The Effect of Anodic Oxidation Voltages on the Color and Corrosion Resistance of Commercially Pure Titanium (CP-Ti)

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ABSTRACT

The effect of anodic oxidation (or anodizing) voltages on the color and corrosion resistance of pure titanium was studied. In this experiment, a commercially-pure titanium was used. To create an "illusion of color," anodizing process modified the oxide layer on the surface of titanium. Because of an interference effect similar to that of a prism, the titanium oxide layer gives the perception of color. Light reflects at different angles from the oxide layer and the underlying titanium, and these reflections interfere with each other. Certain wavelengths of light cancel or merge, resulting in the perception of color from the remaining light. It was concluded that when we use a higher voltage to anodize the titanium, the corrosion rate will increase. It was observed that using 10V gives us the most optimum resistance. For surface roughness, using 40V in both KOH and DAP solutions give us the highest roughness data. Due to the high amount of voltage used in anodizing the titanium, the coating got thicker in the surface and it affects the roughness. The data have shown that the corrosion rate and surface roughness were inversely proportional.

Keywords: anodic oxidation, anodizing, titanium, corrosion, biomedical

1. INTRODUCTION

Commercially Pure Titanium (CP-Ti) is considered to be the best biocompatible metallic material because its surface properties result in the spontaneous build-up of a stable and inert oxide layer. Titanium can be alloyed with iron, aluminum, vanadium, and molybdenum, among other elements, to create strong, lightweight alloys. Coloring is also commonly used for easy identification [1]. The parameters that affect oxide growth and give the titanium a variety of colors are the cell voltage, current density, duration of treatment, temperature, electrolyte composition, pH, substrate composition, concentration, and agitation effect of solution [2].

There are three different types of anodizing on titanium. Type 1 is utilized in specialist high-temperature treatments and is significantly less frequent. Type 2 is mostly used for wear protection as it shields the metal surface from the effects of wear. Type 2 anodizing creates a hard, wear-resistant surface that reduces seizing and friction between sliding titanium surfaces.

Moreover, patients with orthopedic implants benefit from type 2 anodized titanium's reduced friction or lubricity, which improves joint mobility. Lastly, titanium color anodizing is another name for type 3 titanium anodizing. In the medical application, type 3 color anodizing is commonly utilized for instant visual identification of parts. For example, an orthopedic surgeon in the middle of a procedure can simply request a blue bone screw. Effect of applied voltage on the thickness layer of Ti-6Al-4V shown in Fig. 1, and on the other hand, effect of applied voltage on the microstructure shown in Fig. 2 [3].

Titanium alloys are utilized in airplanes components, armor plating, naval ships, spacecraft, and missiles because of their high tensile strength to density ratio, strong corrosion resistance, fatigue resistance, and ability to sustain fairly high temperatures without creeping. Titanium is alloyed with aluminum, zirconium, nickel, vanadium, and other elements for these purposes, and is used to make a variety of components such as vital structural parts, fire walls, landing gear, exhaust ducts (helicopters), and hydraulic systems [4].

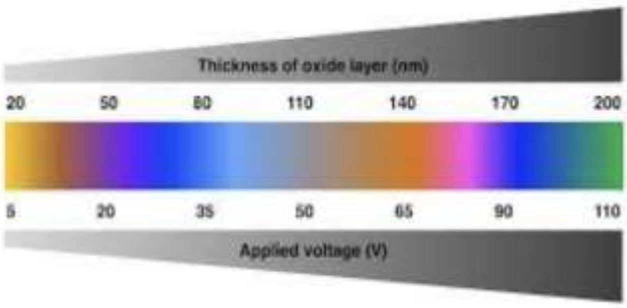

Fig 1. Effect of applied voltage on the thickness layer [2].

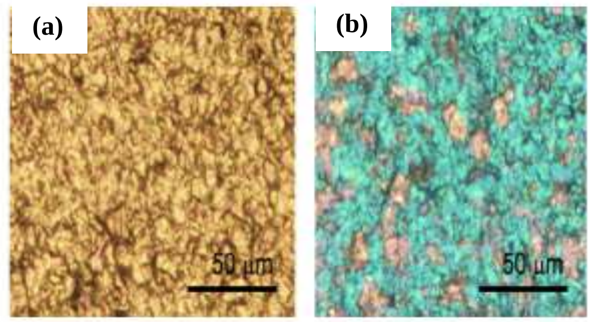

Fig 2. Oxides were grown at (a) 10V and (b) 80 V [2].

The chemical and petrochemical industries rely on welded titanium pipe and process equipment for corrosion resistance. For their high strength, corrosion resistance, or both, specific alloys are employed in oil and gas downhole applications and nickel hydrometallurgy. Titanium is used in pulp and paper industry process equipment that is exposed to corrosive media such as sodium hypochlorite or wet chlorine gas in ultrasonic welding, wave soldering, and sputtering targets are some of the other applications.

Titanium is also employed in automotive applications, particularly in auto and motorcycle racing, where low weight, great strength, and rigidity are required. Tennis rackets, golf clubs, lacrosse stick shafts, cricket, hockey, lacrosse, and football helmet grills, bicycle frames and components are all made of titanium. Titanium bikes have been employed by racing teams and adventure riders, despite the fact that it is not a common bicycle material.

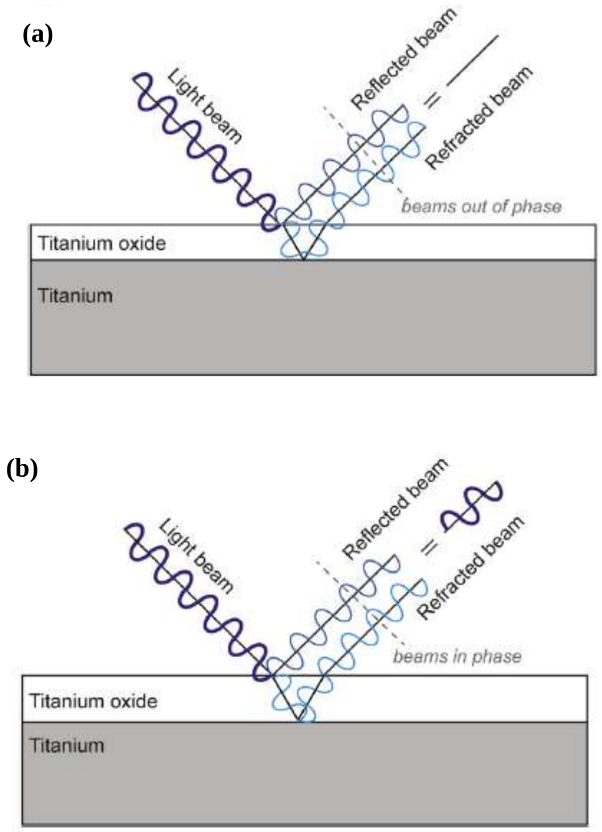

Fig 3. Thin layer interference of the light waves

If oxide thickness is such that reflected and refracted beams become out of phase by half of their wavelength, this produces color elimination as shown in Fig. 3(a) [5]. An example of blue wavelengths out of phase: the resulting oxide will appear yellow/reddish. Vice versa, if the phase displacement is an integer number of wavelengths the corresponding color appears at the surface as shown in Fig. 3(b). An example of blue wavelengths in phase: the resulting oxide will appear blue [5].

In addition, because titanium is biocompatible (meaning it is non-toxic and does not cause the body to reject it), it is used in a variety of medical applications, including surgical instruments and implants such as hip balls, sockets (joint replacement) and dental implants that can last up to 20 years as shown in Fig. 4 [6]. There are specific properties of material used in medical implants. Researchers have to make sure their products reach the required standards in order for it to be used as shown in Table 1.

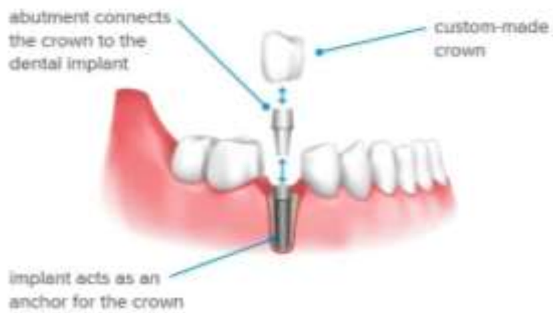

Fig 4. The parts in dental implants

Table 1. Properties of material used in medical implants

	ASTM Grade				
Property	1	2	3	4	5
Yield Strength (MPa)	170	275	380	483	795
Ultimate Tensile Strength (MPa)	240	345	450	550	860
Elongation (%)	24	20	18	15	10
Elastic Modulus (GPa)	103–107	103–107	103–107	103–107	114–120

*Adapted from ASTM F67 (Grade 1 to 4) and F136 (Grade 5)

Titanium's natural propensity to integrate to the jawbone allows it to be used in dental implants that can last up to 30 years. This characteristic is also beneficial in the case of orthopedic implants. The lower modulus of elasticity of titanium allows these devices to better match the elasticity of the bone they are meant to mend. Therefore, a lot of doctors used these titanium surgical instruments in the medical industry [7].

This research will focus on the type 3 anodization. It aims to observe the effect of anodic oxidation voltages on the color and corrosion resistance of pure titanium.

2. RESEARCH SIGNIFICANCE

This research focuses on the effect of anodic oxidation voltage on the color and corrosion resistance of pure titanium. Titanium is widely used in applications such as aerospace, maritime, and chemical industries. It is also employed in automotive, sports, and medical applications.

Commercially Pure Titanium (CP-Ti) is suitable as a biocompatible metallic material due to its surface properties that result in a stable oxide layer. Coloring is used for identification purposes. Parameters affecting oxide growth and titanium color include cell voltage, current density, treatment duration, temperature, electrolyte composition, pH, substrate composition, concentration, and solution agitation. There are three types of anodizing on titanium, with type 2 providing wear protection and benefits for orthopedic implants.

3. RESEARCH METHODS

3.1 Material

The material used in this study was a Grade 4 Commercially Pure Titanium (CP-Ti). It has been cut in the size of 4 cm x 1 cm x 0.2 cm in size as shown in Fig. 5.

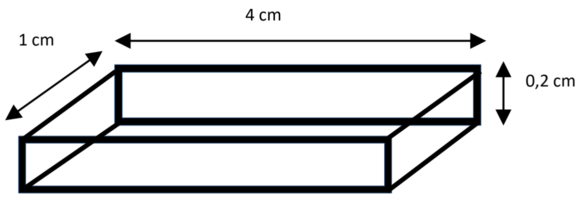

Fig 5. Size of sample

3.2 Experimental Procedure

The experimental set up as shown in Fig. 6. It consists of a DC power source, a beaker for electrolytic bath and platinum as cathode and test samples of anode. The pre-treatment includes grounding the specimen using #240 up to #2000 abrasive papers before being polished with alumina suspension. The specimen was then washed in DI water before being etched in 24,5 ml of sulfuric acid and 4 ml of hydrochloric acid. The process was continued by degreasing the titanium pieces in acetone and ethanol for 3 minutes in an ultrasonic bath.

Fig 6. Anodizing set up

Before the anodizing process, one cleaned titanium piece was examined under the Keyence microscope to test the average surface roughness, the results were shown in Table 2.

Table 2. Average surface roughness data after first cleaning

Area	Surface roughness (μm)
1	3.213
2	3.448
3	2.107
4	2.548
Average of surface roughness	3.213

Anodizing was done in a 58,1g of Potassium Hydroxide (KOH) solution at 25°C for 1 minute at 5, 10, 20, 30, and 40 V voltage variations. A digital multimeter was used to record the current output while anodizing. The sample was washed in DI water after anodization. Next, repeat the step before but this time change the electrolyte using 132,05g of Di-Ammonium Hydrogen Phosphate.

An optical microscope called the Keyence Microscope was used to examine the anodic oxide coating that was formed. Corrosion behaviors were also qualitatively evaluated by the potentiostat/galvanostat using simulated body fluid (SBF) solution so that a polarization curve was concluded [8]. It was placed with ringer lactate substance which is a mixture of sodium chloride, sodium lactate, potassium chloride, and calcium chloride in water. Ringer lactate has the same concentration level as the human system and is usually used for replacing fluids and electrolytes in those who have low blood volume or low blood pressure [9].

4. RESULTS AND DISCUSSION

After the anodization, the average surface roughness of the titanium sample was observed using a Keyence microscope. The data was collected and plotted on a table as shown in Table 3.

Table 3. Average roughness of titanium after the anodization

Sample	Surface roughness (μm)
S0	0.383
S1 (KOH, 10V)	0.432
S2 (KOH, 20V)	0.495
S3 (KOH, 30V)	0.790
S4 (KOH, 40V)	0.772
S5 (KOH, 5V)	0.509
S6 (DAP, 10V)	0.662
S7 (DAP, 20V)	0.480
S8 (DAP, 30V)	0.567
S9 (DAP, 40V)	0.915
S10 (DAP, 5V)	0.555

The color produced by anodizing the titanium at various voltages, as well as the thickness data, are shown in Fig. 7. After the anodizing, the substrate retained its metallic reflecting aspect, indicating that the anodic coating was transparent. When Potassium Hydroxide solution is used, the anodic film turned gold when exposed to a low voltage of 5 V. After the anodizing at 20 V, the surface look changed to a light blue hue, and after the anodizing at 40 V, it changed back to a gold color. When the Di-Ammonium Hydrogen Phosphate is used, the anodic film turned gold when first exposed to a voltage of 5 V.

After the anodizing at 20 V, it changed to a dark blue color, and lastly, after anodizing at 40 V, it turned into a light blue color. Light interference was formed by the transparent anodic material, which resulted in color fluctuation based on the reflecting light waves. The optical characteristics of the anodic film formed changed when the anodizing voltage was changed. The hue of the interference was determined by the number of pores and the thickness of the film.

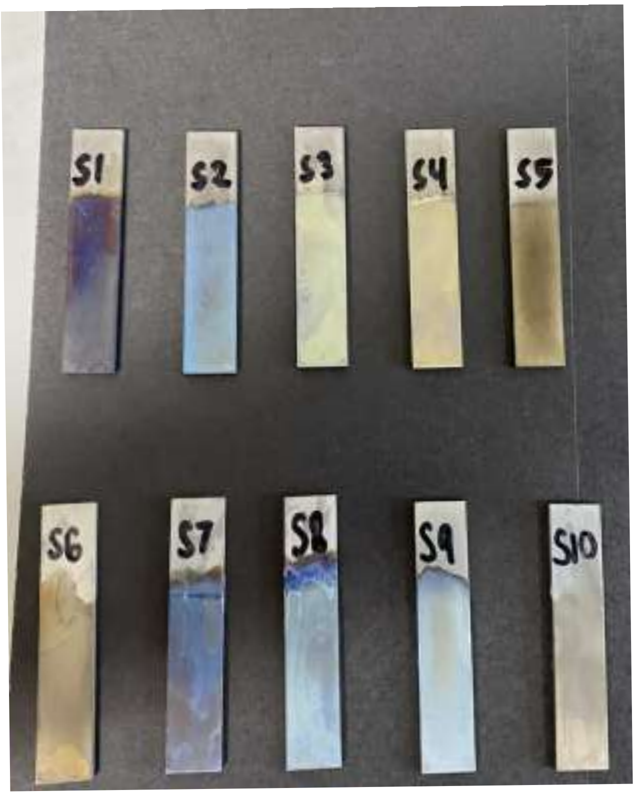

Fig 7. Color of titanium surface after anodizing at various voltage

Table 4. Voltage, ampere and solution were used

Sample	Voltage (V), Ampere (A), and Solution
S1	10V, 0.1A, Potassium Hydroxide
S2	20V, 0.1A, Potassium Hydroxide
S3	30V, 0.2A, Potassium Hydroxide
S4	40V, 0.2A, Potassium Hydroxide
S5	5V, 0.1A, Potassium Hydroxide
S6	10V, 0.03A, Ammonium Hydrogen Phosphate
S7	20V, 0.02A, Ammonium Hydrogen Phosphate
S8	30V, 0.02A, Ammonium Hydrogen Phosphate
S9	40V, 0.01A, Ammonium Hydrogen Phosphate
S10	5V, 0.011A, Ammonium Hydrogen Phosphate

The surface morphology of the anodic oxide coating was evaluated using the Keyence microscope. As illustrated in all of the photos in Fig. 8 using Potassium Hydroxide solution and Fig. 9 using Di-Ammonium Hydrogen Phosphate solution, the anodic film that resulted was porous. Pores are frequently found in anodic oxide films. As shown in Fig. 9, the film generated had a grainy structure. As a result of anodizing at higher voltages 20V, 30 V, and 40V, the grain structure vanished and the film became more compact. With increasing anodizing voltage, the number of pores increased.

Sample	Magnified 120x	Magnified 240x
S0 Without Treatment		
S1 KOH, 10 V		
S2 KOH, 20 V		
S3 KOH, 30 V		
S4 KOH, 40 V		
S5 KOH, 5 V		

Fig 8. Images of anodic oxide films formed on titanium using Potassium Hydroxide

S6 DAP, 10 V		
S7 DAP, 20 V		
S8 DAP, 30 V		
S9 DAP, 40 V		
S10 DAP, 5 V		

Fig 9. Images of anodic oxide films formed on titanium using Di-Ammonium Hydrogen Phosphate

To determine the corrosion rate of each sample, namely by conducting corrosion testing with the potentiodynamic method. From the experimental data, Icorr results are obtained. Polarization curves are also acquired shown in Fig. 10. From the results, it is concluded that the thicker the oxide layer covered on the titanium piece, the better it protects the metal surface.

Sample	Chart Diagram	Corrosion Rate (mmpy)	Icorr (μA)
S0 Without Treatment		1.8898	4.348
S1 KOH, 10 V		1.1293	2.598
S2 KOH, 20 V		1.1948	2.749
S3 KOH, 30 V		1.4092	3.422
S4 KOH, 40 V		1.1505	2.647

Fig 10. Polarization curves of titanium

5. CONCLUSIONS

When the Titanium was anodized at 5,10, 20, 30, and 40 V, it resulted in the color variety of gold, violet, dark blue, and light blues. The anodic film's pore density grew when the voltage was applied, resulting in a lighter tint. The change in voltage also resulted in the pores density of the film to alter, which caused light interference in the film thickness and gave protection to the metal from corrosion. When we apply a greater voltage to anodize the titanium, the corrosion rate increases. It has been discovered that utilizing 10V provides the best corrosion resistance. The coating thickens in the surface due to the high voltage employed in anodizing the titanium, which influences the roughness. corrosion rate and surface roughness are inversely proportional.

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7. AUTHOR CONTRIBUTIONS

Conception and design: Ika Marial Ulfah
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Writing publication: Kandice Felisha Kurniawan, Ika Maria Ulfah, Muhammad Kozin
Approval of final publication: Ika Maria Ulfah, Muhammad Kozin
Resources, technical and material supports: Ika Maria Ulfah, Muhammad Kozin
Supervision: Muhammad Kozin

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