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Study Surface Modification by Bioactive Titanium Dioxide Coating Formed through Chemical and Annealing Treatment of Ti6Al7Nb Alloys

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Ti-6Al-7Nb alloy implants are widely used in biomedical applications due to their excellent mechanical properties and biocompatibility. Surface modification techniques further improve their long-term performance, enhancing functionality and reliability. In this study, a titania (TiO_2) layer was formed on Ti-6Al-7Nb specimens through chemical treatment using an H_2O_2/0.1 M HCl solution at 80 ºC, followed by annealing at 350 - 600 ºC. The modified surfaces were characterized using Scanning Electron Microscopy (SEM), microhardness (HV), surface roughness (Ra), contact angle, X-ray Diffraction (XRD), and Potentiodynamic Polarization (PDP) tests. SEM and XRD analyses revealed that annealing between 350 ºC and 600 ºC gradually transformed the titanium dioxide layer into the anatase crystalline phase, with rutile formation beginning at 550 ºC. At this temperature, microhardness increased and surface roughness decreased, indicating enhanced surface characteristics. Electrochemical testing in 0.9% NaCl (normal saline) at 37 ºC using the PDP method showed that the specimen heat-treated at 500 ºC exhibited the lowest corrosion rate among all conditions. These results suggest that 500 ºC represents an optimal heat-treatment temperature for producing TiO_2 coatings with a well-balanced combination of crystallinity, surface wettability, and corrosion resistance.

Keywords: *Ti6Al7Nb alloys, Surface Modification, Titania layer, Corrosion rate*

1. Introduction

Biomedical components, composed of materials that are synthetic or natural, are often utilized as biological structures for implantation to restore or enhance the quality of life for individuals with missing or faulty biological structures [1,2] as shown in Fig. 1. The design of an implant material should aim to ensure adequate durability, functionality, and a favorable biological response. While durability and functionality are primarily determined by the material's bulk features, the biological response is largely governed by surface characteristics involving topography, roughness, surface chemistry, wettability, and surface charge. At the interface between the implant and the host tissue, a range of biological interactions occurs. These interfacial responses define the biocompatibility of the implant, which is a critical factor influencing its overall success [3,4]. Biocompatible implant materials must be non-toxic, non-carcinogenic, with small or no response of the host body as well as have chemical stabilization and corrosion resistant [5-7]. Titanium and its alloys are renowned for their exceptional material features, involving high specific strength, excellent corrosion resistance, and low density, making them highly versatile across various industries. However, in many industrial applications, these inherent features may not fully meet the demanding operational requirements [8]. Consequently, enhancing the mechanical, tribological, and chemical features of titanium alloys becomes essential to ensure optimal performance under diverse working conditions [9]. Titanium and its alloys offer significant advantages over steels in medical applications, involving low weight, excellent corrosion resistance in harsh environments, low density, low thermal conductivity, non-magnetic behavior, and good processing workability. These features make them highly attractive for biomedical use [10-12]. Furthermore, compared to other metallic

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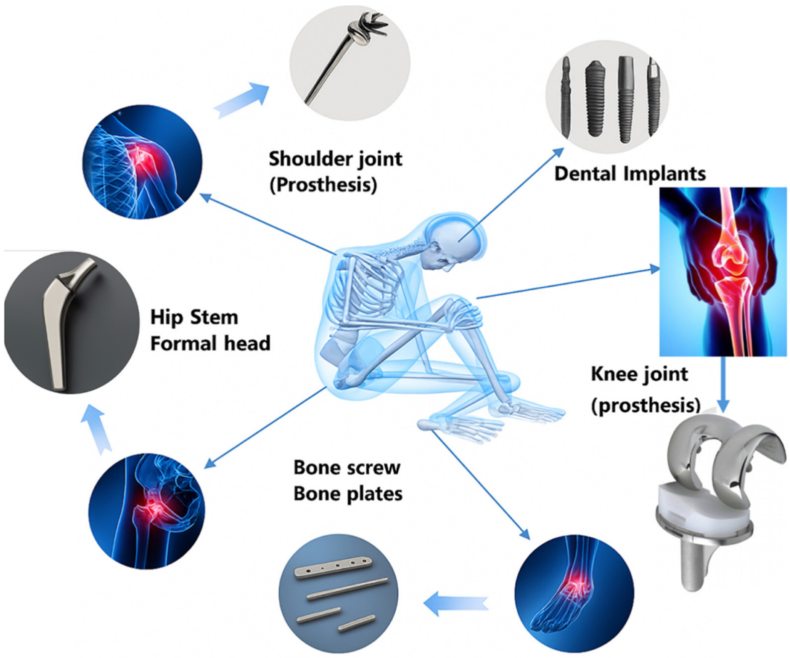

Fig. 1. Various components kinds of human implants [24]

materials, titanium and its alloys exhibit superior biological inertness, maintaining biocompatibility even over extended periods of use [13,14]. Titanium and its alloys are known for several advantageous features, including excellent corrosion resistance, even in bodily fluid environments. Ti-6Al-4V was long considered the most utilized alloy in the biomedical field. However, concerns over adverse tissue reactions and the potential toxicity caused by the in vivo release of vanadium ions have led to its reduced use. To address this issue, the Ti-6Al-7Nb alloy was developed, in which vanadium is replaced by the more biocompatible element niobium [2]. Alloying elements can be generally classified as alpha (α) or beta (β) stabilizers. Advancements in the field have shifted focus toward α + β-type titanium alloys with elevated β-phase content (5 - 30 vol.%) [15]. These alloys provide enhanced formability, moderate high-temperature strength, and the capability for heat treatment to tailor mechanical performance. Their properties can be precisely controlled through adjustments in alloy composition, heat treatment parameters, and cooling rates [16]. Based on the nature of the alloying components, the transition temperature of pure titanium rises or lowers [17]. In recent decades, research has increasingly concentrated on β-type titanium alloys (β-Ti) due to their lower elastic modulus, which closely matches that of human bone, thereby mitigating the risk of stress shielding [18]. Ti-6Al-7Nb is a titanium alloy consisting of a hexagonal α phase (stabilized by aluminum) and a body-centered cubic β phase (stabilized by niobium) [19]. The morphology and fractional volume of the phases in the alloy are mostly influenced by conditions encountered throughout the manufacturing process [20]. The enhanced wear and corrosion resistance of the Ti-6Al-7Nb alloy, in contrast to Ti-6Al-4V, is mostly since the production of Nb_2O_5. This oxide exhibits greater chemical stability, reduced solubility, and enhanced biocompatibility compared to V_2O_5, which develops on the Ti-6Al-4V surface [20,21]. The incorporation of niobium enhances passivation by promoting the development of a niobium-rich pentoxide layer. Electrochemical tests conducted in fetal bovine serum have demonstrated that Ti-6Al-7Nb offers improved corrosion resistance and reduces the release of titanium and aluminum ions, making it a safer alternative to Ti-6Al-4V [22]. Since its high biocompatibility, little response to surrounding tissues, and good corrosion resistance, titanium alloys are frequently utilized as biomaterials. The various implant applications. Both medical and dental fields, that include in artificial hip and knee joint replacements and dental implant prosthesis [23].

The surface of a material has a critical role in determining the biological response to artificial medical devices [25]. Surface modification not only alters the surface's appearance but also enhances features involving adhesion, micro-cleaning, amine functionalization, and overall biocompatibility [26]. Through surface modification, a varied range of surface types can be engineered to elicit specific biological responses in targeted cell or tissue environments, aiming to reduce healing time and minimize adverse reactions [27]. Titanium and its alloys, despite their widespread biomedical use, suffer from poor tribological features, including low wear resistance, which can shorten the service life of implants. Surface coatings are a practical solution to this issue, with surface engineering having a vital function in improving the performance and durability of titanium-based orthopedic devices beyond their inherent limitations [15]. Surface modifications can generally be classified into various categories. Numerous techniques have been developed to enhance the physicochemical and biological features of titanium-based materials. Among these, methods that result in the formation of titanium dioxide (TiO_2) layers are particularly noteworthy, as TiO_2 offers excellent corrosion resistance, biocompatibility, and promotes osseointegration. Mindroiu *et al.*, [28] demonstrated that osteoblasts cultured for 48 hours on Ti-6Al-7Nb alloy discs showed good adhesion and morphology comparable to cells on plastic controls. Notably, cell density was significantly greater on anodized, chemically, or thermally treated samples than on untreated ones. Additionally, the MTT assay confirmed 100% cell viability, indicating the absence of cytotoxic effects from ion release. These results underscore the beneficial impact of surface treatments on the biological performance of Ti-6Al-7Nb alloys and supporting the current study's emphasizing the importance of surface modifications in improving implant biocompatibility [26]. Common surface modification techniques include chemical and physical treatments, laser cladding, thermal oxidation, plasma spraying, and ion implantation [29-31].

The novelty of this research lies in its systematic approach to optimizing the formation of anatase and rutile phases of TiO_2 on the Ti-6Al-7Nb alloy surface through controlled thermal treatment after chemical oxidation. Unlike conventional studies focusing on Ti-6Al-4V, this work emphasizes the safer, more biocompatible Ti-6Al-7Nb alloy. It identifies 500 ºC as the optimal annealing temperature for achieving a homogeneous anatase TiO_2 layer with superior electrochemical and mechanical performance. Furthermore, it provides a detailed correlation between phase composition, microstructure evolution, surface wettability, and corrosion behavior offering a comprehensive insight into tailoring implant surface features through thermo-chemical processing. This study aims to investigate the surface modification of biomedical-grade Ti-6Al-7Nb alloy through a combination of chemical oxidation utilizing H_2O_2/HCl solution and annealing heat treatments at various temperatures (350 - 600 ºC) to form a bioactive TiO_2 coating. The objective is to enhance the surface characteristics of the alloy—specifically its corrosion resistance, microhardness, surface roughness, wettability, and bioactivity to optimize its performance for biomedical implant applications.

2. Materials and Methods

The Ti-6Al-7Nb alloy rod was cut by utilizing a wire-cutting machine into ϕ13 × 3 mm dimension, degreased with acetone, then rinsed with distilled water. A chemical oxidation treatment has been performed by immersing the specimens in a solution composed of 8.8 M H_2O_2 and 0.1 M HCl, maintained at 80 °C on a hot plate for 60 minutes, the method of X.X. Wang *et al.* Following the oxidation process, the samples were thoroughly rinsed with distilled water and dried in an oven at 90 °C. Subsequently, a thermochemical treatment was applied by heating the samples in a furnace at 350, 400, 450, 500, 550 and 600 ºC for 120 minutes, with a controlled heating rate of 10 ºC/min. The NABERTHERM furnace is utilized for thermochemical treatment. After chemical oxidation, this step promotes the formation of crystalline oxide phases on the sample surface, enhancing their surface features for biomedical application. The test of X-ray diffraction analysis (XRD- 6000) had been taken to describe the coating specimens, Cu Kα radiation (λ = 1.5405 Å) were utilized in the XRD, with a scanning speed of 3º/min from (20º to 90º) of 2θ and a power of Kv/30 mA. The contact angle test was performed to assess the wettability of coating samples, in accordance with the ASTM D7334-08 [32]. Utilizing normal saline (0.9%

NaCl), the contact angle (θ) of droplets on the solid surface was measured. Contact angle measurements were conducted utilizing the CAM 110-04W optical equipment, which includes a CCD camera, readings were taken immediately after dispensing a (1) µL droplet onto the solid surface. Specimen surface morphology was examined using a TESCAN VEGA3 scanning electron microscope (Serial No. 118-0014), operated at an accelerating voltage of 10 kV. Tafel potentiodynamic polarization tests were performed in a 0.9% NaCl solution using a DY-2323 bi-potentiostat at a scan rate of 1 mV/s. A conventional three-electrode cell was employed, in which the sample (1.0 cm^2 exposed area) served as the working electrode, an Ag/AgCl electrode as the reference, and a platinum electrode as the counter electrode. The corrosion rate (C.R.) is calculated utilizing Faraday's Law, derived from Tafel plots of both anodic and cathodic branches. This measurement follows ASTM G102-2004 standards [33]. The corrosion rate (C.R.) is determined by utilizing the following formula.

$$C.R = \frac{O.13\ I_{corr.}\left(E.W\right)}{A.\rho} \tag{1}$$

Where:

C.R = Corrosion rate, E. W. = equivalent weight (g /eq.), A = area (cm^2), ρ = density (g/cm^3) ,0.13 = metric and time conversion factor, $I_{corr.}$ = The total current (µA) .

In this study, a commercial Ti-6Al-7Nb alloy (ASTM F1295) was utilized. The material was provided as rods with a diameter of 10 mm, manufactured through hot rolling and subsequently annealed. The room-temperature mechanical properties of the Ti-6Al-7Nb alloy are listed in Table 1.

The bulk Young's modulus remains unchanged during tensile testing of samples subjected to short oxidation durations, as the oxidation does not penetrate sufficiently to alter the substrate's microstructure. This highlights that the crystalline phase of titanium oxide predominantly influences the surface mechanical properties without causing a significant effect on the overall elastic modulus of the alloy [34].

3. Results and Discussion

3.1 The chemical composition

A rod of Ti-6Al-7Nb alloy made in (Shanxi Joint industry co., ltd), was utilized to prepare the substrates. The chemical composition of this alloy is demonstrated in Table 2.

The results demonstrate that the alloy composition is in full compliance with ASTM specifications. Aluminum (Al) and Niobium (Nb) are the primary alloying elements. Al serves as an alpha-phase stabilizer, enhancing the alloy's strength and creep resistance. Nb, a beta-phase stabilizer, improves corrosion resistance and biocompatibility without inducing cytotoxic effects, making it a safer alternative to vanadium utilized in the Ti-6Al-4V alloy. The compositional balance of this alloy makes it suitable for orthopedic and dental implant applications, where both mechanical performance and biological safety are critical.

3.2 XRD Analysis

Fig. 2 presents the XRD pattern of the Ti-6Al-7Nb sample treated with an H_2O_2/HCl solution at 80 ºC for two hours, followed by heat treatment at various temperatures ranging from 350 ºC to 600 ºC, reveal significant phase transformations dependent on the annealing temperature. At 350 - 400 ºC, the diffraction peaks are weak and broad, indicating a poor crystalline

Table 1. Mechanical properties of Ti–6Al–7Nb alloy at room temperature

YS (MPa)	UTS (MPa)	TE (%)	AR (%)	E (GPa)	HV
800	900	10	25	103	280

YS: yield stress; UTS: ultimate tensile strength; AR: area reduction; TE: total elongation. E: Young's modulus.

Table 2. Chemical composition analysis of the Ti-6Al-7Nb alloys

Element	Al	Si	Fe	Ni	Nb	Ti
ASTM (wt%)	5.5-6.5	≤ 0.31	≤ 0.25	≤ 0.31	6.5-7.5	Rem.
wt%	6.12	0.11	0.21	0.29	7.26	Rem.

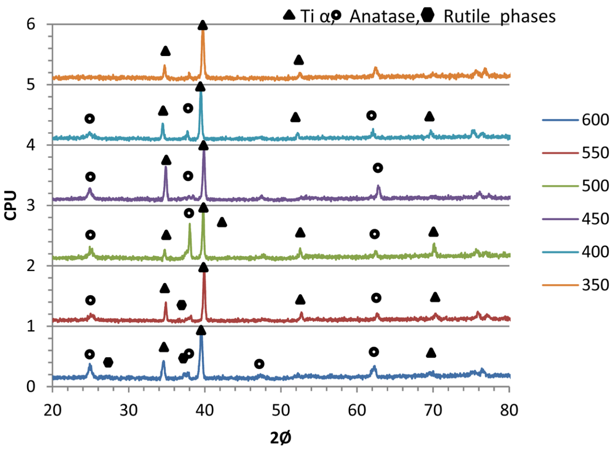

Fig. 2. XRD patterns of the Ti-6Al-7Nb sample after chemical treatment, followed by heat treatment at various temperatures

or amorphous surface layer. With the increasing temperature to 450 ºC, peaks corresponding to the anatase phase begin to emerge, demonstrating the onset of crystallization. The sample treated at 500 ºC exhibits the most intense and well-defined anatase peaks, indicating that this temperature promotes optimal formation of the anatase TiO_2 phase, which is known for its superior bioactivity and surface reactivity, anatase surfaces present a higher density of Ti–OH groups, enhancing fibronectin adsorption this in turn promotes cell adhesion, ultimately facilitating osseointegration compared to the rutile phase [35]. At 550 ºC, peaks corresponding to the rutile phase start to appear alongside anatase, suggesting the initiation of the anatase-to-rutile phase transformation. As the temperature increases to 600 ºC, the rutile phase becomes increasingly dominant, with a corresponding decrease in anatase peak intensity. This confirms the progressive transformation of anatase into the more thermodynamically stable rutile phase at higher temperatures. Therefore, 500 ºC is identified as the optimal heat treatment temperature for achieving a dominant anatase phase, making it favorable for biomedical applications where enhanced osseointegration is desired.

3.3 Optical Observation

Fig. 3 demonstrates microstructure of Ti-6Al-7Nb alloy substrates with two magnifications. The microstructure demonstrated a duplex microstructure consisting of the near Equiaxed alpha (α) (white phase) and transformed beta (β) (dark phase).

Optical microscopy images (600**x**) of samples subjected to chemical and heat treatments at 500 ºC and 600 ºC (Fig. 4) reveal distinct corrosion behaviors. In Fig.7b, pitting corrosion is observed at microstructural defect sites, where chloride ions locally disrupted the passive TiO_2 film. The presence of cracks, attributed to the heat treatment, likely acted as preferential initiation sites for localized attack. In contrast, Fig. 4a shows regions with a stable and compact TiO_2 oxide layer exhibiting uniform corrosion, characterized by the gradual highlighting of grain boundaries without severe localized damage. These microstructural observations are consistent with the corresponding Tafel plots Fig. 5, which indicate that areas with defects display higher anodic currents and susceptibility to pitting, whereas intact oxide layers maintain lower corrosion rates and uniform dissolution. Overall, the results demonstrate that the integrity and homogeneity of the TiO_2 passive layer are decisive in governing the corrosion mechanism: microstructural imperfections promote localized pitting, while a stable, compact oxide film favors uniform corrosion with improved resistance to chloride-induced degradation.

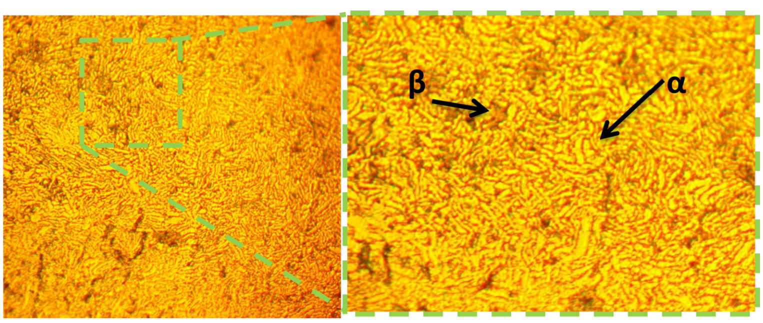

Fig. 3. Optical Micrographs of Ti6Al7Nb Alloy with two magnifications (200X &600X)

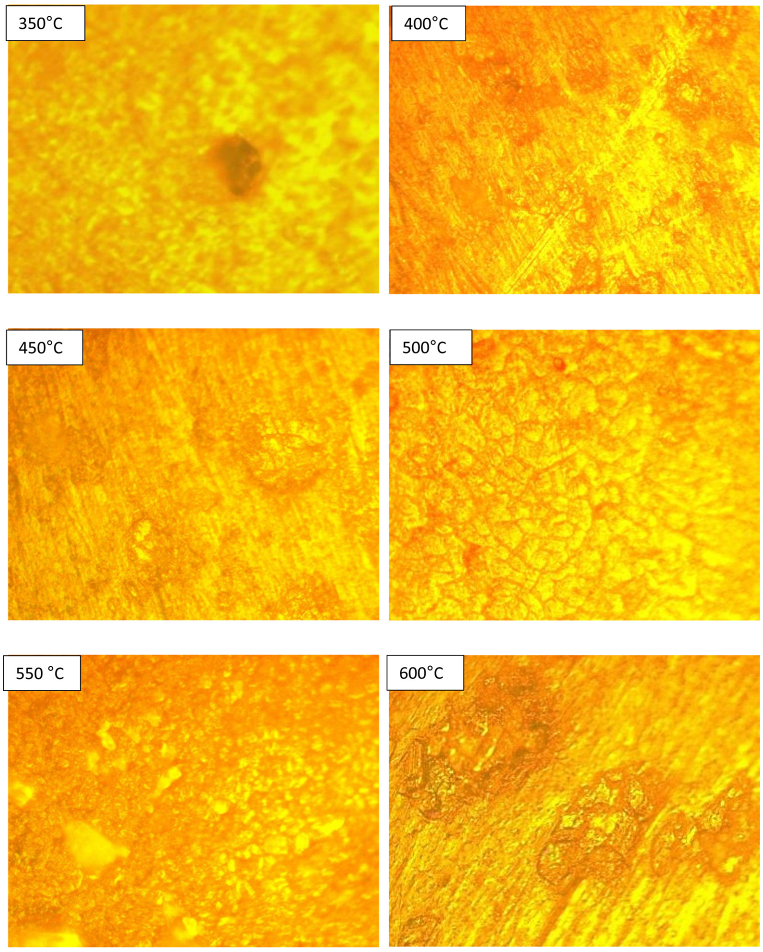

Fig. 4. OM images of the titanate layers on the Ti6Al7Nb surfaces treated with the H_2O_2/HCl solution at 80 °C for 60 min and subsequently heat treated after corrosion

3.4 Corrosion Behavior Tests

Fig. 5 demonstrate the potentiodynamic polarization curves for the TiO_2 coated of Ti 6Al-7Nb alloy by a chemical and annealing heat-treatment, coatings prepared at after various heat treatment temperature in Normal saline (0.9%NaCl) at 37 °C±1. The corrosion current densities (icorr.) and corrosion potentials (Ecorr.) are derived from the potentiodynamic curves by employing Tafel extrapolation and it is listed in Table 3. Furthermore, the corrosion rates (CR) were also included in Table 3, and it calculated according to the equation (1).

Coatings formed at temperatures between (350 - 400) °C and (550 - 600) °C exhibit higher corrosion current deviations, indicating increased corrosion activity. This suggests that both low and high treatment temperatures lead to coatings with reduced corrosion resistance. At 350 °C, the formation of an amorphous or incomplete oxide layer is likely, the surface is still inhomogeneous and may contain micro-defects or porosity. Corrosion resistance is expected to be low, consistent with the Tafel curve results while at 400 °C, the initial development of an active oxide layer begins. The optimal coating appears

Table 3. Electrochemical factors of the potentiodynamic polarization curves

Annealing temp.	$I_{corr.}$ (μA/cm^2)	$E_{corr.}$ (mV)	CR (mpy)
350	1.173	-27	0.410
400	2.531	-11	2.531
450	1.173	27	0.410
500	0.507	143	0.178
550	0.979	112	0.339
600	11.14	23	3.903

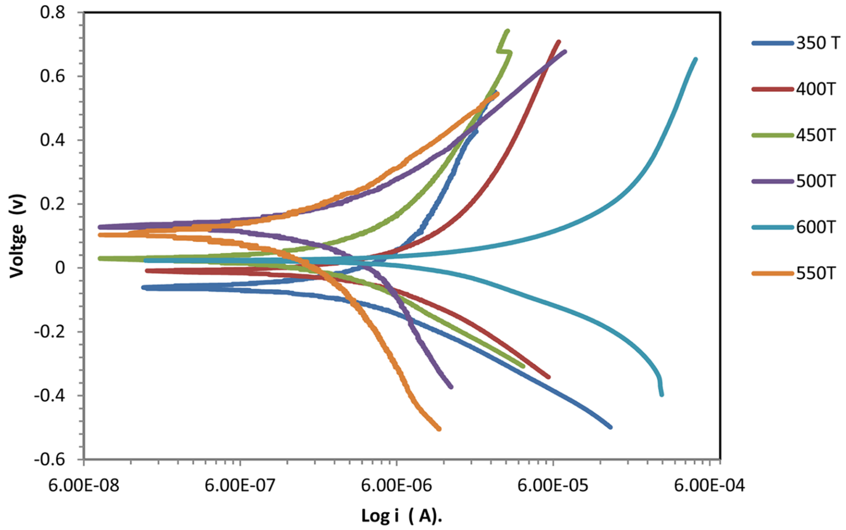

Fig. 5. Potentiodynamic polarization curves on Ti-6Al-7Nb alloy of different Temp. of heat treatment of TiO_2 coatings

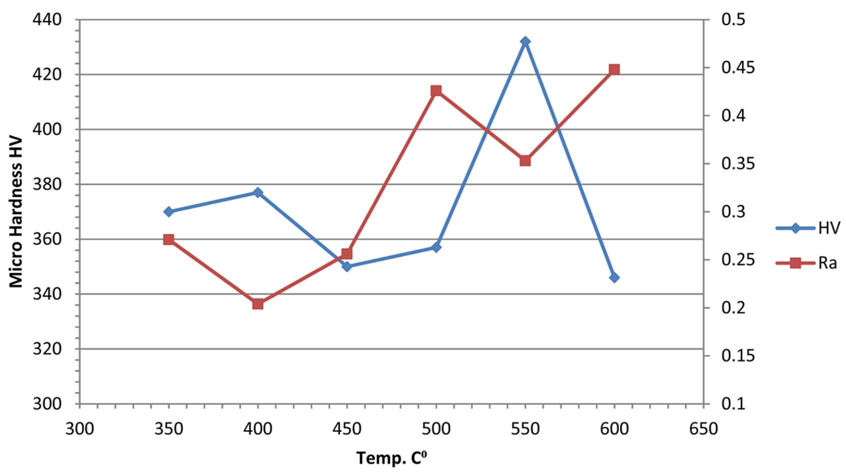

Fig. 6. Relationship between the Micro hardness (HV), Surface roughness (Ra) and different Temp. of heat treatment of TiO_2 coatings

at 450 ºC, where a well-defined anatase phase is strongly formed. At 500 ºC, a stable oxide layer with favorable protective features is observed. However, from (550 to 600) ºC, the corrosion current magnitudes increase again, likely since the transformation of anatase into the rutile phase, as confirmed by XRD patterns demonstrated in Fig. 2.

3.5 Micro Hardness and Roughness

The micro-hardness and surface roughness of the ceramic-coated specimens were evaluated under an applied load of 50 g (0.49 N) with a constant dwell time of 10 s. The results show a distinct improvement in the hardness of the coated surfaces as the annealing temperature increases, indicating the beneficial role of thermal treatment in enhancing the mechanical integrity of the coating. Fig. 6 shows that the hardness values of all specimens subjected to chemical treatment followed by heat treatment at temperatures ranging from 350 to 600 ºC are higher than that of the untreated Ti-6Al-7Nb substrate (280 HV). The hardness reaches its maximum at 550 ºC, which can be attributed to the formation of a dense, compact oxide layer that strongly adheres to the substrate.

3.6 Contact Angle Test

Contact angle is a significant measurement technique to assess material surface wettability was also found to

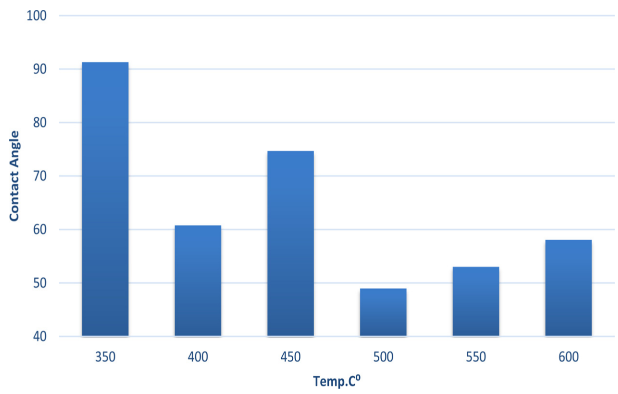

Fig. 7. Relationship between Contact angle and different Temp. of heat treatment of TiO_2 coatings

be another important method for assessing the bioactivity of titanium surfaces. Fig. 7 demonstrates the contact angles tested of TiO_2 by chemical and thermochemical treatment; coatings prepared after various heat treatment temperature in Normal saline (0.9%NaCl). The sample exhibits a decrease in contact angle accompanied by increased surface roughness as the heat-treatment temperature is raised to 500 ºC. The contact angle reached magnitude to 48.9557º and increased with increase heat treatment temperature, from Fig. 7 heat treatment at 500 ºC favors the formation of the anatase phase of TiO_2 where anatase is known to be more hydrophilic than the rutile phase, promoting lower contact angles and better water spreading. Notably, all temperatures from (350 to 600)ºC yielded surfaces classified as hydrophilic, since the contact angle between the surface and water droplet remained below 90º.

3.7 Scanning Electron Microscopy Image Analysis of Different TiO_2 Coatings

The SEM images presented in Figs. 8 a-q illustrate the surface morphology of TiO_2-coated Ti-6Al-7Nb alloys subjected to chemical and annealing heat treatments at different temperatures. In the 350 ºC, the images of SEM (Figs. 8a, b) indicate that the coating layer is amorphous and non-homogeneous, which aligns with the XRD results demonstrating the absence of the characteristic peak at 25.3º, typically associated with the anatase. The microstructure at 400 ºC demonstrates the beginning of anatase phase formation, with some TiO_2 still in an amorphous form. The TiO_2 coating is not uniform and contains pores, as demonstrated in Fig. 8c, d, and e. At 450 ºC, the TiO_2 coating displays a well-defined crystalline anatase phase. However, the surface remains non-uniform and rough, with visible cracks, as in Fig. 8f.

At 500 ºC, the coating exhibits a well-crystalline anatase phase, the temperature is generally considered ideal for producing homogenous, rough surface texture, and minimal visible cracking of TiO_2 as demonstrated in Figs. 8g, h

Between 550 ºC and 600 ºC, the rutile phase begins to form, as confirmed by the XRD patterns. At 550 ºC, the coating surface appears more homogeneous and smoother, as demonstrated in Fig. 8 m, n, which aligns with Fig. 4, this is likely since the higher thermal stability of the rutile phase compared to the anatase phase. At 600 ºC, the coating surface appears non-homogeneous and rough. The results of the study, within the temperature range of 350 - 600 ºC, indicated that microcracks falling within the permissible roughness range (1 - 2 µm) may contribute to enhancing biocompatibility by improving osseointegration through increasing the contact area between the bone and the implant, thereby facilitating osteoblast adhesion, proliferation, and growth. However, excessive or uncontrolled crack formation may compromise both mechanical integrity and biocompatibility, adversely affecting the functional performance of the implant [36,37]. As demonstrated in Figs. 8 k, q, the cracks observed at this temperature are likely caused by thermal stresses or shrinkage resulting from phase transformation.

4. Conclusion

In this study, Ti-6Al-7Nb alloy subjected to chemical surface treatment utilizing H_2O_2/0.1 M HCl at 80 ºC followed by heat treatment, can develop a titanium dioxide (TiO_2) coating. The key observations and inferences from this study are as follows:

The phase transformation of TiO_2 progresses from an amorphous structure at 350 ºC to the anatase phase at (400 - 500)ºC, and further to a mixture of anatase and rutile phases at (550 - 600)ºC during heat treatment.

The highest micro-hardness magnitude of 432HV was achieved at 550 ºC, accompanied by a 17.8% reduction in surface roughness, likely since the initiation of rutile phase formation.

Electrochemical testing demonstrated that the coating

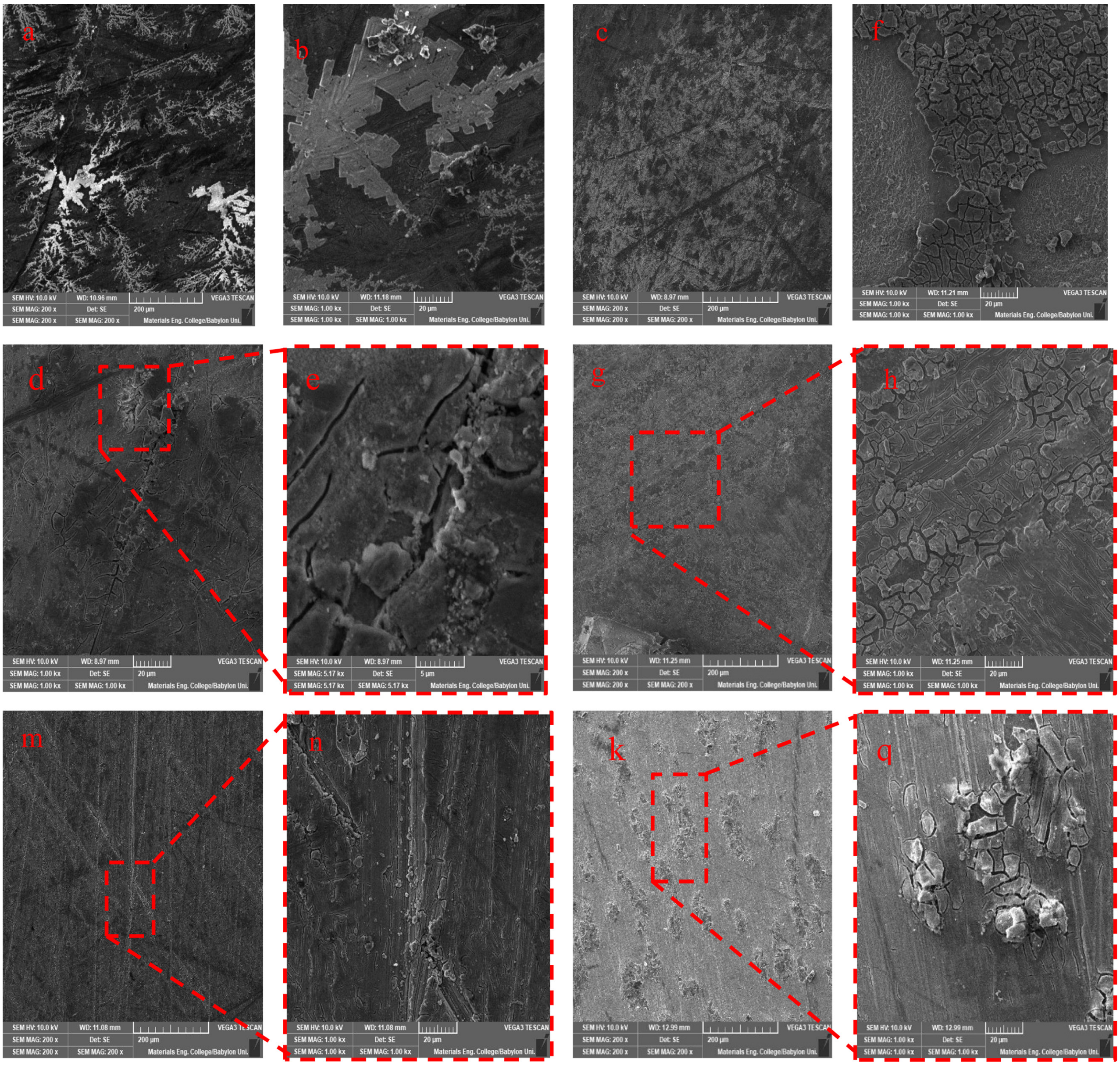

Fig. 8. SEM images of the titanate layers on the Ti6Al7Nb surfaces treated with the H_2O_2/HCl solution at 80 ºC for 60 min and subsequently heat treated at: a, b) 350 ºC, c, d and f) 400 ºC, e) 450 ºC, g and h) 500 ºC, m and n) 550 ºC and k and q) 600 ºC for 1 h, indicating the change in morphology of the TiO_2 layers

treated at 500 ºC exhibited the lowest corrosion rate of 0.178 mpy, while the highest corrosion rate of 3.39 mpy was observed at 600 ºC of heat treatment.

The optimal temperature is 500 ºC for producing TiO_2 coatings with balanced crystallinity, corrosion resistance, Surface roughness, microhardness and wettability.

These findings support the potential of thermos-chemically treated Ti-6Al-7Nb implants for improved performance in biomedical applications.

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