Oxidation behaviour of Ti6Al4V and titanium based-matrix composites (TiC/Ti6Al4V and TiB/Ti6Al4V)

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Abstract. This study explores the oxidation behaviour of Ti64 and titanium-based matrix composites, specifically TiC/Ti64 and TiB/Ti64. Through experimental analysis for oxidation tests using the Ultra furnace, the oxidation characteristics were examined to understand the behaviour of these materials under elevated temperatures (400ºC, 500ºC and 600ºC for 6 hours). Scanning Electron Microscopy (SEM) was used to examine the as-built and after-oxidation microstructures. X-ray Diffraction (XRD) was used to identify phases present before and after oxidation. At 400°C, all materials (Ti64, TiC/Ti64 and TiB/Ti64) exhibited minimal oxidation with no increase in mass. As the oxidation temperature increased to 500°C, Ti64 showed visible oxygen diffused zones (ODZ$_S$) on the surface under SEM, whereas TiC/Ti64 showed resistance to oxidation as no ODZs were formed on the surface. TiB/Ti64 exhibited intermediate oxidation behaviour and formed an oxide layer, TiO_2 observed on XRD. At 600°C oxidation, Ti64 continued to oxidize significantly, while TiC/Ti64 and TiB/Ti64 showed increased oxidation, though to a lesser extent compared to Ti64.

1 Introduction

Ti6Al4V (Ti64) is the dominant titanium alloy used in biomedical, automotive, chemical, marine, energy, and aerospace industries. The widespread use of Ti64 is attributed to its exceptional features, including high specific strength, excellent corrosion resistance, superior strength, low density, and better biocompatibility [1]. These properties make Ti64 a desirable material for these particular applications (biomedical, automotive, chemical, marine, energy, and aerospace industries.

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Although Ti64 exhibits exceptional qualities, it has limitations in its use for high temperature applications due to the degradation of mechanical properties, physical properties and chemical properties [2]. This limitation arises from the rapid chemical reactions that occur at excessive temperatures, causing Ti64 to oxidize and form a protective passive layer that prevents further oxidation. The oxidation process results in a deterioration of the mechanical properties of the material, which can be problematic for applications that need precise geometry [3]. Guleryuz and Cimenoglu [3] conducted a study on the oxidation behaviour of Ti64 at temperatures ranging from 600ºC to 800ºC. It was found that exposing titanium and its alloy to a high temperature environment with an abundance of oxygen leads to the formation of a TiO_2 oxide layer (OL) and an oxygen diffusion zone (ODZ) on the material's surface. Additionally, it was stated that titanium alloys, which include metals such as aluminium, also develop an oxide layer composed of Al_2O_3 and TiO_2. Through their research, it was determined that the increase in temperature was closely related to the increase in weight gain of Ti64 [3].

Due to the poor performance of titanium and its alloys in oxidizing conditions at high temperatures, alternative methods to enhance the oxidation resistance of Ti64 through coating with oxidation resistant materials were developed [4]. Huang et al. [4] investigated the thermal stability and oxidation resistance of laser clad TiVCrAlSi high entropy alloy deposited on Ti64. The use of TiVCrAlSi high entropy alloy as a coating for Ti64 was attributed to its exceptional characteristics, which included elevated hardness, favourable thermal stability, and outstanding resistance to wear, oxidation, and corrosion. According to the study's findings, the Ti64 material that was coated exhibited a significant enhancement in its ability to resist oxidation at high temperatures. This increase was attributed to the fact that the coated Ti64 showed minimal weight growth when exposed to a temperature of 800ºC, in comparison with the uncoated Ti64. In addition, stable phases such as (Ti, V)5Si3, Al8(V, Cr)5, and a BCC solid solution were seen to develop when the temperature was below 880ºC. The significant enhancement was attributed to the formation of a protective oxide layer, such as TiO_2, SiO_2, Cr_2O_3, and Al_2O_3. Brice et al. [5] investigated the oxidation behaviour and microstructural degradation of Ti64 and Ti641B sheets. The studies demonstrated that the presence of boron (B) enhances the ability of Ti64 to resist oxidation and it does not gain more mass. However, it concluded that the precise mechanism by which B improves oxidation resistance is not well understood [5][5][5]. Zang et al. [6] was motivated to study the impact of B on the oxidation behaviour of Ti64, this is because previous studies have only focused on how B enhances the mechanical properties of Ti64, specifically in terms of creep and elastic strength[5]. Several researches concentrate on enhancing the material's oxidation resistance by post-processing and surface coating, which require more time[7]. An alternative way of improving the oxidation resistance of Ti64 is to add ceramics such as TiC and TiB_2 due to their thermal stability and oxidation resistance. The addition of ceramic TiC or TiB_2 to Ti64 forms a titanium-based matrix composite[8]. Pan et al.[9] used a titanium-based matrix composite to enhance the mechanical characteristics, specifically the strength and ductility, of Ti64. The titanium-based matrix composite was found to possess a unique network microstructure that had a remarkable influence on its mechanical properties. Ti64-based matrix composites are a promising material due to their demonstrated ability to enhance the mechanical properties (strength and ductility) of Ti64[9].

This study aims to study the oxidation behaviour of Ti64-based matrix composites manufactured through additive manufacturing as it is crucial for assessing their suitability for high temperature applications and devising strategies to address any potential oxidation-related concerns. Ti64-based matrix composites show great potential as materials for high-

temperature applications [9]. This research has the potential to facilitate the creation of enhanced Ti64-based matrix composites that are more resistant to oxidation. As a result, the performance, durability, and safety of the final products can be improved.

2   Methodology

A laser-based additive manufacturing technique known as Directed Energy Deposition (DED) was used in the manufacturing of the Ti64, TiC/Ti64 and TiB/Ti64. Figure 1 shows the DED system which consists of several essential components, such as the KUKA robotic arm, which allows for perfect movement. The powder feeder system allows for precise transfer of powders (Ti64 and TiC) into the melt pool. The melt pool is created by a focused energy source of the 1073 nm, IPG fiber laser. The manufacturing of TiC/Ti64 and TiB/Ti64 composites as well as samples of Ti64 was done by depositing material layer by layer through the use of this laser. All of the samples were produced using a laser energy density of 90 J/mm^2, which was used for the production process. Both the shielding gas and the carrier gas were argon. The shielding gas flowed at a rate of 15 L/min and the carrier gas flowed at a rate of 1.5 L/min. The mass flow rate percentage of Ti64 in the TiC/Ti64 and TiB/Ti64 alloys was set to 95%, whereas the mass flow rate percentage of TiC and TiB were both 5%. The mass flow rate percentage for the as-built Ti64 was 100%.
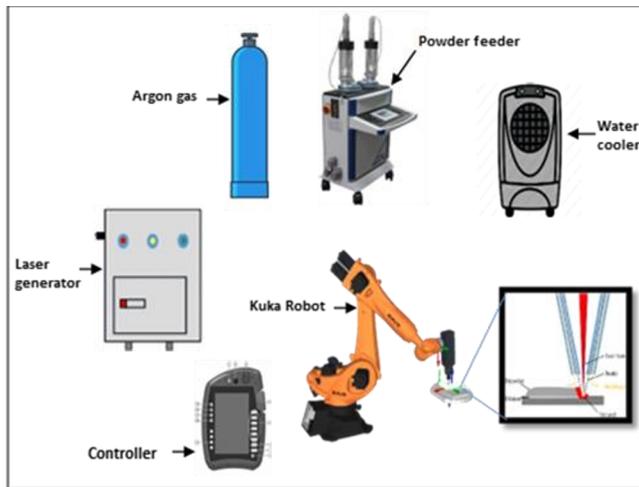

Fig. 1. Directed Energy Deposition schematic diagram.

2.1 Sample preparation and material characterization

The specimens were cut into square forms measuring 5 mm x 5 mm using a Struers Lobotom-5 cutting machine with a Ti-a-250SC cutting disk. The samples were prepared metallographically, polishing the surface to a 1 μm surface finish. The samples were grinded and polished till achieving a reflective surface similar to a mirror. The sample's microstructure was examined by scanning electron microscopy (SEM), while X-ray diffraction (XRD) was used to confirm the presence of phases on the surface of the material. The samples were then washed using acetone in an ultrasonic bath and their masses were recorded before and after placement in a furnace.

2.2 Oxidation test

The oxidation tests were conducted on Ti64, TiC/Ti64, and TiB/Ti64 samples, each specimen was prepared by cutting them into rectangular pieces of 5 mm x 5 mm and polishing them to get a uniformly 1 μm surface finish. Before conducting the tests, the specimens were weighed to ascertain the starting mass. Afterwards, the samples were placed into a high temperature furnace in an oxygen environment at temperatures of 400°C-600°C, in the presence of an oxygen-rich environment. The oxidation experiments were conducted for six hours at 400°C, 500°C and 600°C. Following the designated period at each temperature, the samples were removed from the furnace and left to cool down at room temperature. After the oxidation test, the samples were reweighed to determine their final mass.

3   Results and discussion

3.1 Oxidation kinetics analysis

Figure 2 shows the oxidation kinetics of Ti64, TiC/Ti64, and TiB/Ti64 for 6 hours at temperatures of 400°C, 500°C and 600°C. At 400°C, there was no increase in mass per unit area, indicated by a constant value of 0 mg/cm^2 for all materials. This behaviour is attributed to the relatively slow oxidation rate at this temperature and was supported by findings by several studies, which determined that in Ti64 the growth and formation of the oxide layer occur above the temperature of 600°C [5], [10], [11][12]. However, when the temperature was raised to 500°C, the materials (all the samples) gained mass, indicating that the material oxidized. At a temperature of 500°C, the oxidation rate increased and the reaction between Ti and O_2 becomes more rapid, as molecules effectively collide more often with the exposed surface to create ODZs, as compared to the oxidizing temperature of 400°C. At a temperature of 500°C, Ti64 showed a significant increase in mass gain of 0.68 mg/cm^3, while TiB/Ti64 showed a lower mass gain of 0.35 mg/cm^3 and TiC/Ti64 showed no change in mass. Ti64 experienced an increase in mass as a result of the reaction between titanium and oxygen, leading to the formation of an OZD and similar observations were reported by Casadebaigt et al. [13] where Ti64 oxidized at 500°C. This suggested that TiB played a role in preventing further oxidation of the material by the formation of an oxide layer (TiO_2). Although some ODZs were formed, their quantity was significantly lower than that observed in Ti64 and Brice et al. [5] also observed similar behaviour when B was added to Ti64 to improve the oxidation resistance of Ti64. It was further determined that the mechanism of how B improves Ti64 oxidation resistance is not well understood. The TiC/Ti64 composite exhibited no change in mass even at a temperature of 500°C, this shows that oxidation at 500°C is not enough to change the mass of the material, and this indicates that the addition of TiC in Ti64 protects against oxidation. This protective effect is attributed to the presence of TiC at the grain boundaries of the material, acting as a barrier against oxidation.

At a temperature of 600°C, the process of oxidation for Ti64, TiC/Ti64, and TiB/Ti64 materials shows unique behaviours. While there was no change in mass for TiC/Ti64 at 500°C, the composite material now exhibits a noticeable increase in mass at 600°C, although it is still smaller than the increase reported in Ti64. This change indicates that TiC continues to provide a certain level of resistance against oxidation by reacting with O_2 forming oxide layer which prevent further oxidation. The elevated temperature accelerates the oxidation process, resulting in a significant increase in mass. This behaviour suggests that although TiC is still efficient in preventing oxidation, its protective barrier is significantly weakened at

600°C in comparison to lower temperatures and by also forming a TiO_2 oxide layer. On the other hand, at a temperature of 600°C, TiB/Ti64 still shows a smaller increase in mass compared to Ti64. This suggests that TiB still provides some kind of protection against oxidation, even at this high temperature and the formation of the TiO_2 oxide layer. While TiB/Ti64 exhibits higher oxidation, levels compared to 500°C, the number of ODZs generated is significantly smaller than that seen in Ti64. This emphasizes the advantageous effect of TiB in lowering oxidation rates during oxidation. At a temperature of 600°C, Ti64 experiences a significant increase in mass as a result of increased oxidation kinetics. Higher temperatures increase the reactivity between titanium and oxygen molecules, resulting in the formation of a thicker TiO_2 oxide layer compared to lower temperatures.

Fig. 2. Mass gain vs temperature graph of Ti64, TiC/Ti64 and TiB/Ti64 during oxidation.

3.2 Microstructure analysis

The microstructures shown in Figures 3(a), (b), and (c), represent Ti64 samples before oxidation. All samples show the martensitic microstructures consisting of alpha phase, beta phase and alpha prime phase. Figure 3 (a) shows the coarser martensitic structure while Figures 3(b) and (c) show fine martensitic structures that are due to the thermal gradients which mainly contribute to this martensitic transformation and can refine the size of martensitic laths.
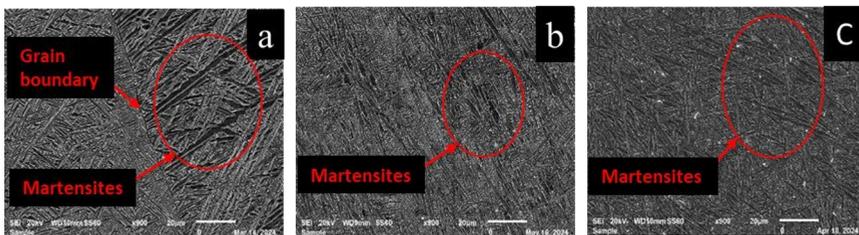

Fig. 3. As-built microstructure images of Ti64 before oxidation test.

Figure 4(a), (b) and (c) shows the microstructure of Ti64 after oxidation test at 400°C, 500°C and 600°C respectively. At 400°C oxidation, Ti64 exhibit no visible signs of oxide formation,

or oxygen-diffused zones (ODZs) and only shows the pit formation. The absence of oxide phases on the surface, even under heat, suggests minimal oxidation occurring at this temperature in which the material usually forms a thin passive layer to protect itself. Upon increasing the temperature to 500°C, distinct differences in Ti64 begin to emerge. Ti64 shows on the surface, indicating the onset of oxidation at this elevated temperature. According to the study by Guleryuz et al [14], the ODZ develops above 400°C, hence at this temperature ODZs are observed. Maytorena-Sánchez et al [12] reported that significant oxidation in Ti alloys typically initiates at temperatures above 600°C . As the temperature is further elevated to 600°C, the extent of oxidation becomes more pronounced across all materials. Significant TiO_2 layer forms on the surface, as evidenced by the micrographs. The reaction for the formation of the TiO_2 oxide layer :

$$Ti\ (s) + O_2\ (g) \rightarrow TiO_2\ (s) \qquad\qquad (1)$$

This layer indicates extensive oxidation, which compromises the surface integrity and overall oxidation resistance of the alloy at this temperature.
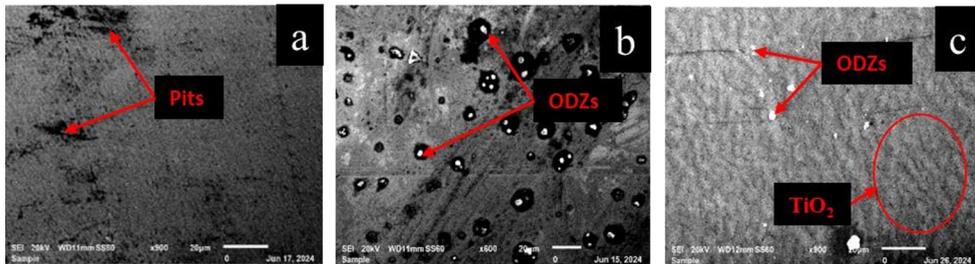

Fig. 4. Microstructure images of Ti64 after oxidation at temperatures of 400°C (a), 500°C (b) and 600°C (c).

The microstructure shows significant features of TiC/Ti64 alloy, as shown in Figures 5(a), 4(b), and 5(c). All samples show the network microstructures consisting of TiC and Ti64 as the matrix. Figure 5(a) shows the presence of TiC whiskers while Figures 5(b) and (c) show smaller circular TiC structures and the presence of eutectic particles of TiC.
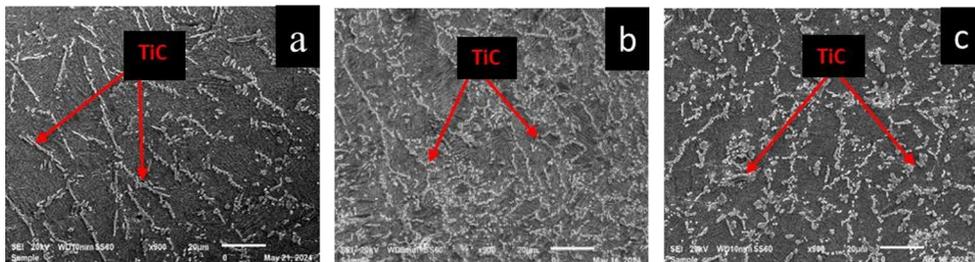

Fig. 5. As-built microstructure images of TiC/Ti64 (before oxidation).

Figure 5(a), (b), and (c) present the microstructure of TiC/Ti64 after oxidation test at 400°C, 500°C, and 600°C, respectively. At 400°C, no noticeable oxide formation is observed on the surface of TiC/Ti64 alloy. The TiC whiskers are no longer visible, and the surface shows no

evidence of ODZs. The oxidation exposure at 400°C appears insufficient to initiate the oxidation process in the TiC/Ti64 alloy. In contrast, TiC/Ti64 behaves differently at 500°C. The microstructure shows that the TiC phases from the as-built condition remain visible, with no presence of ODZs on the surface and oxide layer. This indicates that the presence of TiC in the alloy plays a protective role, likely acting as a barrier that impedes oxidation and limits its penetration into the material since the reactivity of Ti and O_2 is higher than the one of TiC and O2. At 600°C oxidation TiC/Ti64 shows the TiO_2 oxide layer on the surface. However, despite the presence of an oxide layer (TiO_2), at this temperature the TiC react with oxygen to form a passive layer which protects the underlying titanium alloy, reducing the rate and extent of oxidation.
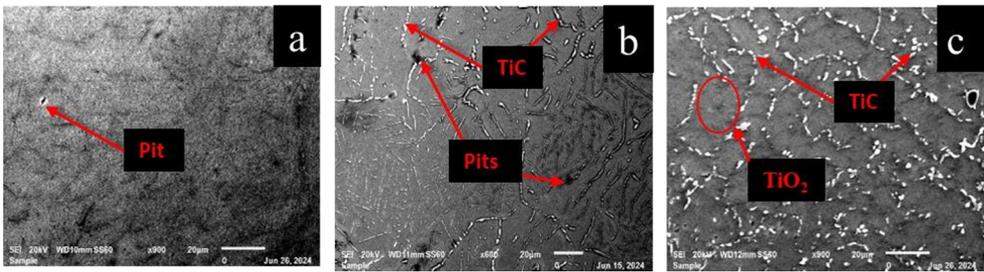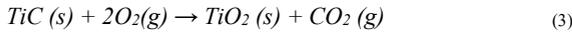
$$TiC\ (s) + 2O_2(g) \rightarrow TiO_2\ (s) + CO_2\ (g) \qquad (3)$$

Fig. 6. Microstructure images of TiC/Ti64 after oxidation at temperatures of 400°C (a), 500°C (b) and 600°C (c).

A different structure is noticeable when examining the TiB/Ti64 microstructure shown in Figures 6(a), (b), and (c). Figure 6(a) depicts a network microstructure consisting of interconnected TiB phases within the Ti64 matrix. Figure 6(b) provides an alternative viewpoint, with TiB phases organized in a linear pattern. Figure 6(c) shows TiB phases located at the grain boundaries of the material, with linkages found at certain interfaces. This location indicates a tendency for TiB phases to concentrate at grain boundaries during the solidification.
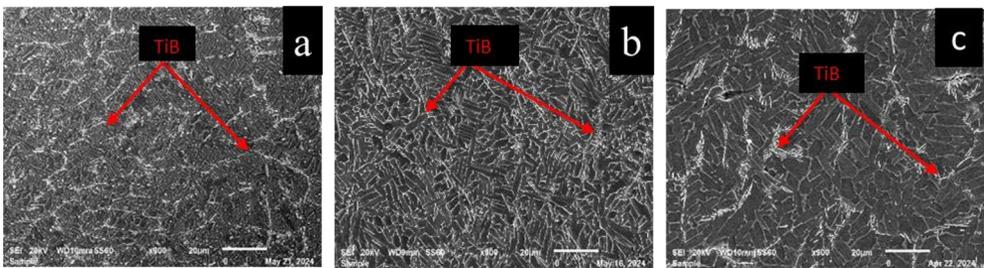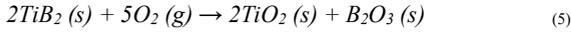

Fig. 7. As-built microstructure images of TiB/Ti64 before oxidation test.

Figure 5(a), (b), and (c) present the microstructure of TiB/Ti64 after oxidation test at 400°C, 500°C, and 600°C, respectively. At 400°C oxidation, TiB/Ti64 exhibit no visible signs of oxide formation and ODZs. The TiB is no longer visible on the microstructure the microstructure shows the pit formation. At this temperature the oxidation is minimal. For

TiB/Ti64, however, the situation is markedly different. At 500°C, ODZs are observed on the surface, indicating that oxidation has occurred. The TiB phase, which was prominent in the as-built microstructure, is no longer discernible at this temperature. This suggests that oxidation has not only affected the surface properties but may have also altered the TiB phase itself, either through diffusion, transformation, or reaction with oxygen forming a passive layer. The reaction of TiB and O_2 :

$$2TiB_2 \text{ (s)} + 5O_2 \text{ (g)} \rightarrow 2TiO_2 \text{ (s)} + B_2O_3 \text{ (s)} \qquad (5)$$

Oxidation becomes more evident at 600°C on TiB, with a clear oxide layer (TiO_2) forming on the surface. Interestingly, the TiB phases are not visible at this temperature on the micrograph and there is the presence of ODZs.
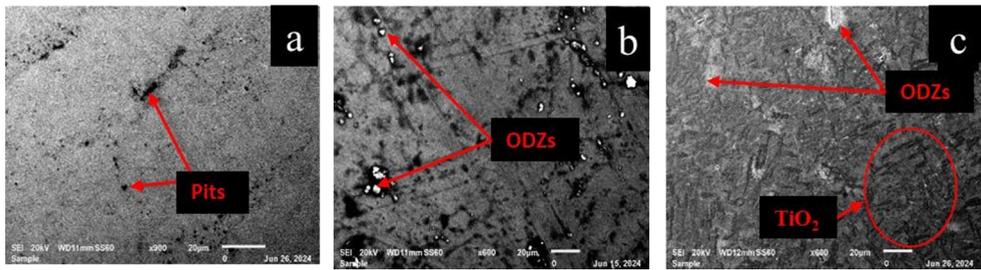

Fig. 8. Microstructure images of TiB/Ti64 after oxidation at temperatures of 400°C (a), 500°C (b) and 600°C (c).

3.3 Phase analysis

The as-built Ti64 XRD pattern shows peaks corresponding to the initial phase composition of the Ti64 alloy before oxidation. The major peaks likely correspond to the α-Ti (hexagonal close-packed) phase, which is the dominant phase in the as-built Ti64 alloy. A few smaller peaks may correspond to the presence of the β-Ti (body-centered cubic) phase. Ti64 oxidized at 400°C shows the overall intensity of the peaks has decreased compared to the as-built sample. This may be due to the oxidation being minimal and microstructural changes as observed in Figure 4(a). The α-Ti phase still dominates, and there are no clear signs of new phases forming. Oxidation at this temperature is likely mild, with limited formation of oxide layers. At 500°C, more noticeable changes in the XRD pattern are evident. The intensity of the peaks continues to rise, indicating continued structural evolution during oxidation. No new peak formed which can indicate the formation of oxide layer TiO_2. Oxidation at 600°C shows significant changes in the XRD pattern, likely indicating extensive oxidation. The increase in the intensity of certain peaks suggests that the oxidation layer is growing, and that the crystallinity of the oxide phase has improved at this temperature. The new peak formed $2\theta \approx 38°$ shows formation of oxide layer TiO_2.
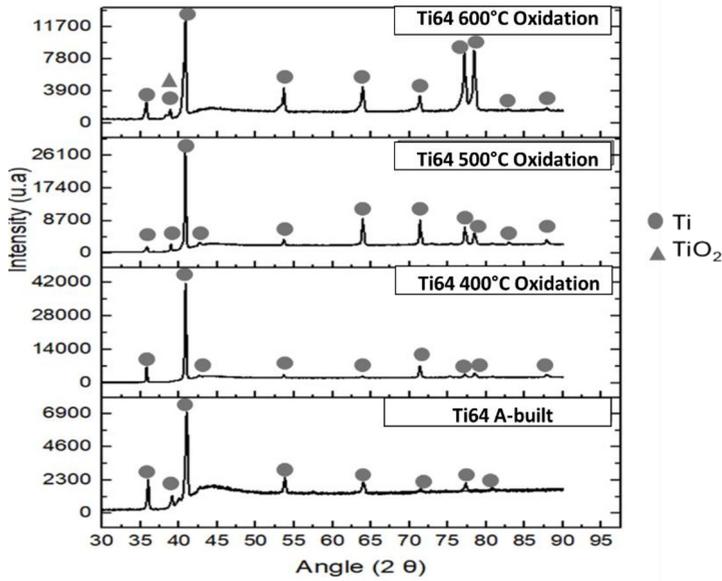
Fig. 9. XRD spectrums of Ti64 after oxidation test.

Figure 10 shows the XRD patterns for the TiC/Ti64 as built and oxidized samples at different temperatures 400°C, 500°C, and 600°C. In the as-built condition, the primary peaks correspond to the α-Ti phase of Ti64 and TiC, with no evidence of oxide formation. As the samples are oxidized at 400°C, the intensity of the peaks increases, indicating microstructural changes, though no significant new phases are detected, suggesting minimal oxidation at this temperature. At 500°C, no new peaks emerged that can indicate the formation of TiO_2, while TiC remains stable. The most significant changes are observed at 600°C, where a new peak formed at $2\theta \approx 43°$ suggesting extensive formation of rutile TiO_2. The stability of TiC throughout the oxidation process highlights its resistance to high-temperature oxidation, while the Ti64 matrix undergoes substantial oxidation, particularly at 600°C. Overall, the composite shows increasing oxidation with temperature, with TiC providing some stability against further oxidation by forming an oxide layer.
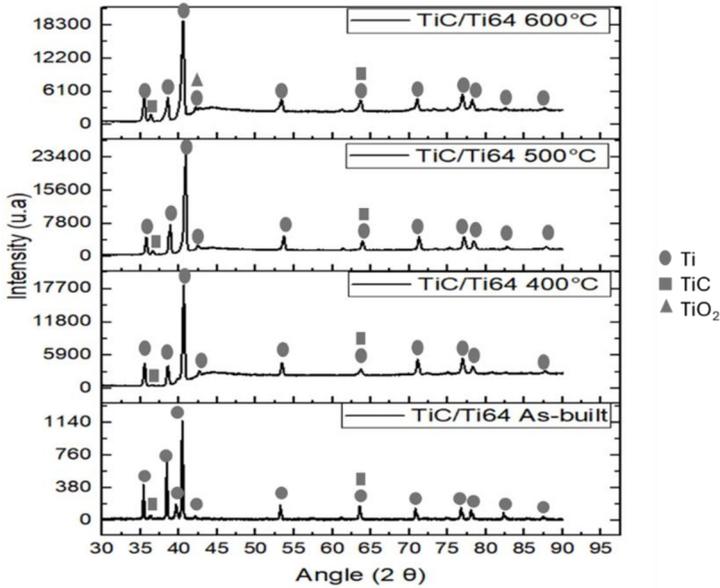
Fig. 10. XRD spectrums of TiC/Ti64 after oxidation

The XRD pattern of the as-built sample serves as the baseline, showing the typical phases of the TiB/Ti64 composite. The strong peaks around 40–45° which correspond to the α-phase of Ti (hexagonal close-packed structure) and the presence of TiB is observed. Upon oxidation at 400°C, the peaks remain consistent with the as-built sample, suggesting that minimal phase changes occur at this lower oxidation temperature. The primary TiB and Ti64 phases seem stable under this condition. At 500°C, there is still no drastic change in peak intensity or location, but there is peak broadening or changes in peak intensity and formation of a new peak at $2\theta \approx 43°$, which could indicate the oxide formation, though the bulk of the TiB/Ti64 phases remain intact. At 600°C, there are more notable changes in peak intensity and possibly additional peaks at $2\theta \approx 43°$ and phase shifting. This is due to the formation of oxide layers TiO_2 on the surface of the samples, which is common in titanium alloys when exposed to high temperatures in oxidizing environments. The oxidation process at this temperature may have induced the growth of TiO_2 phases or other oxides.
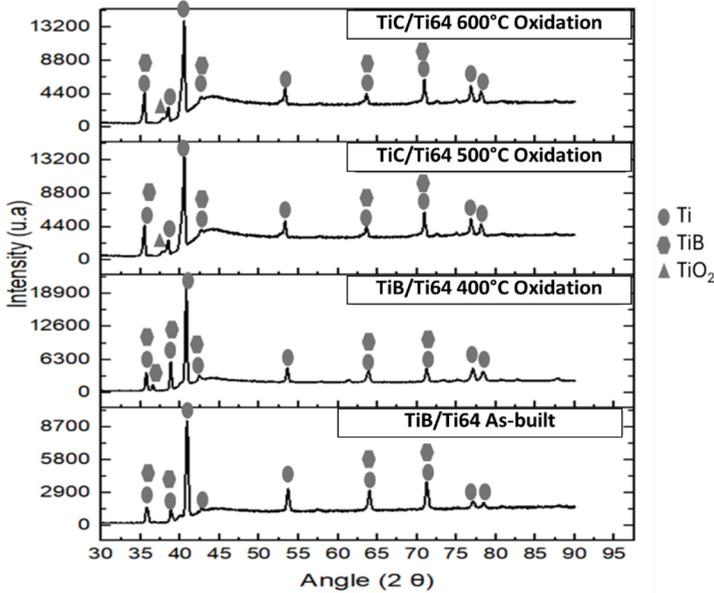
Fig. 11. XRD spectrums of TiB/Ti64 after oxidation

4   Conclusion

In conclusion, the oxidation kinetics of Ti64, TiC/Ti64, and TiB/Ti64 alloys reveal distinct behaviours influenced by temperature variations from 400°C to 600°C. At 400°C, all materials exhibit negligible mass change, indicative of slow oxidation rates consistent with previous studies. However, as temperatures rise to 500°C and 600°C, significant differences emerge. Ti64 shows substantial mass gains due to oxide layer formation, corroborated by microstructural analysis highlighting the increased presence of ODZs. In contrast, TiC/Ti64 exhibits minimal mass increase even at 500°C, attributed to TiC's protective role at grain boundaries hindering oxidation. At 600°C, while TiC/Ti64 shows increased oxidation, the protective effect of TiC remains evident though diminished. TiB/Ti64 also demonstrates reduced oxidation compared to Ti64 at both temperatures, indicating TiB's effectiveness in limiting oxide formation despite some observable surface oxidation at 600°C. Overall, these findings underscore the crucial role of alloying compounds like TiC and TiB in modifying the oxidation resistance of Ti64 under elevated temperatures, offering insights into potential strategies for enhancing material durability in high-temperature environments. Further research will focus on elucidating the specific mechanisms through which TiC and TiB influence oxidation kinetics.

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