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Color Formation on Titanium Surface Treated by Anodization and the Surface Characteristics: A Review

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Abstract

Tooth loss is a common problem found in human beings. The challenge in replacing teeth is to restore the natural tooth appearance. Dental implants have overcome this challenge. Titanium (Ti) is an attractive material for dental implants due to its excellent properties and biocompatibility, but the grayish appearance is a concern. A process of supplying the current voltage to an electrochemical cell forming the oxide layer on metal is called anodization. Anodization is a surface modification technique that creates micro- and nano-roughness and an oxide layer on the Ti surface, improving the properties of dental implants. The higher the voltage applied to the anodizing process, the higher the anodic film thickness forms. The different thicknesses of titanium oxide (TiO_2) film cause variations in refractive index and reflective index, resulting in a desired color on the Ti surface. The most useful colors for dental application have been gold (10 V), yellow (60 to 65 V), and pink (70 to 80 V) in hue. Yellow and gold hues were mostly used in restoration or abutment areas while pink hue was used for gingival areas. The purposes of this literature review are to assess the current knowledge on the anodization process of titanium and its characteristics and to provide an overview of the literature on color formation on Ti surfaces by anodization.

Keywords: anodization, dental implant, titanium

Introduction

Since teeth are important organs for human beings, missing teeth can cause severe damage to other systems in the body in addition to affecting the quality of life as a role of esthetics, function, and speech.[1] Nowadays, replacing missing teeth is accomplished with either removable or fixed prostheses.

Dental implants are one of the treatment options that has become a standard in replacing teeth with a better outcome[1], wherein several materials have been used and developed for dental implants. Titanium (Ti) and its alloys are impressive because of their excellent biocompatibility, physical, mechanical, and chemical properties.[2] The excellent biocompatibility and long-term clinical survival rates of titanium have made this material the gold standard for dental implants.[3] Despite the material used, surface characteristics are another point of concern for good adaptation and biomechanical fixation.[4] Surface characteristics could be enhanced by several techniques of surface modification to provide micro- and nano-level roughness.[4]

Anodization or anodic oxidation technique is a great choice for surface modification as it provides a proper surface for osseointegration while being one of the techniques that produce an oxide layer on the Ti surface.[5-8] This can also overcome the disadvantage of titanium's grayish appearance by interference color from the oxide formation. The applied voltage has varied to form a thicker titanium oxide (TiO_2) layer, resulting in a more desired color on the Ti surface.[9,10]

The purposes of this literature review are to assess the current knowledge on the anodization process of Ti and its characteristics and to provide an overview of the literature on color formation on Ti surfaces by anodization.

Titanium in dentistry

An important consideration for dental implant success is a good adaptation between the implant surface and the surrounding tissue, both soft and hard.[11] Since the Brånemark discovery in the 1950s, the term "osseointegration" has described the direct contact between a loaded implant surface and bone at the microscopic level of resolution.[7,12,13]

For decades, Ti and Ti alloys have been considerably interesting in the dental field, especially in dental implants, because of their excellent biocompatibility, physical, mechanical, and chemical properties. Ti has compatibility with hard and soft tissues, non-cytotoxicity, excellent corrosion resistance, high strength, and low density.[2,9,14,15] Commercially pure titanium (cp-Ti) is classified into 4 grades according to its impurity content (grade 1-4: 0.18-0.4% Oxygen). Moreover, various alloys (e.g., Iron, Aluminum and Vanadium) have been added to cp-Ti to improve its mechanical properties: preventing corrosion, increasing its strength, and decreasing its density. Titanium-6aluminum-4vanadium (Ti-6Al-4V) alloys are the most common form of Ti alloys for dental use.[15-17]

The surface characteristics of Ti implants are one of the key factors that affect the rate of osseointegration and biological response, resulting in the success of implant placement.[18] Initially, the surface of the Ti implant is smooth, created by macroscale machining techniques. Subsequently, the surface roughness at both micro- and nano-level is more effective for osseointegration than smooth Ti, due to the improvement of protein adhesion and tissue integration.[19-21] The modifications on the implant surface have been developed in several techniques including grit-blasting, acid-etching, electrochemical anodic oxidation, plasma spraying, fluoride treatment, laser treatment, calcium-phosphate coatings, or several combinations of these techniques (e.g., combined sand-blasted, large-grit, acid-etched surface).[7,16,18,22]

Anodization / Anodic oxidation

Surface modifications by an electrochemical method called "Anodization/anodic oxidation" is interesting as it is a simple, inexpensive, and effective technique for improving surface properties.[7-9,23] TiO_2 layer, which is very thin (a few nm in thickness) and defective, is naturally formed when the Ti surface meets oxygen. This oxide layer is the true biocompatible surface on Ti dental implants. To enhance the oxide layer for suitable cellular activity on the Ti surface, the anodization process helps thicken and stabilize this layer, transforming it into a highly oriented structure. Accordingly, when the biocompatibility of the surface was increased, osseointegration was promoted.[5-8] This process can also affect the mechanical properties of the Ti dental implant in several ways, including changes in its corrosion resistance, surface roughness and wettability.[7,12,24]

The anodization process (Figure 1) involves using a power supply of constant current/voltage to an electrochemical cell with an electrolyte bath containing electrolytes that provide conduction (acid or basic solution). The common electrolytes are acids (e.g., sulfuric acid (H_2SO_4) and phosphoric acid (H_3PO_4)), and salts (e.g., sodium sulfate (Na_2SO_4), and ammonium sulfate ($(NH_4)_2SO_4$).[9,12,19] The target substrate (Titanium) is on an anode, a positive electrical potential, while Platinum or Titanium is on the counter electrode (cathode). When the electricity runs, the oxygen is produced and combines with Ti on the Ti implant surface on the anode, forming the TiO_2 layer.[7,10,19,25]

The surface characteristics of anodized titanium

The surface characteristics of the anodized Ti surface are influenced by the anodizing conditions: the applied voltage, current density, anodization time, and types of electrolytes and concentration.[7,12] By increasing the voltage and current density, the thickness and resistance of the oxide layer are greater formed, resulting in increasing the surface porosity, thickness, roughness, wettability, and crystallinity. Likewise, extended anodization time brings a higher spark discharge intensity which induces the formation of a high surface area and high crystallinity anodic layer. For the type of electrolyte, the electrolyte could vary the pattern of microporous and crystallinity. Acidic electrolytes (sulfuric acid, acetic acid, phosphoric acid, and hydrochloric acid) and neutral electrolytes (sodium sulfate) form patterns of highly crystalline TiO_2 anodic layer while alkaline electrolyte (potassium hydroxide and sodium hydroxide) form patterns of nanoporous amorphous TiO_2 anodic layer. The electrolyte concentration also directly affects the surface porosity, thickness, roughness, and crystallinity.[7,12]

Cellular and tissue responses to the Ti surface have been widely investigated using fibroblasts, epithelial cells, and osteoblast-like cells.[2] Numerous *in vitro* cell culture experiments have shown that the anodized Ti surfaces expressed a positive effect on cell maturation and differentiation.[16] For hard tissue, Kim *et al.*[26] found that anodized Ti enhanced osteoblast adhesion and filopodia formation and has more mature bone formation than machined Ti, in addition to the bone implant contact (BIC) value being significantly greater. Furthermore, a study by Gulati *et al.*[27] on anodized 3D printed Ti implants showed that micro-scale and nano-topography from anodization provides an excellent cell adhesion substrate for human osteoblastic cells and promotes an osteogenic gene expression profile. These studies indicated that surface treatment by anodization increased osseointegration and bone formation. For soft tissue, Wang *et al.*[28] found that human gingival fibroblasts (HGFs) showed better adhesion strength, a more mature morphology, and greater proliferation and differentiation on rough Ti surfaces than on smooth surfaces. The filopodia of the HGFs on the surfaces of the anodized Ti were in contact with each other and formed a multidirectional net-

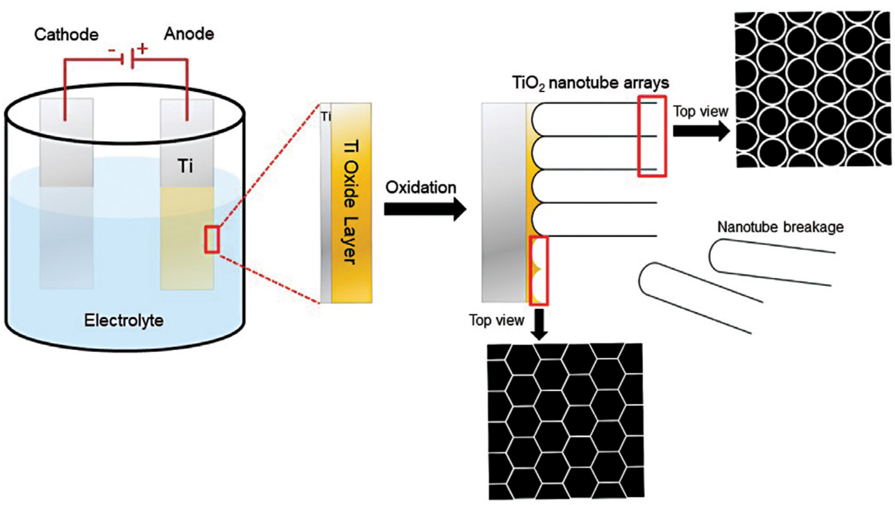

Figure 1: Schematic diagram showing the anodization process and the formation of TiO_2 nanotube arrays. The top view of the structures via scanning electron microscopy shows the circular forms of the tubules. The binding between the nanotube arrays and the Ti surface is generally weak, and breakdown is frequent at the interface. The morphology underneath the tubes is hexagonal. (Modified from Yeo *et al.*[7])

work while the HGFs on the untreated specimen aligned parallel to each other. On the other hand, a study by Fadeyev *et al.*[5] on the adhesion of fibroblasts on anodized Ti showed that the quantity and average area of fibroblasts adhered on nanotubular Ti was not significantly greater than on untreated Ti. However, the nanotubular surfaces from anodization did not cause excessive adhesion of fibroblasts that might lead to the risk of implant rejection from the formation of fibrous capsules.

The parameters that may promote implant-tissue interaction and osseointegration are surface roughness, surface composition, and wettability. There are three levels of surface roughness: macro-, micro-, and nano-scale topologies.[4] As nano-scale roughness and topography Ti surface are better at achieving desirable bonding with bone than the conventional smooth or micro-rough surfaces, the electrochemical anodization helps fabricate the ordered nanostructures with suitable physicochemical properties, especially titania nanotubes.[20,24,27] Yao *et al.*[29] varied the anodization conditions and found that the resulting anodized titanium surface on SEM and AFM can either be heterogeneous nanoparticles (inner diameter sized about 30-40 nm) or ordered nanotube-like structure (inner diameter sized about 70-80 nm). Villaça-Carvalho *et al.*[8] found that the anodized Ti showed changes in the chemical and structural composition of the TiO_2 film, favorable to osteogenic activity. From SEM analysis, a nanotextured surface was also observed with topography more uniform and valleys less depth (Figure 1). The aligned TiO_2 nanotube-layered surface has great potential in biological and clinical applications.[7] Moreover, it seems to be contradictory on the optimal diameter for osseointegration as there were four different stages according to Wu *et al.*[30], including protein absorption, inflammatory cell adhesion/inflammatory response, additional relevant cells adhesion, and angiogenesis/osteogenesis. They believed that TiO_2 nanotubes of about 30 nm diameter are more suitable for the function of relevant protein absorption, TiO_2 nanotubes of about 15 nm diameter are more suitable for inflammatory regulation, TiO_2 nanotubes of 15-30 nm diameter have a positive effect on repairment-related cell adhesion, and TiO_2 nanotubes of about 100 nm diameter are suitable for osteogenesis.[30] However, several studies confirmed that the optimal diameter of TiO_2 nanotubes for osteoblasts functions on titania is less than 100 nm.[24,29]

The wettability (hydrophilicity and hydrophobicity) or water contact angle, which is affected by surface composition, plays an important role in the cell response. Biological fluids, cells, and tissues prefer highly hydrophilic surfaces (lower water contact angle). Since the anodization process changed the amorphous TiO_2 nanotubes into a crystalline form, the surface became more hydrophilic, resulting in the proper environment for cell behavior.[4,24,28] Wu *et al.*[30] suggested that it is hard to conclude precisely the best degree of contact angle for protein absorption but the contact angle of TiO_2 nanotubes below 50 degrees shows better biological activity compared with materials with larger contact angle.

Although the mechanical properties change to promote implant-tissue interaction and osseointegration, the anodized Ti may appear in different colors due to the thickness and crystal structure of the TiO_2 layers.[12] The color formation helps improve the reflection of the underlying metal surface, which produced a gray metal color when white light is emitted on the unanodized Ti.[10]

Color formation on titanium surface by anodization

One of the disadvantages of using Ti as a dental implant abutment is the grayish appearance (the metallic gray color) that affects the peri-implant soft tissue, especially in high esthetic areas (anterior maxilla area) or when some unfavorable soft tissue conditions (thin peri-implant mucosa or recession) are present.[31] This grayish appearance is caused by the reflection of white light on the TiO_2 layer of the metal surface.

The TiO_2 layer, a clear and thin (approximately 5-20 nm in dimension) layer, is produced when Ti or Ti alloys make contact with the oxygen in the air.[10] However, the anodization helps increase the thickness of the oxide layer up to about 100 nm[16], with greater thickness when higher voltage is applied. The higher the voltage, the higher the anodic film thickness forms. The different thicknesses of TiO_2 cause variations in refractive index and reflective index which produces various colors of anodized Ti. The color-producing phenomenon on the Ti surface after anodizing is known as interference colors (light interference effects produce a color change) (Figure 2), which may be useful in prosthetic dentistry.[9,10]

The color formation of anodized Ti can be explained by the multi-beam interference theory (Figure 2). The reflected beams from the TiO_2 surface and the

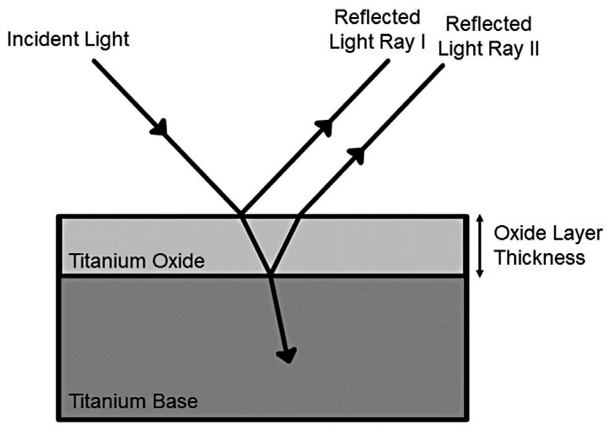

Figure 2: Multi-beam interference theory for color formation on anodized titanium. (Modified from Alipal *et al.*[12], Diamanti *et al.*[14])

surface between TiO_2 and Ti base can produce interference colors. The color will change depending on the increase in oxide thickness and the crystalline structures in the oxide layer. The constructive and destructive interference of certain wavelengths results in various colors.[12]

To obtain the desired color on the Ti surface, the feeding voltage (V) is applied during the anodizing process. This technique provides a straightforward means of coating Ti parts and a color scale. Karambakhsh *et al.*[32] anodized pure commercial Ti surface in sulfuric acid from 5 V to 80 V. The colors produced were gold/brown (5, 10 V), violet (15 V), dark blue (20 V), blue (25, 30 V), green/blue (35 V), light blue (40 V), pale blue (45 V), dark yellow (50 V), yellow (55, 60 V), pink/yellow (70 V), pink (75 V) and pink/purple (80 V) as shown in Table 1. Wadhwani *et al.*[33] altered the abutment color by anodization in a laboratory using trisodium phosphate as an electrolyte. The voltage of 60 V and 85 V were used to achieve yellow and pink colors, respectively. Napoli *et al.*[25] applied electrolytic voltage ranging from 15 V to 150 V on grade II Ti and grade V Ti in a citric acid electrolyte bath, which produced a wide gamma of colors, where brown/yellow (17 V), blue/light blue (30, 40, 60 V), gold (80, 100 V), and fuchsia/purple (150 V) were the colors achieved from the growing potential in this experiment. Wadhwani *et al.*[10] varied the voltage of anodization from 0 V to 85 V on titanium-6aluminum-4vanadium (Ti-6Al-4V) alloy using trisodium phosphate as an electrolyte, resulting in the variation of oxide film thickness and coloration. The resulting colors were gold (10 V), brown (15 V), purple (20 V), dark blue (25 V), light blues (30, 35, 40 V), pale blue (45 V), white/yellow (50, 55 V), yellow (60, 65 V), pink/yellow (70 V), pink (75, 80 V) and pink/purple (85 V). Similarly, Wang *et al.*[28] also produced a variety of colors on the surfaces of Ti alloys by anodization at 5 V to 90 V in phosphoric acid. The colors changed synchronously with the alteration of anodization voltages as gold/brown (5, 10 V), brown (15 V), violet (20 V), dark blue (25 V), blue (30 V), light blue (35 V), green/blue (40, 45 V), light green (50 V), green/yellow (55 V), yellow/orange (60 V), red/pink (65 V), pink (70 V), purple (75 V), purple/blue (80 V), blue (85 V) and green (90 V).

The hue, chroma, and value of color varied in the same pattern as shown in Table 1. This color and brightness changes through anodization only modified the surface of the Ti by altering the TiO_2 layer thickness, yet the surface chemistry, tensile, and other mechanical properties of the materials remained the same. The surface biocompatibility is still maintained as well. Additionally, anodization is a reversible process, where the oxide layer on the Ti surface can simply be removed and returned to its clear state if any error has occurred or the color is not as desired.[10,33]

The spectrophotometer and the CIELab color space (Standard colorimetric space) (Figure 3) play a role in measuring the color change from anodization, hue (type of color), chroma (saturation), and value (lightness). CIELab has a three-dimensional color space: L* (luminosity; black [0] – white [100]), a* (green [-] – red [+]), and b* (blue [-] – yellow [+]) value.[25,28,32] A change in applied voltage causes changes in oxide film thickness, resulting in the variations of the refractive and reflective index on the color of anodized Ti. The CIELab color can be used to compare the different color ranges and the mean color difference (ΔE) can also be calculated using the CIE parameter. From the study by Napoli *et al.*[25] in grade II Ti and grade V (Ti-6Al-4V) Ti, by using CIELab color space, there are no differences in color produced between pure Ti and Ti alloy as they both showed an excellent uniformity of color from a macroscopic point of view. This can be concluded that the anodization can vary color in the same direction even with the use of different grades of Ti.

Table 1: The color formation on titanium surface at different voltages applied in the anodization process from previous studies. (Modified from Wadhwani *et al.*[10], Napoli *et al.*[25], Wang *et al.*[28], Karambakhsh *et al.*[32], Wadhwani *et al.*[33])

Year	Writer	Article	Anodized condition	Voltage applied (V)																				
				0	5	10	15	20	25	30	35	40	45	50	55	60	65	70	75	80	85	90	100	150
2011	Karambakhsh *et al.*[32]	Pure commercial titanium color anodizing and corrosion resistance	Pure commercial titanium Electrolyte: Sulfuric acid Voltage: 5-80 V Time: 30 sec	X																	X	X	X	X
2016	Wadhwani *et al.*[10]	Laboratory technique for coloring titanium abutments to improve esthetics	Titanium-based abutment Electrolyte: Trisodium phosphate solution Voltage: 60 and 85 V Time: 5 sec	C	X	X	X	X	X	X	X	X	X	X	X		X	X	X	X		X	X	X
2018	Napoli *et al.*[25]	Coloring titanium alloy by anodic oxidation	Type II titanium Electrolyte: Citric acid Voltage: 15-150 V	X	X	X		X	X		X		X	X	X		X	X	X		X	X		
			Type V titanium Electrolyte: Citric acid Voltage: 15-150 V	X	X	X		X	X		X		X	X	X		X	X	X		X	X		
2018	Wadhwani *et al.*[10]	Colorizing titanium-6aluminum-4vanadium alloy using electrochemical anodization: Developing a color chart	Titanium alloy (Ti6Al4V) Electrolyte: Trisodium phosphate solution Voltage: 0-85 V Time: 15 sec	C	X																	X	X	X
2018	Wang *et al.*[28]	Changes in the esthetics, physical, and biological properties of a titanium alloy abutment treated by anodic oxidation	Titanium alloy (Ti6Al4V) Electrolyte: Phosphoric acid Voltage: 0-90 V Time: 60 sec	X																			X	X

C = Control, (17) = Voltage applied 17 V

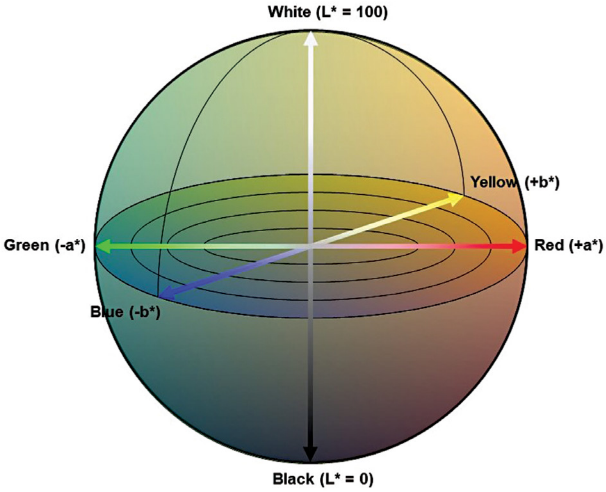

Figure 3: 3D CIELab color space. (Modified from Napoli *et al.*[25], Karambakhsh *et al.*[32])

Clinical application of anodized colored titanium

The preservation or reproduction of a natural mucogingival architecture surrounding dental implants is challenging from an esthetic viewpoint.[34] When the anodized titanium abutment was placed in the high esthetic area, it seems to have a more beneficial outcome over an unanodized Ti abutment on the color change of peri-implant soft tissue surrounding a Ti abutment.[26,31] (Figure 4) This could also reduce the risk of advanced surgery such as tissue augmentation and grafting in compromised cases.[31]

The most useful colors for dental application are gold (10 V), yellow (60 to 65 V), and pink (70 to 80 V) in

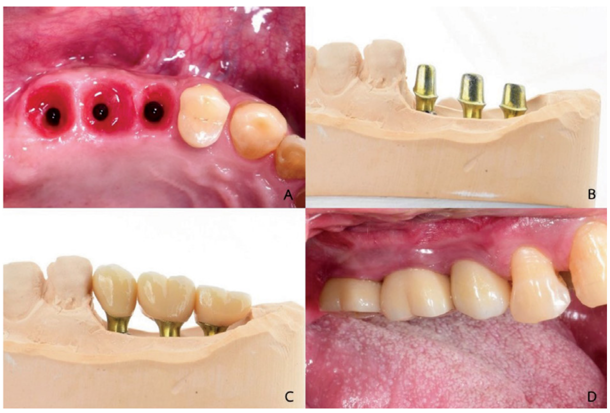

Figure 4: Displays a gold-anodized titanium implant placement. A–Soft tissue before gold-anodized titanium abutment in place, B–Laboratory gold-anodized titanium abutment, C–Laboratory gold-anodized titanium abutment with crown, D–Gold-anodized titanium implant in place. (Photo by Tachakorn Kuntiyaratana)

hue.[10,35] Yellow and gold hues have mostly been used in restoration or abutment areas while pink hue has been used for gingival areas.[33,36] Similarly, several studies on the effects of implant abutment material on peri-implant soft tissue color have concluded that the peri-implant soft tissue color appears to be different from the soft tissue color around natural teeth.[34] Ti abutment has a significantly high color difference[34] wherein gold-anodized and pink-anodized Ti abutments have achieved better esthetics for peri-implant soft tissue than the unanodized Ti abutment.[36-39] Furthermore, zirconia abutments have resulted in the least color difference.[34,37-39]

Besides analyzing the color differences, the pink esthetic score (PES), clinician and patient satisfaction questionnaires were measured where no significant difference was found in patient or clinician perception/satisfaction between gold/pink-anodized abutment, and zirconia abutment materials.[37,39] Patients were significantly more satisfied than clinicians with gingival esthetics surrounding the implant restoration.[37] However, pink-anodized abutments represent a good esthetic alternative to zirconia hybrid abutments, especially in mechanically challenging situations.[39] Although there was no significant difference in the soft tissue response between the zirconia and Ti abutments, the mechanical properties of zirconia are inferior to that of Ti.[15]

The selection of a dental implant system that allows a proper biological response of the hard and soft tissues, represents the first step for the achievement of adequate esthetic results. A proper surgical technique, implant positioning, and soft tissue management, along with the proper prosthetic solution are also necessary for a natural outcome.[34] Anodized Ti has achieved a good esthetic outcome. However, the color formation of the TiO_2 layer is not the only factor in successful implant placement in high esthetic areas. Other surface characteristics and properties of the Ti should also be considered simultaneously.

Conclusions

Anodization is a productive electrochemical method to modify the oxide layer on the Ti surface. Despite the micro- and nano-level roughness of the surface, the interference color from the oxide formation is an advantage that helps overcome the grayish appearance of Ti resulting in a good esthetic outcome. It is a voltage-dependent process that can produce and reproduce a desired color. Pink- and gold-anodized Ti represent a good esthetic alternative to other abutments, especially in mechanically challenging situations. This paper reviewed the surface characteristics of anodized Ti and the color formation on Ti surfaces by anodization. Besides esthetic concern, the most important consideration for dental implant success is a good adaptation between the implant surface and the surrounding tissue, therefore the relationship between each color produced and surface characteristics requires further study.

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Conflicts of Interest

The authors declare no conflicts of interest.

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