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INVESTIGATION OF THE HIGH-TEMPERATURE OXIDATION BEHAVIOR OF TI-BASED ALLOYS USED IN THE AEROSPACE INDUSTRY

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ABSTRACT

In this study, the high-temperature thermal oxidation behavior of the $\text{Ti}_{68}\text{Nb}_{25}\text{Ta}_5\text{Mo}_2$ (at.%) alloy was comprehensively examined through thermogravimetric and differential thermal analysis (TG/DTA). The oxidation experiments were systematically carried out in ambient air to simulate service-like conditions. Uniformly dimensioned alloy specimens were initially subjected to non-isothermal oxidation up to 1000 °C in order to observe the general oxidation trend. Subsequently, isothermal oxidation treatments were conducted at 500, 750, and 1000 °C for a duration of 80 minutes to evaluate the temperature-dependent oxidation kinetics in detail. From the obtained data, the parabolic rate constants were determined as $k_{p500} = 2.54 \times 10^{-5} \text{ mg} \cdot \text{cm}^{-2} \cdot \text{s}^{-1}$, $k_{p750} = 9.27 \times 10^{-3} \text{ mg} \cdot \text{cm}^{-2} \cdot \text{s}^{-1}$, and $k_{p1000} = 9.52 \times 10^{-1} \text{ mg} \cdot \text{cm}^{-2} \cdot \text{s}^{-1}$, indicating a significant increase in oxidation rate with temperature. The kinetic analysis revealed that the thermal oxidation process followed a parabolic behavior, suggesting diffusion-controlled oxide scale growth. Furthermore, the apparent activation energy for the thermal oxidation of the alloy was calculated to be 171 kJ/mol, confirming the temperature sensitivity of the oxidation mechanism.

In addition, differential thermal analysis (DTA) did not exhibit any prominent endothermic or exothermic peaks, implying the absence of phase transitions or thermal events within the examined temperature range. Overall, the findings contribute to a better understanding of the high-temperature oxidation characteristics of Ti-based refractory alloys and provide valuable insights for their potential use in aerospace and high-temperature structural applications.

Keywords: Ti Base Alloys, Thermal oxidation behavior, Activation energy, Aerospace industry.

1 INTRODUCTION

The aerospace industry, encompassing both aviation and space sectors, is characterized by stringent requirements for high reliability, cost efficiency, and minimal operational risk. Consequently, the materials employed or under development for these applications must exhibit the highest possible levels of performance and conform to rigorous international quality standards. In particular, the selection of materials for aircraft manufacturing is not solely based on mechanical properties but is also directly influenced by thermal parameters and environmental conditions. As a result, materials intended for aerospace applications are subjected to a comprehensive and multidimensional evaluation process, taking into account structural integrity, long-term performance, and environmental sustainability.

Materials used in the aerospace industry are generally classified into two main categories: metallic materials and non-metallic (composite) materials. Among metallic materials, aluminum (Al) and its alloys are the most commonly used, followed by titanium (Ti), nickel (Ni), magnesium (Mg), and various steel alloys [1-12]. Among these, titanium and its alloys have gained significant attention for critical applications due to their high specific strength derived from their low density [13-16]. Elemental (pure) titanium is distinguished by its low thermal conductivity, relatively low density and elastic modulus, moderate mechanical strength, excellent corrosion resistance in various environments, and high chemical reactivity with a wide range of elements [17-21]. From a crystallographic perspective, titanium exhibits a hexagonal close-packed (HCP) structure at low temperatures, which transforms into a body-centered cubic (BCC) structure at elevated temperatures [22, 23]. The presence of two distinct crystal structures forms the foundation for the diverse properties exhibited by titanium alloys. As previously noted, titanium alloys offer numerous significant advantages compared to other materials; however, they remain among the more costly metallic materials utilized in contemporary aircraft fuselage manufacturing. To enhance their physical and chemical properties, various alloying elements are incorporated into Ti-based alloys. Niobium (Nb), a refractory metal with a high melting point of approximately 2468 °C, exhibits superior thermophysical properties [24-26]. Similar to all metals possessing a body-centered cubic (BCC) crystal structure, tantalum (Ta) demonstrates unique deformation behavior that is significantly influenced by impurities, alloying additions, crystal structure, temperature, and strain rate. Tantalum and tantalum-based alloys are noteworthy due to their high density, elevated melting points, excellent formability, good thermal conductivity, high fracture toughness retained even at low temperatures, corrosion resistance, and weldability, leading to

their increasing application in critical sectors such as the defense industry [27]. Molybdenum (Mo), which also crystallizes in a body-centered cubic structure, is utilized in small quantities in various alloys to enhance strength and corrosion resistance. Moreover, the addition of molybdenum lowers the β -phase transformation temperature of titanium [28, 29]. Thermal treatments constitute a crucial approach for improving the mechanical properties of these alloys. Additionally, Ti alloys are active materials whose mechanical properties are notably affected by the formation of oxide layers on their surfaces.

In this study, the high-temperature thermal oxidation behavior of the $\text{Ti}_{68}\text{Nb}_{25}\text{Ta}_5\text{Mo}_2$ alloy was thoroughly investigated, and the findings provide significant insights into the alloy's oxidation mechanisms as well as its performance evaluation in high-temperature applications.

2 MATERIAL AND METHOD

Metal powders of Titanium (Ti) with 99.5% purity, Niobium (Nb) and Tantalum (Ta) with 99.9% purity, and Molybdenum (Mo) with 99.0% purity were selected according to the atomic ratios specified in Table 1. To obtain the desired alloy composition, the metal powders were weighed by mass and homogeneously mixed. The mixing process was carried out using laboratory-type mixers with the aid of ceramic balls. The blended metal powders were then pressed into pellets with a diameter of 13 mm under a load of 5000 kgf. The pellets were alloyed via the arc melting method. To ensure the homogeneity of the alloy, the arc melting procedure was repeated three times. The thermal oxidation behavior of the alloy was investigated within the scope of this study. Specimens with equal dimensions of $1.2 \times 1.2 \times 1 \text{ mm}^3$ were cut and surface roughness was removed. For thermogravimetric and differential thermal analysis (TG/DTA) of alloys, the recommended sample mass is typically between 20 mg and 40 mg. Crucible type ceramic crucible was used. The heating rate was set at $20 \text{ }^\circ\text{C}/\text{min}$. The prepared samples were subsequently cleaned ultrasonically. Surface oxidation experiments were conducted using a Perkin Elmer Pyris TG/DTA thermal analysis system. The specimens were heated from room temperature up to $1000 \text{ }^\circ\text{C}$ in an air atmosphere, and mass gains were continuously recorded as a function of temperature throughout the experiment. The alloys exhibited logarithmic mass gain behavior at temperatures of $750 \text{ }^\circ\text{C}$ and above during non-isothermal experiments. Therefore, to enable a more accurate analysis of the oxidation kinetics, isothermal mass gain tests were conducted at temperatures within the logarithmic growth region, specifically at 500, 750, and $1000 \text{ }^\circ\text{C}$. During the experiments, high-purity argon gas (99.999%) was employed to provide an inert atmosphere and prevent oxidation outside the

intended conditions. The isothermal oxidation duration was set to approximately 80 minutes for each temperature. According to thermogravimetric (TG) analysis results, the mass gain was minimal at 500 °C (approximately 0.02 mg/cm²), showed a significant increase at 750 °C, and reached its maximum value of approximately 0.95 mg/cm² at 1000 °C. Based on these data, the thermal oxidation kinetics and activation energy of the alloy were calculated. The experimental findings provide valuable insights into the surface oxidation behavior of the alloy at elevated temperatures.

Table 1. Atomic and weight composition ratios of the constituent metals in the alloy

Sample Code	Atomic ratio (%)				Weight ratio (g)			
	Ti	Nb	Ta	Mo	Ti	Nb	Ta	Mo
TM	68	25	5	2	2,438	1,740	0,678	0,144

3 RESULT AND DISCUSSION

In order to determine the high-temperature thermal oxidation (HTTO) behavior of the prepared Ti-based Ti₆₈Nb₂₅Ta₅Mo₂ alloy, the samples were gradually heated from room temperature to 1000 °C in an air atmosphere under controlled conditions. The obtained mass change data are presented in Figure 1. The analysis revealed that the mass gain exhibited a linear trend up to approximately 450 °C, after which the mass gain followed an exponential increase. Based on these observations, the temperatures of 500 °C, 750 °C, and 1000 °C were selected as critical points for isothermal oxidation experiments. Additionally, the differential thermal analysis (DTA) results of the alloy are shown in Figure 2. The absence of any distinct phase transformation peaks in the corresponding graph indicates that the alloy remains structurally stable within the examined temperature range. These findings provide valuable insights into the alloy's oxidation behavior at elevated temperatures.

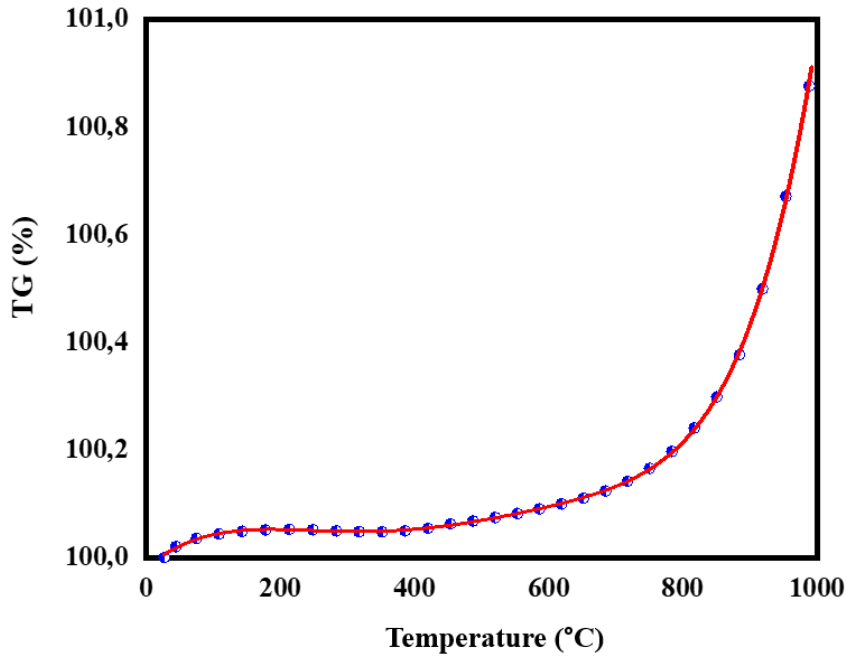


Figure 1. Mass gain curve of the Ti-based alloy from room temperature to 1000 °C.

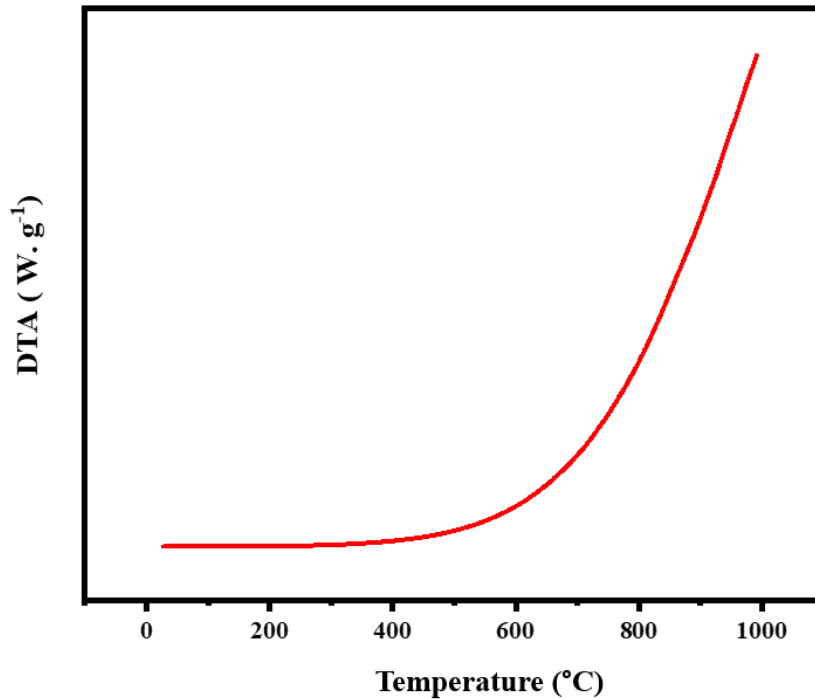


Figure 2. Mass gain curve of the Ti-based alloy from room temperature to 1000 °C.

Figure 3 presents the mass gain curves of the alloy as a function of time at selected temperatures. Examination of the graphs obtained at three different temperatures reveals a significant increase in the alloy's mass over time. This increase is notably more pronounced at elevated temperatures. The mass gain observed at 1000 °C, shown in Figure 3c, is considerably higher compared to other temperatures, exhibiting approximately a 9% increase after an 80-

minute isothermal oxidation process. Based on these data, the thermal oxidation kinetics of the alloy were analyzed, and the thermal oxidation rate constant (k_p) was calculated using the formula provided below [30, 31]:

$$(\Delta W / \Delta A)^2 = k_p \cdot t \quad (1)$$

In this context, $(\Delta W / \Delta A)$ denotes the ratio of the mass gain to the surface area of the sample, t represents the time parameter, and k_p corresponds to the thermal oxidation rate constant. The variation of $(\Delta W / \Delta A)^2$ as a function of time is meticulously illustrated in Figure 4. The thermal oxidation rate constant (k_p) was determined by analyzing the linear segment of this graph. Subsequently, using the derived k_p values, the activation energy (E_0) necessary for the thermal oxidation process of the alloy was calculated through Equation 2. These calculated numerical results are comprehensively compiled in Table 2. Furthermore, to elucidate the temperature dependence of the oxidation kinetics, Figure 5 depicts the Arrhenius plot of $\ln(k_p)$ against $1000/T$, providing valuable insight into the thermal behavior of the alloy.

$$k_p = k_o \cdot \exp (E_o / RT) \quad (2)$$

T represents the temperature, while R denotes the universal gas constant with a value of $8.3144 \text{ J/mol} \cdot \text{K}$.

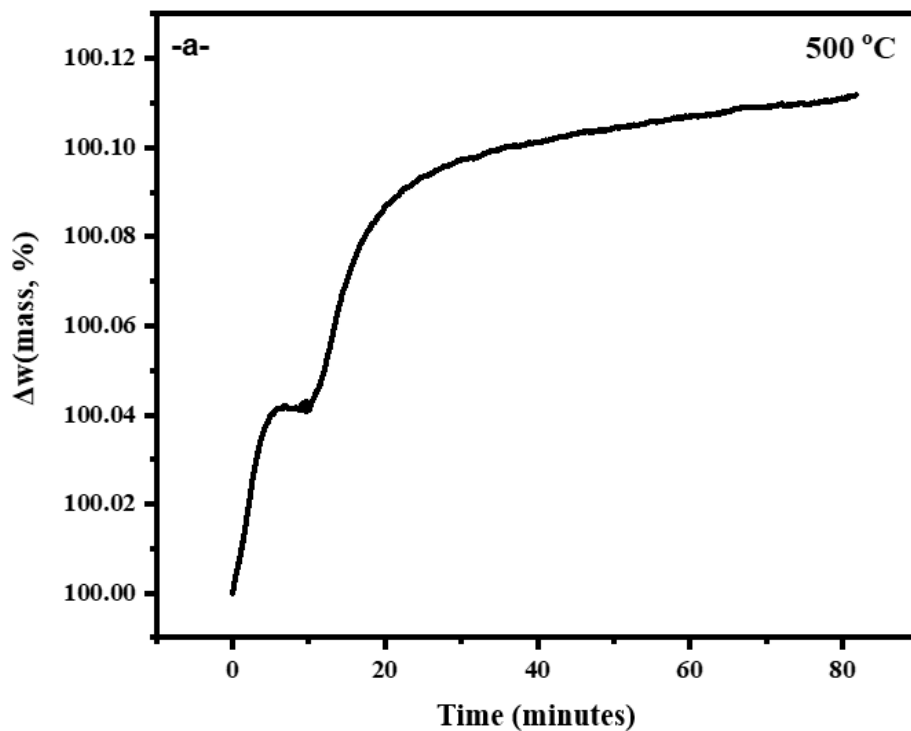


Figure 3. Time-dependent isothermal mass gain curves of the alloy in air atmosphere at a) 500 °C, b) 750 °C, and c) 1000 °C.

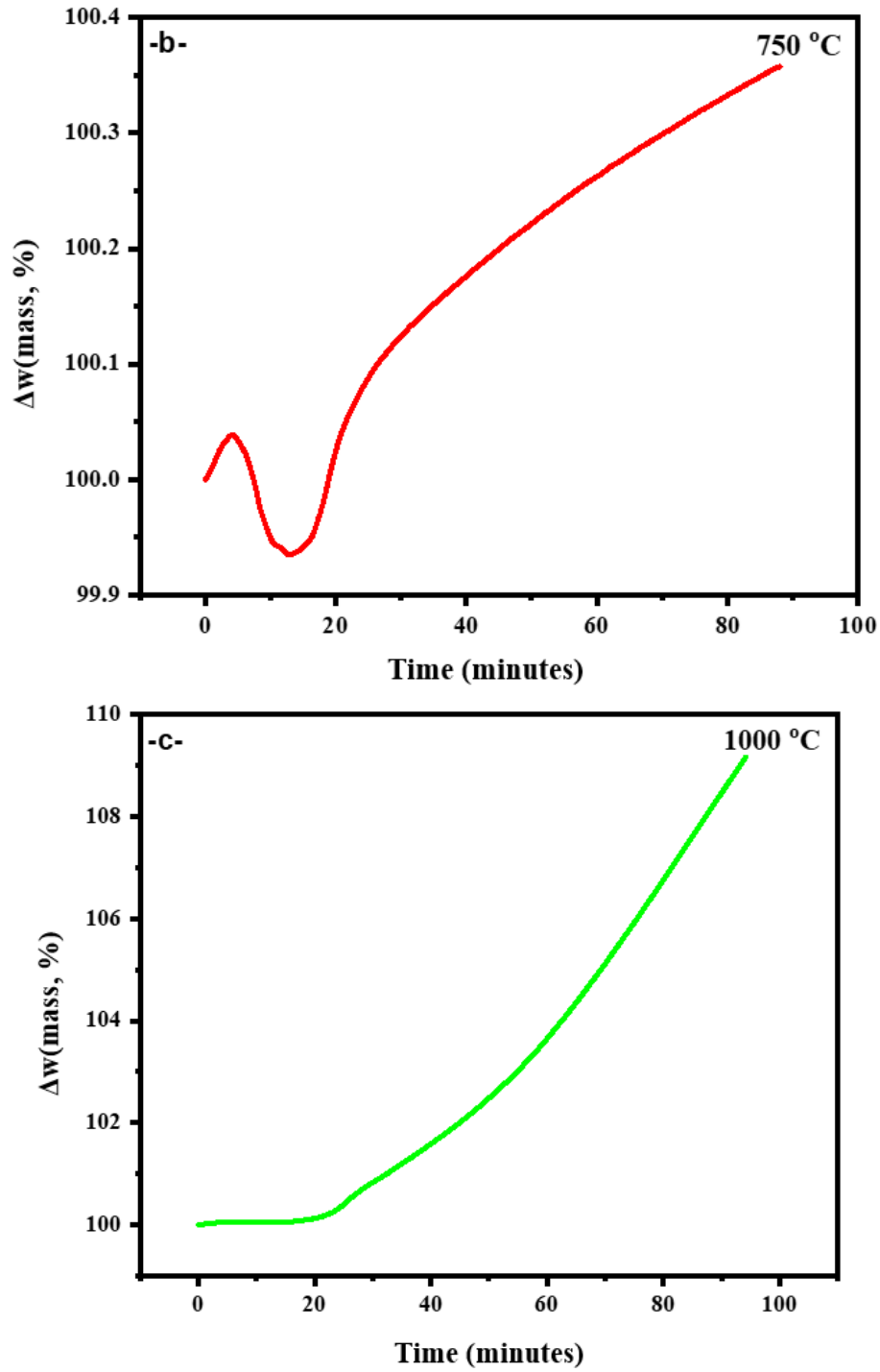


Figure 3 (Continued). Time-dependent isothermal mass gain curves of the alloy in air atmosphere at a) 500 °C, b) 750 °C, and c) 1000 °C.

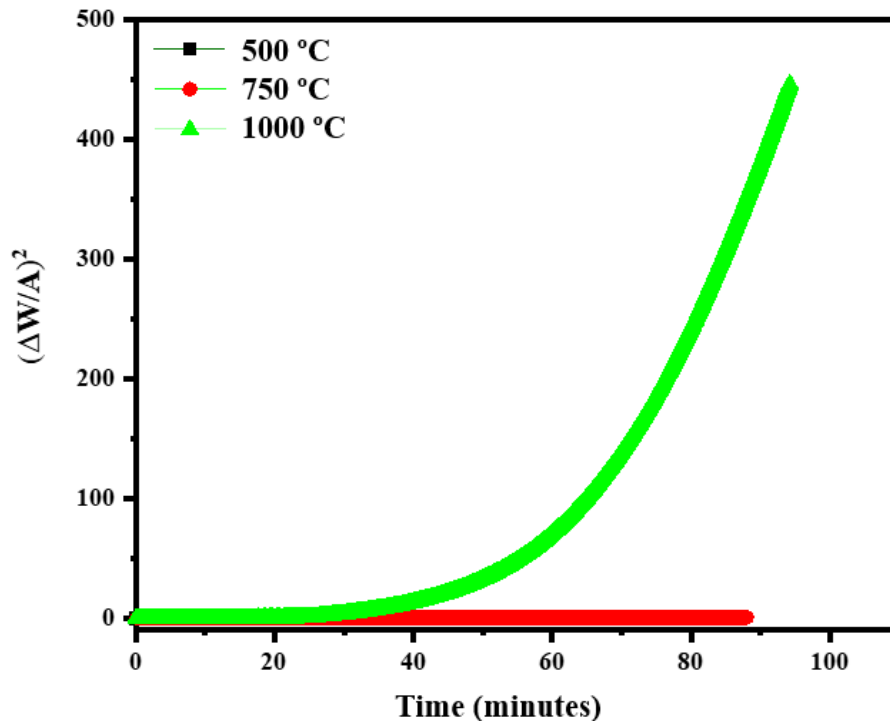


Figure 4. Variation of $(\Delta W/A)^2$ as a function of time.

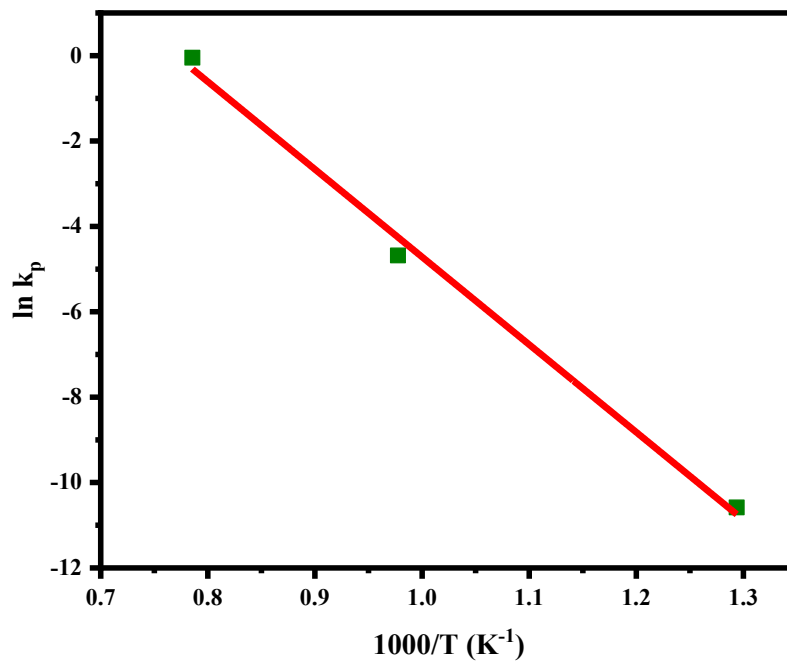


Figure 5. Arrhenius plot of $\ln(k_p)$ versus $1000/T$.

Table 2. Calculated k_p and activation energy values of the alloy

Sample	Oxidation Temperature (°C)	k_p (mg.cm ⁻² s ⁻¹)	Activation Energy (kJ. mol ⁻¹)
TM	500	2.534×10^{-5}	171
	750	0.927×10^{-2}	
	1000	0.953	

Table 3 and Figure 6 present a comparative overview of the activation energy values for thermal oxidation of various Ti and Ti-based alloys, all determined using the same experimental methodology. This comparison provides valuable insights into the oxidation behavior of these materials at elevated temperatures. The findings clearly indicate that the activation energy required for the thermal oxidation of the $\text{Ti}_{68}\text{Nb}_{25}\text{Ta}_5\text{Mo}_2$ alloy synthesized in the present study (<200 kJ/mol) is significantly lower than that reported for many high-performance alloys in the literature.

Table 3. Comparison of the activation energy value of the alloy with those of selected alloys obtained by the same method

Alloy	Eo (kJ/mol)	Reference
Ti-25Nb-5Ta-2Mo	171	This study
Fe-15Cr-0.5Si	256	[32]
Ti-29Ni-21Nb	142	[33]
Ti-28Ni-22Nb	173	[33]
Ti-27Ni-23Nb	186	[33]
Ti-6Al-7Nb	191	[34]
Ti-6Al-2Sn-4Zr	157	[35]
Ti-6Al-2-Sn-4Zr-2Mo	242	[36]
V-4Ti-1Ta	179	[37]
V-4Ti-3Ta	182	[37]
V-4Ti-7Ta	189	[37]
Ti-25Al-18Ta	259	[38]
Ti-26Al	255	[39]
Ti-32Al	295	[39]
Ti-34Al	299	[39]

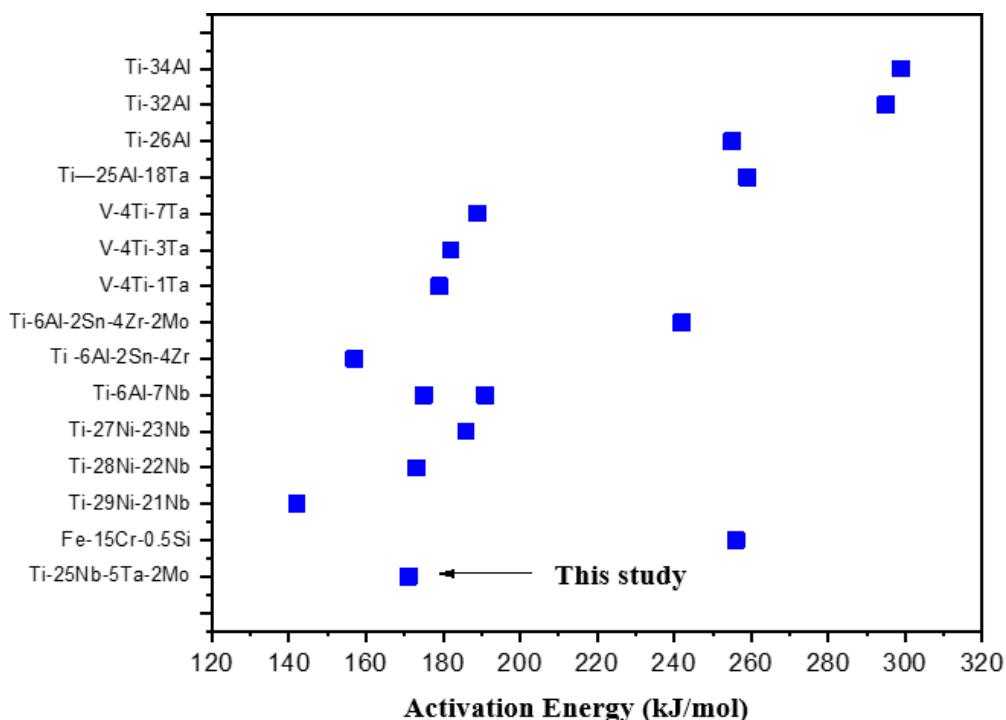


Figure 6. Activation energy values for the thermal oxidation of various alloys obtained using the same method.

For instance, activation energies reported for Ti-34Al, Ti-32Al, and Ti-26Al alloys range between 260 and 280 kJ/mol [39]. Such high activation energy values imply limited atomic mobility, suggesting that oxygen diffusion and/or metal ion transport is thermodynamically restricted in these alloys. As a result, these materials exhibit excellent resistance to oxidation even at elevated temperatures, making them highly suitable for demanding thermal environments such as jet engine components and gas turbines. In contrast, medium-activation-energy alloys such as V-4Ti-4Cr, V-4Ti-4Ta, and Ti-6Al-2Sn-4Zr-2Mo exhibit values in the range of approximately 210-250 kJ/mol. These alloys provide a balanced engineering profile, offering both adequate high-temperature oxidation resistance and favorable formability and processability, which are critical for manufacturing applications. On the other hand, alloys with low activation energy values-such as Fe-15Cr-0.5Si, Ti-29Nb-21Nb, Ti-27Nb-23Nb, and Ti-28Nb-22Nb-typically exhibit activation energies in the range of 150-190 kJ/mol [40-43]. Within this context, the $\text{Ti}_{68}\text{Nb}_{25}\text{Ta}_5\text{Mo}_2$ alloy developed in this study, with an activation energy measured below 200 kJ/mol, can be classified among these low-energy alloys. This relatively low activation energy suggests enhanced atomic diffusion of oxygen through the alloy matrix, which may lead to faster oxidation kinetics and potentially limited structural stability at high temperatures. However, a low activation energy should not always be interpreted negatively. In certain cases, such as for bio-compatible alloys, rapid surface oxidation may facilitate the formation of a passive oxide layer that promotes cellular adhesion. Similarly, these alloys may offer distinct advantages in applications requiring operation at moderate or low temperatures, including medical implants and microelectronic components. In conclusion, the $\text{Ti}_{68}\text{Nb}_{25}\text{Ta}_5\text{Mo}_2$ alloy developed in this work, with its relatively low thermal oxidation activation energy, may not be ideal for long-term use in extreme thermal environments. Nevertheless, it holds significant potential for applications that demand rapid surface reactions at moderate temperatures, such as in biomedical or electronic fields. The comparative analysis provided here offers critical insights into the suitability of the alloy for specific application domains based on its thermal oxidation behavior.

4 CONCLUSION AND SUGGESTIONS

In this study, the thermal oxidation behavior of the TiNbTaMo alloy exposed to air at elevated temperatures was systematically investigated, leading to the following significant findings:

The Ti-based TiNbTaMo alloy exhibits rapid surface oxidation above 450 °C, where the alloy surface becomes distinctly covered with a metal oxide layer once this temperature threshold is exceeded. As the temperature increases, oxygen diffusion not only affects the surface but also penetrates deeper into the alloy, resulting in oxidation reactions within the bulk structure.

It was observed that the oxidation rate increases with temperature, showing an exponential growth trend above 450 °C. This indicates that temperature plays a critical role in the kinetics of oxygen penetration into the metal, suggesting a diffusion-controlled oxidation mechanism predominates.

The thermal oxidation rate constant (k_p) values were found to increase with rising temperature, a trend that was corroborated by the activation energy calculations. Based on the obtained data, the activation energy required for the thermal oxidation of the TiNbTaMo alloy was determined to be approximately 171 kJ/mol.

This calculated activation energy is considerably lower compared to those reported for alloys developed for high-temperature applications such as Ti-34Al, Ti-32Al, and Ti-26Al, which typically exhibit activation energies in the range of 260-280 kJ/mol in the literature. This comparison suggests that the TiNbTaMo alloy has a higher tendency for oxidation at elevated temperatures and consequently may possess limited structural stability under prolonged high-temperature exposure.

Nevertheless, the relatively low activation energy may confer advantages to the TiNbTaMo alloy in specific applications. Particularly, alloys exhibiting efficient surface passivation are desirable for biomedical applications as well as systems operating at moderate to low temperatures, where rapid formation of protective oxide layers can be beneficial. In particular, alloys that exhibit efficient surface passivation are highly desirable for biomedical devices, such as orthopedic implants, dental implants, and cardiovascular stents, where rapid formation of a stable oxide layer enhances corrosion resistance, biocompatibility, and long-term durability. Additionally, these alloys may be suitable for moderate- to low-temperature engineering systems, including aerospace components, power electronics, and chemical processing equipment, where the prompt development of protective oxide layers can mitigate oxidation-related degradation and extend service life. Such characteristics make TiNbTaMo alloys promising candidates for applications where both corrosion resistance and thermochemical stability under mild operating conditions are critical.

In conclusion, although the TiNbTaMo alloy demonstrates a certain degree of resistance to surface oxidation at high temperatures, its low activation energy necessitates careful consideration of structural stability for long-term high-temperature applications. However, this characteristic also renders the alloy a promising candidate for niche applications-especially in biomedical fields or technologies operating under lower temperature regimes. To extend the present findings, future studies should focus on alloying and microstructural modifications to improve oxidation resistance, alongside surface engineering approaches such as coatings or thermochemical treatments to enhance long-term stability. The use of advanced in-situ techniques (e.g., synchrotron XRD, TEM, spectroscopy) is recommended to clarify oxidation kinetics and mechanisms, while comparative assessments under varied environments and application-oriented testing in biomedical and moderate-temperature conditions will be essential to validate the alloy's practical viability.

Conflict of Interest Statement

There is no conflict of interest between the authors.

Statement of Research and Publication Ethics

The study is complied with research and publication ethics.

Artificial Intelligence (AI) Contribution Statement

This manuscript was entirely written, edited, analyzed, and prepared without the assistance of any artificial intelligence (AI) tools. All content, including text, data analysis, and figures, was solely generated by the authors.

Contributions of the Authors

Esra BALCI: Data analysis, writing and editing.

Büşra TUNÇ: Analysis, software and writing original draft.

Fethi DAĞDELEN: Conceptualization, supervision, methodology

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