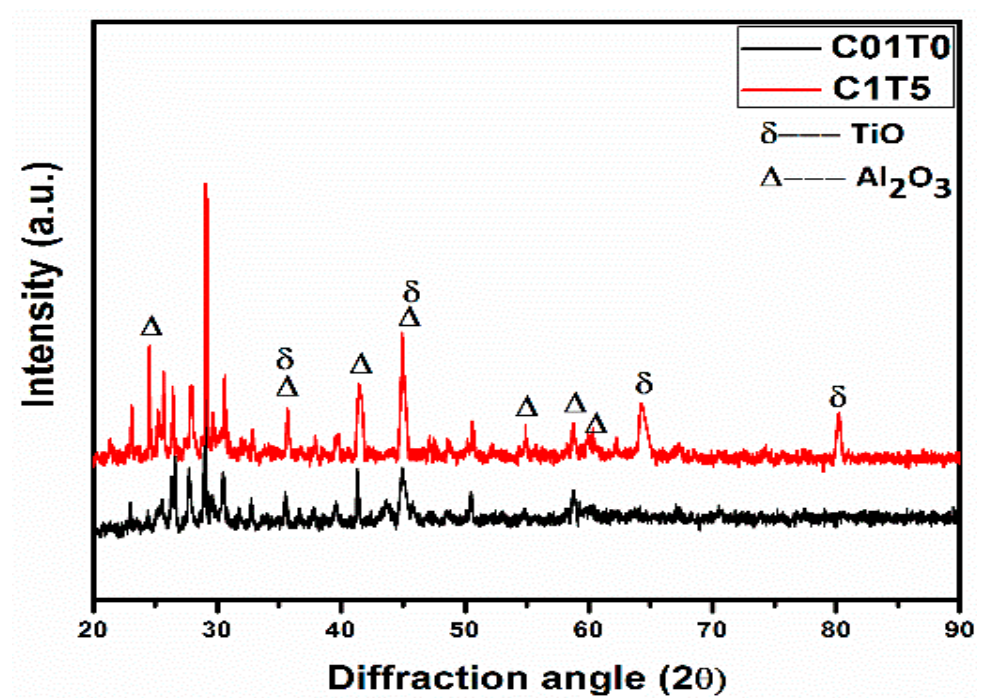


Figure 5. X-ray diffraction (XRD) patterns of oxidized samples.

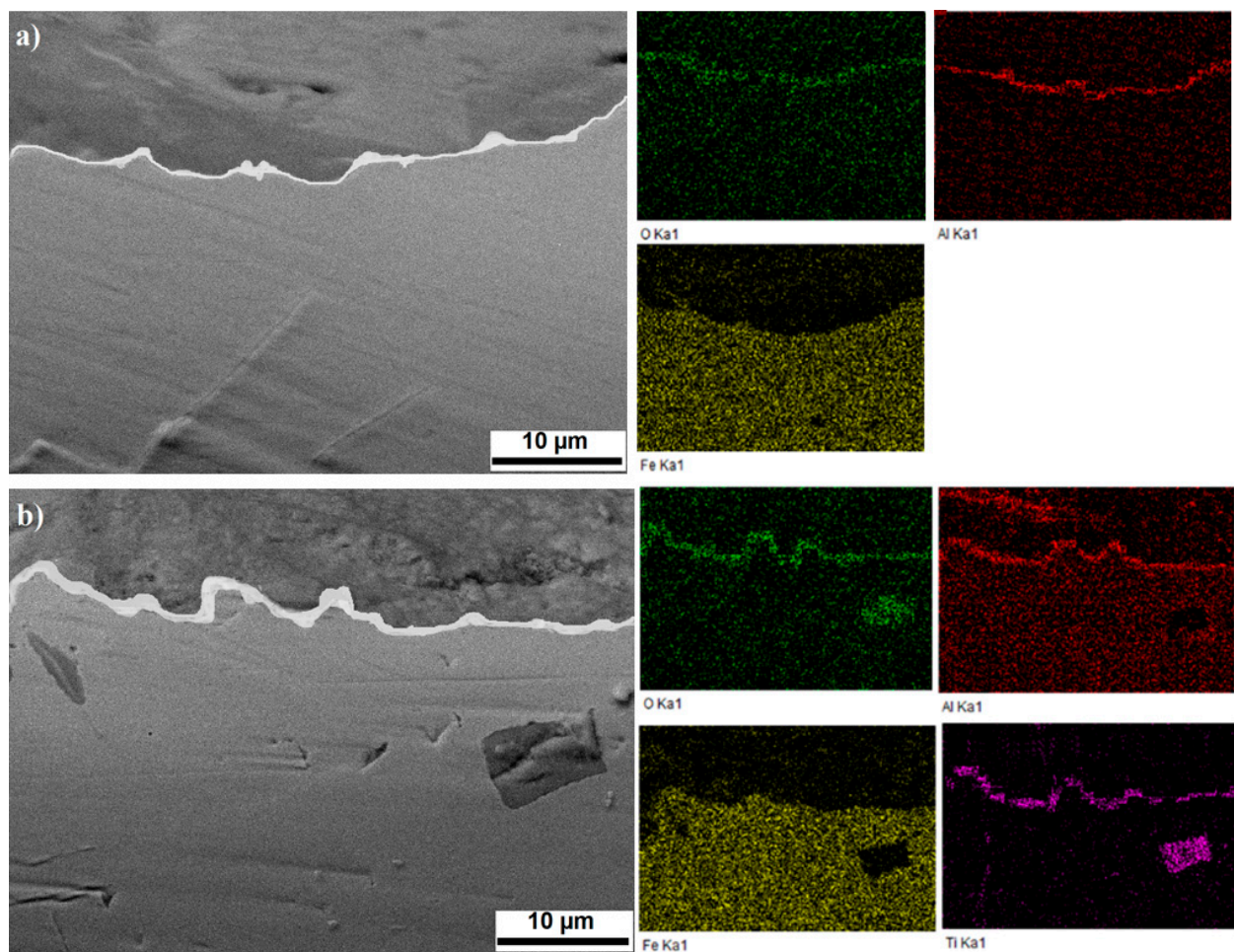


Figure 6. SEM Micrographs of (a) C01T0 and (b) C1T5 after oxidation at 700 °C /70 h and EDS elements analysis of cross-sections show the presence of Fe, O and Al in C01T0 and Fe, O, Ti and Al in C1T5.

The examination of the cross-sectional areas of oxidized specimen exposed the oxide layers were formed on the surface at different temperatures. The microstructures of cross-sectional areas show that no penetration to the interior of the specimen was observed. The further confirmation was done by the EDS mapping of element distribution and detailed study on the element analysis of the oxidized layer is shown in Figure 6. In both the samples, it can be observed that there is reduction in the oxygen content and associated drop in aluminum content during movement from layer to matrix. No iron content was observed in the formed layers and appeared only in the region immediately adjacent to the surface. This clearly indicates that the aluminum ions penetrate the nascent oxide layer, possibly accelerating the formation of Al_2O_3 layer in both the samples (C01T0 and C1T0). The formation of oxide layer is correlated using XRD diffraction pattern as shown in Figure 5. The unidentified peaks in the data may be related to Na_2SO_4 or complex compounds of V_2O_5 and Na_2SO_4 .

In C1T5 sample (Figure 6b), the oxide layer shows an extra element corresponding to titanium. At elevated temperature exposure of alloys to oxygen-bearing medium, the aluminum present in matrix is preferentially oxidized which leads to the formation of alumina (Al_2O_3) layer. But due to thermodynamically possibility, the presence of the iron in the matrix may also lead to formation of iron oxide (Fe_2O_3) layer. It has been found from the previous studies that major oxidized surface product forms between 500 and 700 °C has been reported to be $\alpha\text{-Al}_2\text{O}_3$ [20]. The EDS results also confirm the formation of Al_2O_3 layer in all the samples. This agrees with the observations of others, where it was found that in addition to Al_2O_3 oxides, as well as other metal oxides. Transient formation of aluminium oxides act as nucleation sites for Al_2O_3 , and titanium present in the matrix leads to the formation of more stable TiO layer. The dense concentration of titanium shows the present of the TiO oxide layer in addition to Al_2O_3 layer in C1T5 (Figure 6b). The formation of additional TiO layer was also observed in Fe-36.5Al-2Ti alloy as reported elsewhere [13]. This additional TiO layer leads to better corrosion resistance of Ti containing alloys (C01T1 and C1T5) as compared to without Ti alloy (C01T0 and C1T0) at elevated temperature in molten salt applications.

4. Conclusions

Addition of Ti to iron aluminide containing carbon leads to formation of carbides namely TiC and $\text{Fe}_3\text{AlC}_{0.5}$. Further, the incorporation of Ti is also found in FeAl matrix leads to refining in the grain of the alloy. The difference between alloys on Ti addition is not only the morphology of surface but also in phases present in the matrix. After corrosion test, only Al_2O_3 layer was observed on the surfaces of C01T0 and C1T0, while Ti addition leads to formation of an additional layer of TiO with Al_2O_3 oxides for C01T1 and C1T5, respectively. Thus, Ti addition to iron aluminides with carbon leads to significantly improvement in the compactness of oxide scales in the surface of FeAl-based alloys. The compactness of layer formed shows better protective effect. Further, the better adherence of TiO to the substrate of FeAl alloy makes more difficult of scale spalling. The high-temperature corrosion behavior of the FeAl-based alloys confirms that the Al_2O_3 and TiO oxide layers play significant role in the corrosion protection in corrosive environment at high-temperature applications.

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