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Effect of Thermal Oxidation Parameters on Morphological Properties of Thermal Oxide Coatings on Ti6Al4V Titanium Alloy

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ABSTRACT

The effects of thermal oxidation temperature and treatment route on the morphology of oxide coatings formed on Ti6Al4V alloy were systematically investigated. Ti6Al4V samples were subjected to different oxidation treatments, namely annealing, normalizing, and air-quenching—at temperatures of 850 and 900 °C. The surface morphology and compositional features of the resulting oxide coatings were characterized. In addition, the coating thickness and weight gain per unit area ($\Delta W/A$) were quantitatively evaluated. The results indicate that nearly all oxidation treatments produced continuous, well-adhered, and largely defect-free oxide coatings on the Ti6Al4V substrate. Annealing at both temperatures led to the formation of uniform and dense oxide layers predominantly composed of TiO₂ (rutile phase). In contrast, normalizing resulted in comparatively thinner oxide coatings exhibiting minor surface scars and increased roughness, while air-quenched specimens developed thicker oxide layers accompanied by slight surface spallation and micro-crack formation at both temperatures. The oxide coating formed by annealing at 900 °C exhibited a moderate thickness of $195 \pm 9.75 \mu\text{m}$. Moreover, annealing at 900 °C yielded the highest $\Delta W/A$ of $126 \pm 6.3 \text{ mg/cm}^2$. Overall, among the investigated treatments, annealing at 900 °C produced oxide coatings with the most favorable morphological characteristics.

1. INTRODUCTION

Several materials, having higher strength-to-weight ratios, such as ferrous alloys (Aqil Inam, 2017; Hafeez and Farooq, 2018; Hafeez *et al.*, 2020), beryllium alloys (Schneeberger), magnesium alloys (Matli *et al.*, 2020), amorphous alloys (Abbas *et al.*, 2024; Yasir *et al.*, 2024), high entropy alloys (Anwer *et al.*, 2023), and titanium alloys, have been progressively developed to meet the increasing demand in various critical applications. For many decades, titanium alloys have been attaining special attention among all due to

their excellent mechanical, electrochemical, and biomedical properties (Liu and Shin, 2019; Zhao *et al.*, 2019). Two-phase ($\alpha + \beta$) Ti6Al4V alloy is one of the well-known and extensively used titanium alloys, covering almost 50% of the titanium demand in the world market today. Ti6Al4V alloy was developed for aircraft structural applications, including gas turbines, jet engines, and airframe parts (Benedicto *et al.*, 2018; Duan *et al.*, 2019; Milton *et al.*, 2019). Its superior properties, such as higher strength, lower density, and higher fracture toughness, also make it a suitable material for several applications in automobile industries and

marines (Kümmel *et al.*, 2019). Its excellent corrosion resistance to corrosive acids and alkalis makes it a suitable choice for chemical industries (Liu and Shin, 2019). Its ability to produce natural oxide layer on its surface provides superior biocompatibility, bioactivity, and chemical stability, which further widens its fields of applications to biomedical industries. Its biomedical applications include surgical instruments (Xu *et al.*, 2019), orthopedic (hip, knee and dental) implants (Bartolomeu *et al.*, 2019; Battocchio *et al.*, 2019; Cho *et al.*, 2019; Miranda *et al.*, 2019; Zhou *et al.*, 2019), bone defects repairing, arthroplasty in orthopedics (Li *et al.*, 2019), and implantable devices (joint prostheses devices, fracture fixation devices) (Luo *et al.*, 2018).

The morphological properties Ti6Al4V alloy can be further enhanced by various techniques, such as laser technology (Bai *et al.*, 2025; Gain *et al.*, 2025; Jiao *et al.*, 2025), plasma-enhanced chemical vapor deposition of carbon amorphous coating (Cho *et al.*, 2019), electropulsating treatment (Zhao *et al.*, 2019), diazonium compounds (Sandomierski *et al.*, 2018), electron beam technique (Ramskogler *et al.*, 2017), plasma copper, nickel alloying process (Yao *et al.*, 2014), anodization (Diamanti *et al.*, 2013), and high-temperature oxidation treatment. One of the economical and easier methods of improving morphological properties of Ti6Al4V is high-temperature oxidation treatment (Deen *et al.*, 2014).

Various alloys and their coatings have the tendency to oxidize in the oxygen enriched environment (Inam *et al.*, 2020; Hafeez *et al.*, 2021; Cho *et al.*, 2022; Zhu *et al.*, 2024; Liang *et al.*, 2025). The titanium-based alloys, specifically Ti6Al4V absorb oxygen, nitrogen, and hydrogen from the environment during thermal treatment and form a thin, stable thermal oxide passive layer on the surface. This layer resists further oxidation as well as oxygen diffusion at low treatment temperatures. But at a sufficiently higher temperature, oxygen

penetrates through the oxide layer and reacts with the substrate at the substrate/oxide interface, resulting in a thick oxide layer. The formation of the mechanically stable and chemical resistant oxide layer by thermal oxidation treatment significantly improves the morphological properties of Ti6Al4V and other titanium-based alloys (Wen *et al.*, 2012; Jamesh *et al.*, 2013; Aniolek *et al.*, 2016; Aniolek, 2017; Aniolek *et al.*, 2018; Fargas *et al.*, 2018; Aniolek and Kupka, 2019). These properties are highly sensitive to thermal oxidation treatment parameters, including the type of treatment, oxidation temperature, and oxidation time. It has been reported that increasing thermal oxidation temperature causes considerable improvement in surface hardness and roughness (Jamesh *et al.*, 2013). Aniolek and Kupka (2019) reported that thermal oxidation at 700–800°C caused the formation of a much thicker oxide layer. Jamesh *et al.* (2013) observed that the microstructure of the TiO₂ layer, formed at 850°C, contained α -titanium, and rutile phases.

The current work aims to investigate the effect of thermal oxidation parameters such as oxidation treatments and temperatures on morphological properties of thermal oxide coatings on Ti6Al4V titanium alloy. Morphologies and thickness values of all thermal oxide coatings were evaluated by scanning electron microscopy. The deposition of thermal oxide coatings on Ti6Al4V alloy was also validated by exploring their elemental composition through an energy dispersive spectrometer. The weight per unit area of thermal oxide coatings was also measured by precision electric weight balance.

2. EXPERIMENTAL WORK

2.1 Materials

The medical-grade titanium Ti6Al4V alloy, having a chemical composition given in Table 1, was received in the form of a sheet of 4 mm



Table 1. Chemical composition of titanium Ti6Al4V alloy (wt.%)

Al	V	C	Fe	N	H	O	Ti
6.0	4.0	0.1	0.4	0.005	0.0125	0.20	89.24

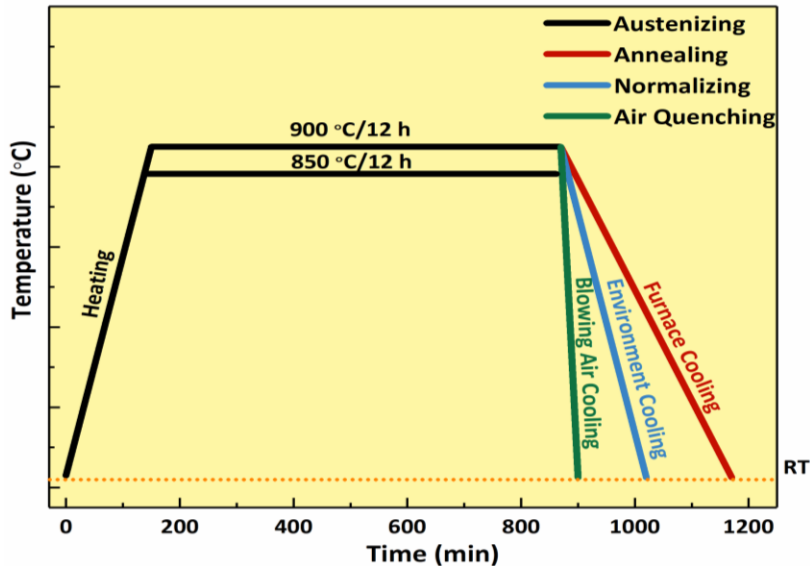


Figure 1. Schematic illustration of thermal oxidation treatments, applied to titanium Ti6Al4V alloy

thickness. Ti6Al4V sheet was cut into samples of dimension 10 mm × 10 mm × 4 mm by a computer numerical control wire-cut machine for subsequent thermal oxide treatments and characterization.

2.2 Thermal Oxidation Treatments

Before thermal oxidation treatments, the Ti6Al4V alloy samples were manually ground on SiC papers of grades P60, P100, P200, P400, P600, P800, P1000, and P1200 and polished on SiC papers of grades P1500, P2000, P2500, and P3000, using an automatic grinder/polisher (Ecomet 250 Grinder Polisher, USA). After the preparation, the samples were placed in two separate electric muffle furnaces, maintained at 850 °C and 900 °C temperatures for 12 h. These samples were then subjected to the annealing process by very slow furnace cooling, the normalizing process by

environmental cooling, and the air-quenching process by blowing air cooling, separately (Hafeez, 2020). The thermal oxidation treatments are schematically illustrated in Figure 1.

2.3 Characterizations

To analyze the morphology of thermal oxide coatings deposited on Ti6Al4V alloy, a scanning electron microscope (SEM, Hitachi SU-8000, Japan) was used. Micrographs were captured from the cross-section as well as from the surface. To further validate the deposition of thermal oxide coatings, the elemental compositions of the coatings were measured, using an energy dispersive spectrometer (EDS) equipped with a new generation silicon drift detector (SDD), attached to the SEM. Weight gain per unit surface area ($\Delta W/A$) of coatings was also determined through a precision

electric weight balance (Sartorius GC–1603P, France) by dividing weight difference (weight of samples before oxidation and after oxidation

treatment) by the unit surface area of corresponding sample.

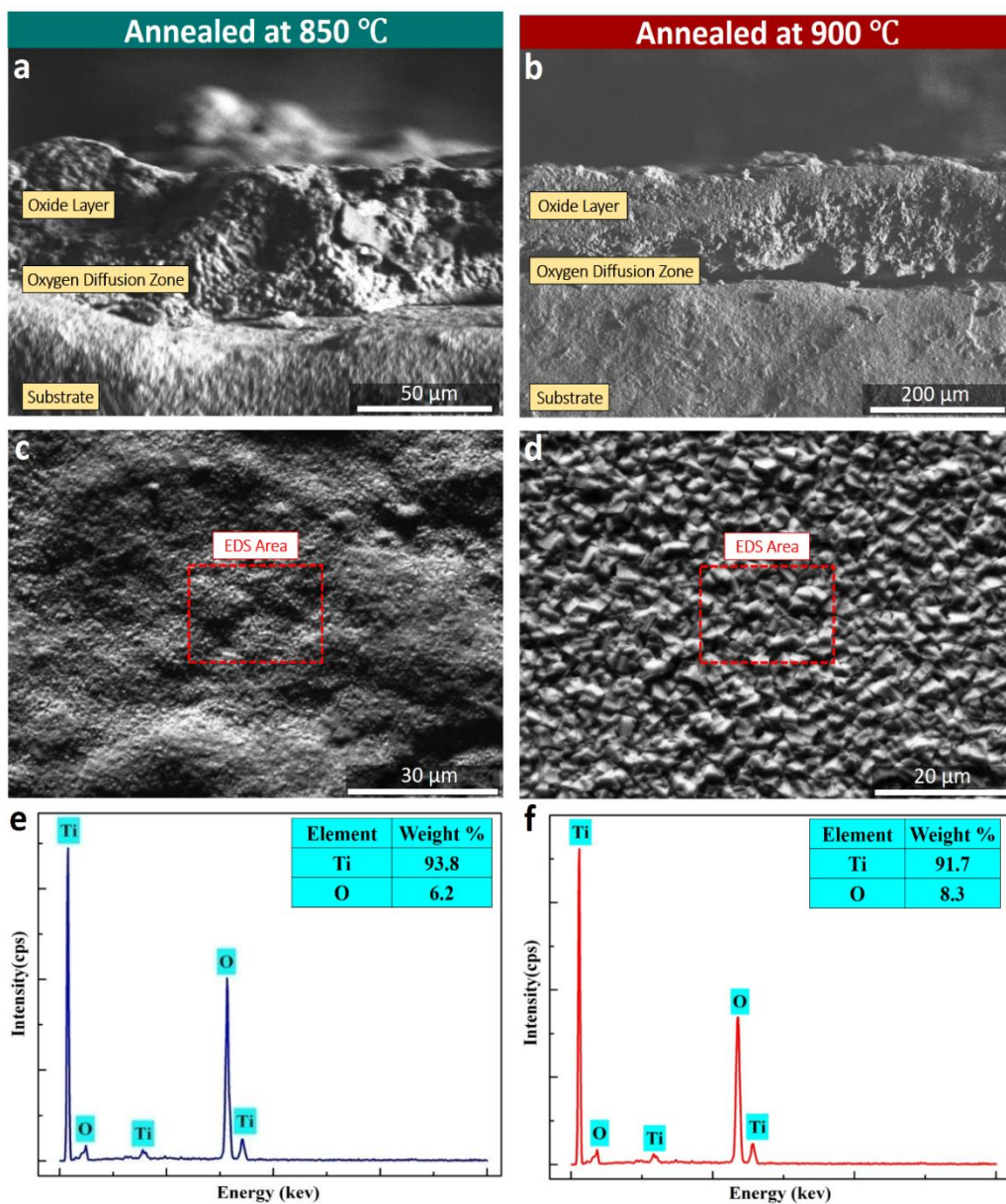


Figure 2. SEM micrographs of thermal oxide coatings on Ti6Al4V alloy, obtained after annealing at 850 °C; **(a)** cross-section, **(c)** top surface, and **(e)** corresponding EDS spectrum and at 900 °C; **(b)** cross-section, **(d)** top surface, and **(f)** corresponding EDS spectrum, for 12 h



3. RESULTS

3.1 Morphological Properties

SEM micrographs and corresponding EDS spectra of thermal oxide coatings on Ti6Al4V alloy, deposited by annealing, normalizing, and air-quenching processes at 850 and 900 °C for 12 h, are illustrated in Figures 2–4. Micrographs validated the presence of a homogeneously segregated thermal oxide layer on the surface of both the Ti6Al4V samples, annealed at 850 and 900 °C. Sufficiently thick thermal oxide coatings were observed in cross-sectional micrographs of both samples as illustrated in Figure 2(a) and (b). Uniform and defect-free thermal oxide coatings were developed after the annealing processes at both temperatures, which thoroughly covered the surface of Ti6Al4V samples as illustrated in

Figure 2(c) and (d). EDS spectra further validated the deposition of thermal oxide coatings on both the annealed samples. From the elemental compositions, it can be predicted that thermal oxide coatings comprised TiO_2 and rutile structure after annealing at 850 and 900 °C as presented in the EDS spectra in Figure 2(e) and (f).

SEM micrographs of the thermal oxide coating, deposited on Ti6Al4V alloy by normalizing at 850 °C for 12 h, are illustrated in Figure 3. Normalizing at 850 °C provided slightly thinner oxide coating, compared to the annealing process, having shallow scars on its rough surface (Figure 3(a) and (b)). EDS spectra validated the presence of the oxide layer by exhibiting 11.2 wt.% oxygen in elemental composition (Figure 3(c)).

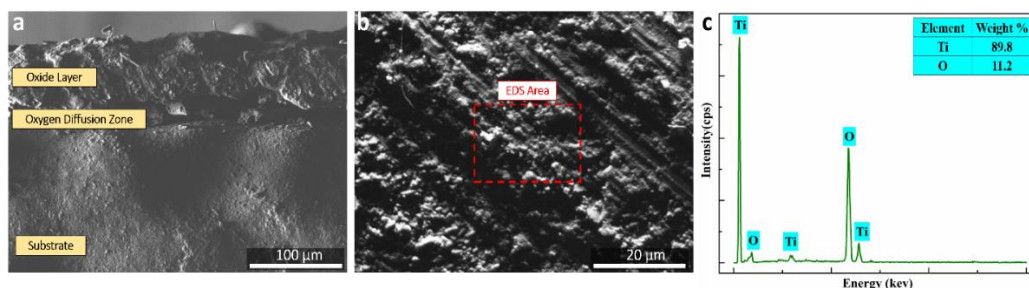


Figure 3. SEM micrographs of thermal oxide coating on Ti-6Al-4V alloy, obtained after normalizing at 850 °C; (a) cross-section, (b) top surface, and (c) corresponding EDS spectrum, for 12 h

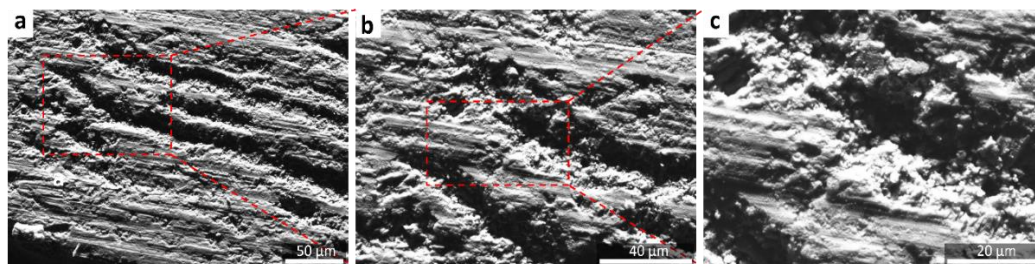


Figure 4. SEM micrographs of thermal oxide coating on Ti6Al4V alloy, obtained after air-quenching at 900 °C for 12 h, from top surface at (a) 500×, (b) 1000×, and (c) 2000× magnifications

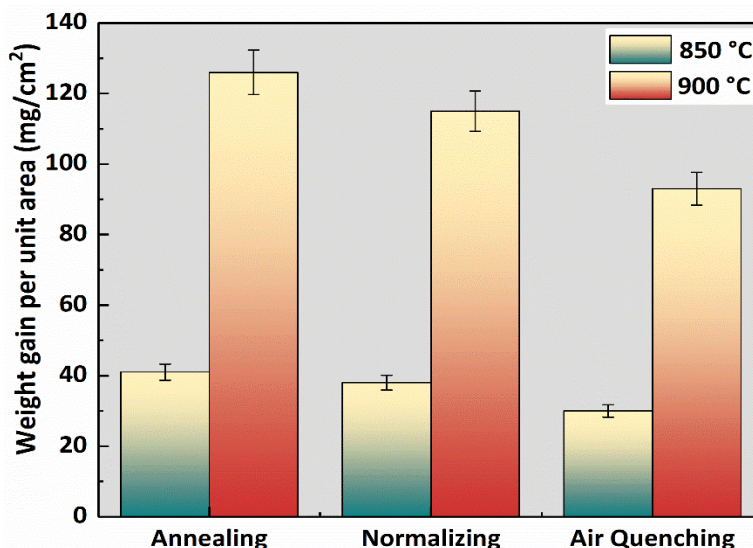


Figure 5. Variations in thickness of thermal oxide coatings on Ti-6Al-4V alloy as a function of thermal oxidation treatments, performed at 850 and 900 °C for 12 h

Although no spallation was observed in annealed and normalized samples, air-quenched samples exhibited greater spallation. Both the air-quenched Ti6Al4V samples exhibited significantly thicker coatings than annealed and normalized samples but demonstrated surface spallation with micro-cracks on the coatings as illustrated in Figure 4.

3.2 Thermal Oxide Coatings Thickness

The thickness of thermal oxide coatings, deposited on Ti6Al4V alloy by various thermal oxidation treatments at 850 and 900 °C for 12 h, were measured by SEM. Thickness values of thermal oxide coatings were found to be highly sensitive to both oxidation temperature as well as oxidation treatments. Variations in the thickness of thermal oxide coatings as a function of thermal oxidation temperatures and treatments are plotted in Figure 5. From the cross-section, all the thermal oxide coatings were observed to be continuous, well adhered to the substrate, and defect-free. The thermal oxide coatings, deposited at 900 °C, demonstrated greater thickness values than

coatings deposited at 850 °C, under all thermal oxidation treatments.

The annealing process at 850 and 900 °C for 12 h developed thermal oxide coatings of a moderate thickness of $62 \pm 3.1 \mu\text{m}$ and $195 \pm 9.75 \mu\text{m}$, respectively. However, annealing at 900 °C offered significantly thicker oxide coating than at 850 °C temperature. The normalizing at 850 °C offered 48% thinner oxide coating with thickness $32 \pm 1.6 \mu\text{m}$ while normalizing at 900 °C produced oxide coatings of almost an identical thickness ($188 \pm 9.4 \mu\text{m}$) compared to the annealing process. Therefore, the air-quenching process provided oxide coating with a maximum thickness of $85 \pm 4.25 \mu\text{m}$ and $278 \pm 13.9 \mu\text{m}$ at both oxidation temperatures.

3.3 Thermal Oxide Coatings Weight Gain Per Unit Area

The variations in $\Delta W/A$ of thermal oxide coatings, obtained after oxidation at 850 and 900 °C for a processing time of 12 h as a function of thermal oxidation treatments, are plotted in Figure 6. The values of $\Delta W/A$ were

also found to be highly sensitive to both oxidation temperature and treatments. For all treatments, the values of $\Delta W/A$ were found to be significantly greater at 900 °C than at 850 °C.

The normalizing process at both 850 and 900 °C produced thermal oxide coatings with lower values of $\Delta W/A$ than annealed samples ($38 \pm 2.1 \text{ mg/cm}^2$, $115 \pm 5.75 \text{ mg/cm}^2$, respectively). After air quenching, minimum values of $\Delta W/A$ were achieved at both oxidation temperatures ($30 \pm 1.75 \text{ mg/cm}^2$, $93 \pm 4.65 \text{ mg/cm}^2$). However, air-quenching at 900 °C provided a higher value of $\Delta W/A$ than 850 °C.

4. DISCUSSION

The influence of thermal oxidation parameters on the morphology, thickness, and integrity of oxide coatings on Ti6Al4V alloy was critically examined in light of existing literature and observed trends. This might be attributed to the sufficiently higher oxidation temperature and longer oxidation time similar to the work of Fargas *et al.* (2018). This observation aligns with the general understanding that elevated temperatures and prolonged exposure enhance oxide layer uniformity and stability. Due to the very slow cooling inside the furnace, no spallation was observed in both annealed samples, similar to the work of Jamesh *et al.* (2013). Such slow cooling likely minimized thermal stresses, preserving coating adherence. The spallation in the thermal oxide layer can be caused by a larger volume ratio of rutile to Ti, larger lattice mismatch, and a greater difference in the coefficient of thermal expansion (Jamesh *et al.*, 2013; Aniołek, 2017). These spallation and micro-cracks might be attributed to the fast cooling rate, which generated greater thermal stresses from the difference in thermal expansion coefficients between TiO_2 and rutile structures (Jamesh *et al.*, 2013; Aniołek, 2017).

The role of oxidation temperature in determining coating thickness was also evident. The greater thickness observed at 900 °C can be attributed to the role of oxidation temperature in oxide layer deposition. The thermal oxidation mechanism begins with the adsorption of oxygen molecules from the environment, followed by nucleation of oxides, deposition of a thinner thermal oxide coating, and ultimately growth to a thick stable thermal oxide coating (Aniołek, 2017). It has been reported that prolonged thermal oxidation results in the deposition of more uniform and thicker thermal oxide coatings with superior surface properties (Aniołek *et al.*, 2018). Therefore, sufficiently higher thermal oxidation temperature and time were selected in this work. This might be because the oxidation temperature plays a considerable role in oxide layer deposition. With the increase of oxidation temperature, the oxygen diffusion mechanism intensifies and causes the deposition of a thicker scale on the substrate (Aniołek, 2017).

During annealing, the Ti6Al4V substrate was kept for a longer time inside the furnace at higher temperature due to slow cooling. Higher temperature accelerates the reaction of the substrate with oxygen due to which the annealing process offered a thick oxide coating than the normalizing process. In normalizing, the cooling rate was greater than annealing. Therefore, the substrate cooled down to room temperature in a short time, compared to the annealing process. The air-quenching process involves throwing of accelerated air, having oxygen in excess quantity. Due to the presence of excess oxygen, and the higher temperature of the substrate, the substrate/oxygen reaction became very fast.

These thickness trends were further supported by the $\Delta W/A$ measurements. The consistently higher $\Delta W/A$ values at 900 °C compared to 850 °C align with previous findings (Aniołek *et al.*, 2018). It has been reported that the oxidation



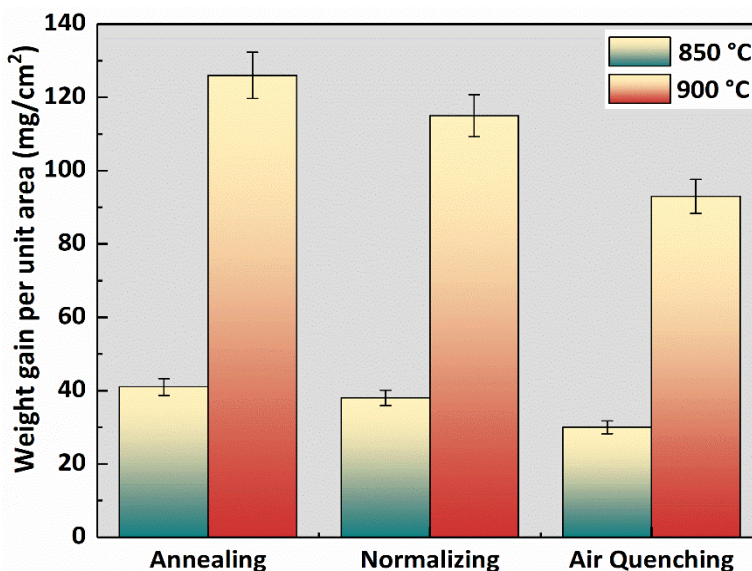


Figure 6. Variations in weight gain per unit area of thermal oxide coatings on Ti6Al4V alloy as a function of thermal oxidation treatments performed at 900 and 850 °C for 12 h

temperature had the most important and considerable impact on the intensity of growth of oxide coating, whereas oxidation time played a secondary role (James *et al.*, 2013; Aniolek, 2017). Therefore, the annealing process at 850 and 900 °C for 12 h provided the highest values of $\Delta W/A$ (41 ± 2.3 mg/cm², 126 ± 6.3 mg/cm²) but annealing at 850 °C deposited a coating with relatively lower $\Delta W/A$.

5. CONCLUSIONS

The effects of thermal oxidation temperature and treatment conditions on the morphological characteristics of thermal oxide coatings formed on the Ti6Al4V titanium alloy were systematically investigated. All thermally oxidized coatings exhibited continuous, well-adhered microstructures with a markedly reduced defect density. Coatings formed at 900 °C consistently displayed greater thickness and higher $\Delta W/A$ compared with those produced at 850 °C. Notably, the oxide coating obtained by

annealing at 900 °C exhibited the most uniform and defect-free morphology and consisted predominantly of TiO₂ in a rutile crystal structure. Although this coating possessed a moderate thickness of approximately 195 ± 9.75 μm, it showed the highest $\Delta W/A$ value (126 ± 6.3 mg/cm²) among all the investigated conditions.

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