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Abstract
Adopting a high-throughput combinatorial approach, a compositionally graded Ti–xAl (0 ≤ x ≤ 8 wt%) specimen was prepared to conduct a rapid systematic investigation of the influence of composition and exposure time on the oxidation performance of the titanium-rich section of the binary Ti–Al system. The compositionally graded specimen was solution heat treated and subjected to oxidation tests at 650 °C for different exposure times. The morphology, structure, and composition of the oxide scale as well as the microstructural changes in the base material were studied across the entire composition range, using a suite of characterization techniques. The observations revealed the presence of Al2O3 in the topmost layer of the oxide scale in addition to TiO2, indicating its early formation during oxidation. An increase in Al concentration improves the scaling rate of Ti; however, this is observed only for extended exposure times (i.e., 50 and 100 h), and a parabolic oxidation law is obeyed in the composition-time domain. The formation of the α2 phase (Ti3Al) also takes place for relatively higher Al contents (i.e., 8 wt%).

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Comments
On the Influence of Alloy Composition on Oxidation Performance and Oxygen-Induced Phase Transformations in Ti-(0-8)wt%Al Alloys

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Abstracts

Adopting a high-throughput combinatorial approach, a compositionally graded Ti-xAl (0 ≤ x ≤ 8 wt%) specimen was prepared to conduct a rapid systematic investigation of the influence of composition and exposure time on the oxidation performance of the titanium rich section of the binary Ti-Al system. The compositionally graded specimen was solution heat-treated and subject to oxidation tests at 650 °C for different exposure times. The morphology, structure and composition of the oxide scale as well as the microstructural changes in the base material were studied across the entire composition range, using a suite of characterization techniques. The observations revealed the presence of Al2O3 in the topmost layer of the oxide scale in addition to TiO2, indicating its early formation during oxidation. An increase in Al concentration improves the scaling rate of Ti; however, this is observed only for extended exposure times (i.e. 50 and 100 hrs) and a parabolic oxidation law is obeyed in the composition-time domain. The formation of the α2 phase (Ti3Al) also takes place for relatively higher Al contents (i.e. 8 wt%).
Introduction

When considering the high temperature oxidation resistance of titanium alloys, aluminum is considered to be the most important of the conventional alloying elements owing to its ability to form a thermodynamically stable and slow-growing oxide. The concentration of aluminum in most structural solute-lean titanium alloys does not exceed 6 wt% while for γ/α₂ titanium aluminides (based on the intermetallic compounds Ti₃Al and TiAl), the aluminum content can approach 50 at% which provides a protective Al₂O₃ oxide layer [1-4]. The formation of an aluminum oxide in the Ti-Al system can also be promoted by other alloying elements, e.g. Nb and Mo [5]. Importantly, the similar thermodynamic stability of aluminum and titanium oxides that is proportional to the activity of these elements complicates the operating mechanism governing the heterogeneous oxide scale formation during high temperature exposure [2].

There are two mechanisms by which Al strongly influences the high-temperature oxidation resistance of titanium. Firstly, Al results in the formation of aluminum oxide simultaneously with the titanium oxide. Second, Al changes the oxygen solubility of the metal substrate. It has long been established that the formation of a heterogeneous oxide scale (i.e. Al₂O₃+TiO₂) effectively reduces the flux of oxygen ions through the scale, thus improving the oxidation resistance. This improvement in oxidation resistance is due to the fact that the formation and growth of Al₂O₃ is proceeded by outward migration of metal ions, which is opposite to the oxidation of titanium [6, 7]. However, with regard to the oxygen solubility of the metal substrate there is a contradiction between the reported trends. The results of earlier research efforts implied that addition of Al increased the concentration of oxygen existing in solid solution from the total oxygen uptake [8-10]. However, more recent work reports an opposite behavior, i.e. Al reduces the solubility of oxygen in Ti during high temperature exposure [11].

It should be noted that any critical, fundamental assessment of oxygen ingress into alpha Ti as a function of composition in the Ti-Al system is viable only for low solute contents because of the ordering transformation of α to α₂ (Ti₃Al) that occurs for Al concentrations above ~7 wt% Al. In addition, the oxygen solubility of this intermetallic phase is considerably lower than that of the alpha phase. The presence of oxygen itself promotes α to α₂ ordering and leads to the stabilization of the intermetallic phase in the subsurface region [12-15].

As noted previously, due to the similar thermodynamic stabilities of aluminum and titanium oxides, the product of the oxidation reaction in Ti-Al system is a mixture of both oxides. However, the sequence of selective oxidation and order of the formation of the oxides in the multilayer scale varies as a function of parameters such as oxidation atmosphere, temperature, alloy composition and contamination. According to the results reported by Shida et al. [11], the oxide scales formed on Ti-Al alloys were
composed of TiO$_2$/Al$_2$O$_3$/TiO$_2$+Al$_2$O$_3$ moving from the scale surface towards the metal/oxide interface (at 900 °C with the Al content ranging from 20 to 50 wt%). The oxidation study of Ti-6242 at 700 °C, by Ebach-Stahl, showed that the oxide scale consists of (both TiO$_2$ and Al$_2$O$_3$ in the form of a non-layered structure [4].

While the majority of research efforts on the oxidation of Ti-Al system are focused on alloys with high solute levels (synonymous with titanium aluminides) and based on traditional weight gain/loss measurements, the aim of this study is to systematically assess the influence of Al concentration and the exposure time on the oxidation behavior of Ti-Al within the composition range associated with commercially available structural titanium alloys. To achieve this, Laser Engineered Net Shaping (LENS™) was employed to deposit a compositionally graded titanium-aluminum specimen (Ti-xAl; 0 ≤ x ≤ 8 wt%). Subsequently, a series of oxidation tests were conducted on the graded specimen at 650 °C. This combinatorial approach not only eliminates experimental variability regarding time, temperature, and atmosphere as the potential source of errors in oxidation studies, but also ensures that the identical testing conditions are maintained when comparing multiple tests. A suite of advanced characterization techniques has been used to assess, as a function of average local composition, the: operating oxidation mechanism; structure and composition of the oxide scale; depth of oxygen ingress; and microstructural evolution in the subsurface region.

This paper is a continuation of previously studied Ti-Mo and Ti-Cr systems [16, 17] under the same oxidation conditions as a part of an inclusive project on the oxidation assessment of binary Ti-X systems (X=selected transition metals). The use of these simple model alloys offers a better understanding of the role of composition on the oxidation performance of titanium and would provide insights for interpretation of the behavior of more complex Ti-based systems (i.e. commercially available alloys) at elevated temperatures.

**Experimental procedure:**

Laser Engineered Net Shaping (LENS™) technology, a rapid prototyping technique, was employed to produce a Ti-xAl compositionally graded specimen, with the composition range of 0 ≤ x ≤ 8 wt% Al, using an Optomec LENS™ 750 system at the University of North Texas. High purity metal powders of Ti (99.9% pure, -150 mesh from Alfa Aesar) and Al (99.8% pure, plasma spray grade from Micron Metals) were used for direct laser deposition and in-situ alloying. In order to produce 3D near-net shape metallic pieces by LENS™, a computer aided design (CAD) file is converted into a tool path file for the line-by-line and layer-by-layer deposition of blends of elemental powders.

Two independently controlled powder feeders allow for incremental changes in the composition of the deposited powders during the process. In order to achieve the intended composition gradient one of the
powder feeders was filled with pure Ti powder and the other one with Ti-8Al mechanically mixed elemental powders. An inert gas (here Ar) is used to carry the pre-defined quantities of powders from each powder feeder to the deposition box with a controlled atmosphere (the oxygen level is kept below 20ppm). A focused (~0.750mm in diameter) high power Nd-YAG laser beam delivers sufficient energy to create a small molten pool on a 6 mm thick Ti-6Al-4V substrate into which the fluidized powders from the two powder feeders are injected via four converging copper nozzles. The substrate is fixed onto a computer-controlled motorized stage with in-plane (in x and y direction) motion that is coupled with the vertical motion of the deposition head containing the laser focusing lens and the copper nozzles. The final product is a 38 mm × 25 mm × 12 mm rectilinear solid composed of 100 layers with the nominal thickness of 0.25 mm. The composition is varied by 1 wt% after deposition of each 11th layer (i.e., 11, 22, 33, etc).

The compositionally graded specimen was longitudinally sectioned into several pieces, thus preserving the composition gradient in each piece. The solution heat treatment was conducted using a tube furnace at 1050 °C, a temperature above β transus temperature for the entire composition range, for 30 min followed by water quenching. To minimize the oxidation reaction the samples were wrapped in Ti foil (99.7% pure) and under a continuous flow of Ar gas during the solution heat treatment.

The solutionized samples were ground successively through 120–800 grit SiC abrasive papers and fine polished using 0.04 µm colloidal silica suspension. To assure that the oxidation tests start with a fresh metal surface, the samples were cleaned in a multistep cleaning process including acetone, water + surfactant and methanol. The oxidation tests were carried out isothermally at 650 °C for three selected exposure times: 25-, 50- and 100-hrs. The samples were placed in a box furnace with the polished and cleaned surfaces oriented upward and exposed to still-air atmosphere.

After the oxidation tests the samples were sectioned perpendicular to the oxidized surface, polished and cleaned for the subsequent detail characterization of the resulting oxide structure and microstructure. The specimens were characterized using an FEI Nova NanoSEM 230 field-emission gun scanning electron microscope (SEM) as well as an FEI™ Tecnai G2 F20 transmission electron microscope (TEM) both equipped with energy dispersive spectroscopy (EDS) detector. Site-specific TEM sample preparation was carried out using an FEI DualBeam (FIB/SEM) Nova 200NanoLab.

Results and discussion:

Figures 1(a–c) show SEM backscattered electron (BSE) micrographs of the bulk microstructure of three selected locations with nominal compositions of Ti-3Al, Ti-6Al and Ti-8Al from the compositionally graded Ti-xAl specimen after solutionizing at 1050 °C for 30 min followed by water quenching. This specimen and these images represent the starting microstructure for any subsequent
oxidation reaction and the possible associated phase transformations. It is expected that the $\beta \rightarrow \alpha'$ (hcp martensite) phase transformation takes place for the entire composition range embedded in the graded specimen due to the fact that $\alpha$ stabilizing effect of Al increases martensite-start temperature ($M_S$) of Ti thus, formation of $\alpha'$ martensite cannot be suppressed and a massive martensite morphology is adopted upon quenching [18, 19]. As an example Jepson et al. reported that the $M_S$ for a Ti-2.9Al alloy is 918 °C while addition of Al up to 9 wt% increases $M_S$ to 1015 °C [20].

The solutionized and water quenched samples were subject to oxidation tests at 650 °C and the electron backscattered micrographs of the cross-sections of Ti-3Al, Ti-6Al and Ti-9Al coupons after 25 and 100 hrs oxidation are presented in Figs. 2(a-f). Given the large difference between the atomic weights of the metal substrate and the oxide scale, the latter is not visible in the micrographs where the image settings (i.e. brightness and contrast) are adjusted to resolve the microstructural features in the metal substrate. It is immediately apparent that the microstructure is, as expected, devoid of the $\beta$ phase owing to the lack of $\beta$ stabilizing elements and consists of relatively large equiaxed grains. There exist a noticeable difference in the response of the microstructure to polishing progressing from the bottom of the graded specimen to the top (corresponding to the lower and higher contents of Al respectively). Although detailed TEM analysis is required to determine the crystal structure of the equiaxed grains beneath the oxidized surface, the spatially (and hence compositionally) dissimilar response to the polishing process, suggests the subsurface phase evolution has led to the formation of a new phase (or phases). It can also be seen that for the Ti-3Al component (Fig 2(a)) the equiaxed grains are slightly etched during polishing, while it does not happen to Ti-6Al and Ti-9Al components. As the oxidation time is extended to 100 hr the morphologies remain more or less unchanged for identical compositions (see Fig. 2(d-f)). There appears a narrow band just below the metal/oxide interface that is characterized by a smooth surface which due to the formation of round edges during the cross section polishing of the oxidized surface.

A many-beam bright-field TEM and a STEM micrograph of a Ti-8Al composition following the 50hr oxidation at 650°C are shown in Figs. 3(a,b). The TEM sample contains both the oxide scale and the metal substrate. Based on the local structure, the oxide scale can be divided into three layers parallel to the metal/oxide interface: the outermost layer exhibiting a porous structure (L1); the relatively dense layer in the middle (L2); and the thin porous layer in contact with the metal substrate (L3). The STEM-EDS concentration profile of Al (see Fig. 3(c)) revealed that unlike L2 and L3, the topmost layer (L1) consists of Al rich regions. To further study the composition of the scale layers, distribution of the elements present was mapped and the results are shown in Fig. 4. Captured in the elemental maps are the protective Pt layer, L1 and the top portion of L2. It is clearly evident that the Al-rich phase ($Al_2O_3$) is uniformly
distributed across L1 along with Ti oxide, thus indicating the formation of a heterogeneous oxide layer while L2 and L3 consist predominately of TiO$_2$.

As noted, the operating oxidation mechanism that leads to the formation of a multilayer and heterogeneous oxide scale in Ti-Al systems is rather complex due to the similar thermodynamic stability of TiO$_2$ and Al$_2$O$_3$ oxides (directly proportional to the activity of Ti and Al) and progressive alteration of ionic transportation balance which is associated with the phase fraction of these oxides [2]. On the other hand the extent of oxygen ingress and subsequent stabilization of intermetallic phases in the metal substrate just below the metal/oxide interface (which will be discussed later in this paper) causes a large deviation from ideal Raultian behavior which describes a linear relation between chemistry and the activity of the constituent. According to the thermodynamic calculations, the oxidation of Ti is more favored for Al concentrations below 50 at% Al in Ti-Al system [1]. However there is contradiction regarding the relative stabilities of Ti and Al oxides between experimental measurements and thermodynamic calculations, which is believed to arise from the differences in oxygen solubility of elemental Ti compared to Ti-Al intermetallic phases [1, 2, 15, 21-26]. Regardless of the phase evolution in the substrate, the selective oxidation of Ti and attending rejection of Al atoms causes a pileup ahead of the oxidation front (i.e., the metal/oxide interface) until the concentration of Al is sufficiently high to trigger the oxidation of Al. Therefore a multilayer and heterogeneous oxide scale forms with the outermost layer composed of only TiO$_2$, as is repeatedly mentioned in the literatures for Ti-Al systems [8, 11, 27-29]. The order of formation of these alternate layers in the oxide scale is governed by several factors such as the initial composition of the base metal, oxidizing atmosphere, oxidation time and temperature [4, 5, 30].

It should be noted that TiO$_2$ is a non-stoichiometric n-type oxide in which the non-metal deficit (oxygen vacancy) is the predominant defect structure. Therefore, the inward diffusion of oxygen ions is favored and the metal/oxide interface is considered to be the oxidation front. The oxide of Al (Al$_2$O$_3$), on the other hand, is a metal deficit p-type oxide that induces the outward migration of metal ions. Considering these characteristics of the oxides, the balance of inward/outward mass transportation during oxidation of Ti is affected by the addition of Al, further adding to the complexity of oxidation reaction in this system. For instance, for the case of pure Ti, 90% of the oxidation reaction takes place by the inward diffusion of metal ions through TiO$_2$ scale while for TiAl (γ) this portion is reduced to 70% [6, 7].

Figure 5 shows bright-field TEM and STEM micrographs as well as the associated concentration maps for Ti and Al for a Ti-3Al component after 50 hr oxidation at 650 °C. Similar to the Ti-8Al composition, Al$_2$O$_3$ is present in the outermost oxide layer mixed with TiO$_2$. The rest of the oxide scale consists of only TiO$_2$. The top portion of the oxide layer however, is less porous when compared to the case of Ti-8Al due to the lower phase fraction of Al$_2$O$_3$, thus allowing for the sintering and grain growth of TiO$_2$ crystals.
during extended oxidation exposure. The mechanism by which the mixed (TiO₂+Al₂O₃) outermost oxide layer forms cannot be unequivocally identified and requires monitoring of the scale composition during the oxidation reaction. However, since the mixed layer is separated from the metal/oxide interface by a relatively thick TiO₂ layer that favors the inward diffusion of oxygen rather than outward migration of metal ions, it is possible that the mixed oxide layer is the product that forms during the initial development stages of the oxide scale.

The phase evolution associated with the oxidation reaction in the subsurface region ahead of the oxidation front plays a crucial role in the kinetics of reaction. The phase transformation, induced by the ingress of oxygen, causes variation in the partitioning coefficient of alloying elements between the existing phases, stabilization of particular phase or precipitation of a new phase [16, 17]. When oxidation is accompanied by the formation of intermetallic phases in the metal substrate, as in the case of Ti-Cr and Ti-Al systems, there will be a marked change in the oxygen solubility of the alloy that can ultimately modify the oxidation rate law. Therefore, the high temperature oxidation behavior of the system cannot be simply explained by a single rate law such as either parabolic or linear rate laws [8, 17, 31, 32].

Selected area diffraction (SAD) patterns recorded from the regions just below the metal/oxide interface of the Ti-3Al and Ti-8Al components are presented and compared in Figs. 6(a-d). The selected area diffraction patterns for Ti-3Al (Fig. 6(a,b)) can be consistently indexed as [0 1 1 1] and [2 1 1 0] zone axes of hcp α Ti. For Ti-8Al however, superlattice spots are present in addition to the principle reflections which indicates that the SAD patterns belong to the Ti₃Al intermetallic compound with the hcp structure of DO₁₉ type.

If the total equivalent-Al content in Ti-Al-O system at 650 °C exceed ~5.5wt%, the α → Ti₃Al transformation is possible. This can be inferred from the isothermal section of Ti-Al-O ternary phase diagram generated using the PANDAT software (see Fig 7). PANDAT works on the basis of CALPHAD (CALculation of PHAse Diagram) approach [33] for the calculation of phase diagram in multi-component systems which makes use of PanTitanium database based on multiple studies of microstructure and phase equilibria in Ti-based systems [34]. This isotherm is consistent with experimentally observed higher temperature isotherms [35], as well as observations made in the literature [13]. According to the Al equivalency equation [36], the role of oxygen in this phase transformation is profound which arises from the fact that oxygen reduces the solubility of Al in the α phase and facilitates the formation of Ti₃Al.

From the thermodynamic calculations (Fig 7), the α → Ti₃Al transformation for the entire composition range represented in the graded specimen is attainable depending upon the level of oxygen enrichment near the oxidized surface. Interestingly, the formation of Ti₃Al just below the metal/oxide interface (due to Al rejection from the α phase) improves the oxidation resistance of low concentration Ti-Al
components. This same phenomenon can also occur in γ-titanium aluminides (TiAl) as a result of Al depletion in the metal substrate where the formation of Al₂O₃ is more favored, thus degrading the oxidation performance of these alloys [11, 37, 38].

The oxidation reaction proceeds as a multi-step process including both oxygen dissolution into the base metal and the formation of an oxide scale, each of which should be considered independently. The measurement of the thickness of the oxide scale is a far more useful metric for the assessment of the scaling resistance when compared to the conventional technique of total weight gain which is the summation of the oxygen dissolved in the metal substrate and the oxygen consumed in the formation of oxide scale, requiring a differentiation of the total weight gain to these two processes when drawing meaningful conclusions. This is due to the fact that higher oxygen uptake does not necessarily represent higher scaling rate. Indeed, in some cases they can be inversely proportional [8, 39]. To study the scaling behavior of Ti-Al system, the scale thickness was plotted as a function of Al content across the composition range of the graded specimen for the three oxidation times (see Fig. 8(a)) accompanied by the secondary electron micrographs corresponding to three data points in the scattered plots, showing the metal substrate and the oxide scale together (see Fig. 8(b-d)). The values are the average of six independent measurements for each composition. It was observed that for the 25hr exposure time there was not an obvious influence of composition on the scaling rate and that the thickness of the oxide does not change considerably across the studied composition range while for the longer exposure times (50 and 100 hrs) the scale thickness decreases progressively with increase in Al content. For compositions less than 4 wt% Al, there is a jump in scale thickness when the oxidation time is increased from 25 to 50hr while the passage of time from 50 to 100 hr did not have a marked influence. This is consistent with parabolic oxidation rate which is considered as a general rate law for the oxidation of Ti alloys at 650 °C [8, 40]. There is also an unusual decrease in thickness after 100 hr for compositions ≥ 5 wt% Al which could have been caused by local spallation and regrowth of the scale during isothermal oxidation.

The oxidation observations of Ti-Al system at 650 °C, with regard to the structure and composition of both the oxide scale and the metal substrate, can be schematically summarized as a function of Al concentration (Fig. 9). It should be emphasized that the presented schematic diagram is valid for the composition-time domain studied in this research work.

**Conclusions**

The following salient conclusions have been made from the combinatorial assessment of the influence of Al content on the oxidation behavior of Ti-Al system at 650 °C:
1) The formation of $\alpha_2$ phase ($\text{Ti}_3\text{Al}$) in the subsurface region of the oxidized Ti-Al alloys for relatively higher concentration of Al (e.g. 8 wt%) was observed, resulting from a combined effect of composition and oxygen enrichment during high temperature exposure.

2) Despite the low solute levels, the topmost layer of the oxide scale is a mixture of $\text{TiO}_2$ and $\text{Al}_2\text{O}_3$ which implies on the formation of Al oxide in the early stages of the oxidation reaction.

3) For Al contents below ~4 wt%, extended exposure times (i.e. 50 and 100 hrs) reveal the influence of composition on the scaling rate and the observations are consistent with the parabolic oxidation rate law for Ti-Al system in the studied composition-time domain.

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Fig. 1. Backscattered electron micrographs of a) Ti-3Al, b) Ti-6Al and c) Ti-8Al compositions after solution heat-treatment at 1050 °C for 30 min followed by water quenching.
Fig. 2. a-f) Backscattered electron micrographs from the region just below the oxidized surface of Ti-3Al, Ti-6Al and Ti-8Al compositions after 25 and 100 hrs oxidation at 650 °C.
Fig. 3. a,b) Bright-field TEM and HAADF STEM micrographs of a Ti-8Al composition after 50 hr oxidation at 650 °C. c) The corresponding STEM-EDS Al concentration profile.
Fig. 4. The STEM-EDS elementals maps showing the distribution of Ti, Al, O and Pt.
Fig. 5. Bright-field and STEM micrographs of a Ti-3Al composition after 50 hr oxidation at 650 °C, as well as the corresponding STEM-EDS concentration maps for Ti and Al.
Fig. 6. The SAD patterns recorded from the subsurface region of a,b) Ti-3Al and c,d) Ti-8Al compositions after 50 hr oxidation at 650 °C.
Fig. 7. Partial isothermal section of the Ti-Al-O phase diagram at 650 °C generated by PANDAT 8.1 software.
Fig. 8. a) Scale thickness versus Al wt% for binary Ti-Al system oxidized for 25, 50 and 100hrs at 650 °C. b-d) The secondary electron micrographs corresponding to three data points in the scattered plots.
Fig. 9. Schematic diagram of the oxidation of Ti-Al system as a function of Al content at 650 °C.