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# Oxidation behavior of binary Ti-xW ( $0 \le x \le 30$ , wt%) alloys at 650 °C as a function of W concentration



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#### 1. Introduction

The relatively low oxidation performance and subsequent surface embrittlement rank high among the most important life-limiting characteristics for structural Ti-based alloys at elevated temperatures. The oxidation behavior of Ti-based alloys is influenced by several factors involving both the material (e.g., composition) and the exposure conditions (e.g., time, temperature, atmosphere). To interpret the overall oxidation performance of a material system it is necessary to understand the role of individual constituents, both qualitatively and quantitatively on the different stages of oxidation. The prospect of understanding and predicting oxidation behavior of multi-component Ti-based alloys is rather daunting, given the complex nature of oxidation reactions and the numerous operating mechanisms in multi-component systems. Any prediction must be made on an understanding of the operating mechanisms for, firstly, oxidation behavior in binary systems and, secondly, enhancing/obstructing tendencies of the binary elemental species in the presence of a third element. Thus, an experimental approach has been adopted that maximizes throughput to probe the effect of alloy composition on oxidation behavior while simultaneously holding other variables (e.g., temperature, time, atmosphere) precisely constant [1-3].

### ABSTRACT

The role of composition on the oxidation behavior of the Ti-W system at 650 °C was investigated utilizing a compositionally graded specimen, Ti-xW ( $0 \le x \le 30$ , all compositions in wt%). Microstructural evolution of the base material and thickness of the oxide layers were assessed as a function of composition. Some of the observations with regard to the evolved base metal microstructure, including:  $\alpha$  lath and  $\beta$  rib/precipitates and lamellar structure, were found to be comparable to what is reported for Ti-Mo system. It was also shown that formation of a thinner oxide does not necessarily imply on the lower oxygen ingress.

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Although the assessment of oxidation for Ti and simple Tibased systems have been extensively documented, the majority of the efforts are focused on the oxidation behavior of complex technical alloys (e.g.  $\beta$ -21s, Ti-6Al-4V and Ti-aluminides) rather than a systematic exploration of composition space, particularly for the elements that are not common alloying elements for titanium (e.g. W) [4–17]. This dearth of binary and ternary systems makes the interpretation of compositionally-mediated operating oxidation mechanisms and near-surface microstructural evolution nearly impossible.

To date, W has not been an elemental constituent to any technically important commercially used Ti-based alloy. This is due to two characteristics, namely its very high density (particularly avoided in aerospace applications) and the difficulties associated with segregation issues in casting or the partial reaction of W particles in powder metallurgy [18,19]. Despite the traditional absence of this element in Ti alloys, its influence on the oxidation behavior of Ti aluminide alloys have been investigated in some research efforts [20-22]. According to Shida et al. [20], for a mixed TiO<sub>2</sub> + Al<sub>2</sub>O<sub>3</sub> oxide scale formed on a Ti-35.4 wt% Al alloy (containing 1-6 wt% W) during oxidation at 900 °C, W segregated into the Ti oxide and led to a slower growth rate for this phase. It was also reported that internal oxidation was prevented in W containing alloys as a result of significant reduction in the oxygen solubility in the intermetallic TiAl systems [20]. The critical concentration of Al required for the formation of a protective Al<sub>2</sub>O<sub>3</sub> oxide layer in Ti-Al alloys can be reduced by addition W as it increases the activity of Al and acts as sintering aid for the formation of Al oxide [1,20,21].



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The purpose of this study is to assess the influence of the concentration of W (across a wide composition range) on the oxidation properties of Ti-xW binary compositions at 650 °C using a compositionally graded binary Ti-xW ( $0 \le x \le 30$  wt%) specimen. The Laser Engineered Net Shaping (LENS<sup>TM</sup>) was employed to achieve this goal. The advantage of such a systematic approach is to maintain identical testing conditions across the composition range and to avoid potential experimental variability regarding time, temperature and atmosphere. Both the oxide scale and the metal substrate were characterized using advanced characterization techniques to assess different aspects of oxidation and oxygen-mediated reactions (e.g., phase transformations) as a function of local average composition. Although this paper is focused on the influence of composition on the oxidation behavior of Ti-W system, where relevant the evolution of the bulk microstructure will also be discussed relative to the parent microstructures.

Such a combinatorial approach and utilization of simple binary systems provide mechanistic insights for the subsequent understanding of the oxidation behavior of more complex Ti-based alloys (i.e. commercially available alloys). The present work is part of a more inclusive project on the oxidation assessment of other binary Ti-X systems (X = selected alloying element) including Ti-Mo, Ti-Cr and Ti-Al [23–26], though correlations with industrially used alloys has been drawn [27]. Briefly, these three systems were selected because the alloying elements Mo, Cr, and Al represent the three distinctive types of elements in Ti-based alloys, namely a monotectoid  $\beta$ -stabilizer (Mo), a eutectoid  $\beta$ -stabilizer (Cr), and an  $\alpha$ -stabilizer (Al). Similar to Mo, W is a monotectoid  $\beta$ -stabilizer, and it is thus reasonable to expect some common observations with Mo, especially when considering the subsurface microstructural evolution.

## 2. Experimental methods

A compositionally graded Ti-xW ( $0 \le x \le 30$  wt%) specimen was produced using an Optomec LENS<sup>TM</sup> 750 at the University of North Texas from high purity elemental metal powders of Ti (99.9% pure, -150 mesh from Alfa Aesar) and W (99.8% pure, plasma spray grade from Micron Metals). The LENS<sup>TM</sup> technology uses a computeraided design (e.g. CAD) file from which a machine code tool path can be generated for the subsequent laser deposition of threedimensional components. The CAD file is first translated into a \*.stl file, and subsequently sliced into a sequence of layers with a nominal thickness of 0.25 mm. Each layer consists of multiple parallel lines with a nominal hatch width of 0.38 mm. The tool path information that is generated with these variables is communicated to the computer-controlled motorized stage and a deposition head (consist of focusing lens and powder nozzles). The 2D (x,y) inplane motion of the stage accompanied by -z vertical motion of the deposition head produce near-net-shape metallic pieces.

The LENS<sup>TM</sup> is equipped with two independently controlled powder feeders which were loaded with pure Ti powder in powder feeder #1 and with a Ti–30 W mechanically mixed elemental powder blend in powder feeder #2. An inert gas (here Ar) carries the powders from powder feeders into a controlled atmosphere box. The fluidized powders are injected (via four convergent Cu nozzles) into a localized melt pool created by a focused high energy Nd:YAG laser (350–500 W). A 6 mm thick Ti-6Al-4V substrate was used as the base for laser deposition of the powder blend and in-situ alloying.

The oxygen level was maintained at a level below 20 ppm throughout the deposition and the specimen was deposited in the form of a  $38 \text{ mm} \times 25 \text{ mm} \times 12 \text{ mm}$  rectilinear solid. The independent computer control of the powder flow rate allows for pre-programmed incremental changes in the relative mass flow rate from powder feeders and consequently variation in the local composition along the length of the sample. The final product produced for this research effort was a compositionally graded Ti-xW specimen where x ranges between 0 and 30 wt%.

The specimen was longitudinally sectioned into several pieces (thus conserving the composition range in every piece, see Fig. 1(a)) and subject to a  $\beta$  solution heat treatment at 975 °C for 100 h followed by water quenching. The solutionization temperature was selected to be well above the beta transus for every composition along the gradient. The time was selected to allow for sufficient diffusion of W, a notoriously slow diffusing element in Ti, and the water quench was conducted to allow for a more fundamental comparison among different binary titanium systems. To minimize oxidation, the samples were encapsulated in an evacuated and Ar back-filled quartz tubes. In addition, Ar was flowing constantly through the tube furnace during the solution heat-treatment (see Fig. 1(b)). Several pieces of titanium sponge were also placed in the quartz tube to getter up the residual oxygen and further protect the specimen from oxidation during extended solution heat treatment. Following solutionization, the samples were polished prior to the oxidation tests to assure that the exposed metal surface was flat and uniform. The samples preparation steps included grinding using 240-800 grit SiC abrasive papers followed by polishing with 0.04 colloidal silica suspension. Cleaning of the samples following grinding and polishing was carried out using a sequence of solutions starting with acetone, followed by water+surfactant, and finally with methanol.

The polished and cleaned samples were oxidized at 650 °C for three different holding times of 25, 50 and 100 h. The oxidation tests were carried out in a box furnace and the samples were placed with the polished surface oriented upward and exposed to still laboratory-air. The samples were cross-sectioned after oxidation tests and the cross section of the oxidized surface was polished following the aforementioned steps.



Fig. 1. a) Schematic of the compositionally graded Ti-xW specimen and b) solution heat treatment setup.

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