Dental Research Journal

Original Article

In vitro effect of anodization of titanium abutments on their tensile bond strength to implant-supported lithium disilicate all-ceramic crowns

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ABSTRACT

Background: The retention of cement-retained implant-supported restorations can be affected by surface treatments such as anodizing. This study aimed to assess the effect of the anodization of titanium abutments on their tensile bond strength to implant-supported lithium disilicate (LDS) all-ceramic crowns.

Materials and Methods: This *in vitro*, experimental study was conducted on 26 straight abutments in two groups of anodization and control. In the anodization group, seven flat 9V batteries connected in series were used to generate 64 V energy. A glass container was filled with 250 mL of distilled water, and 1 g of trisodium phosphate was added to it to create an electrolyte solution. The anode was then disconnected and the abutment was rinsed with acetone and deionized water. The surface roughness of abutments was measured by a profilometer. The abutments were scanned by a laboratory scanner, and maxillary central incisor monolithic crowns were fabricated by inLab SW18 software. The crowns were seated on the abutments and temporarily cemented with TempBond. They were then incubated in artificial saliva and subjected to 5000 thermal cycles. The tensile bond strength of crowns was then measured. Data were analyzed by the Student's *t*-test and Mann–Whitney *U*-tests ($\alpha = 0.05$).

Results: The mean bond strength was significantly higher in anodized abutments ($P = 0.003$). The surface roughness of anodized abutments was slightly, but not significantly, higher than that of the control group ($P > 0.05$). The frequency of adhesive failure was almost twice higher in anodized abutments.

Conclusion: Anodization of titanium abutments significantly improved their tensile bond strength to implant-supported LDS all-ceramic crowns.

Key Words: Dental abutments, dental implants, single-tooth, surface properties, titanium oxide

Received: 24-May-2023
Revised: 09-Jul-2023
Accepted: 26-Jul-2023
Published: 27-Sep-2023

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INTRODUCTION

Retention loss of implant prosthesis and debonding of crowns are among the common problems of implant-supported restorations, especially in cement-retained implant abutments and short abutments.[1] Loss of cement integrity, cement dissolution, and cement debonding are the most common causes of reduced bond strength of cement

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www.ncbi.nlm.nih.gov/pmc/journals/1480

How to cite this article: Karimi Pour H, Ansari H, Tehrani AM. *In vitro* effect of anodization of titanium abutments on their tensile bond strength to implant-supported lithium disilicate all-ceramic crowns. Dent Res J 2023;20:99.

in cement-retained restorations.[2] Cement loss in implant-supported cement-retained restorations causes problems such as restoration loss, peri-implant alveolar bone resorption, prosthesis fracture, and trauma to the opposing teeth.[3] The geometrical shape of the abutment, abutment height and diameter, abutment surface area, surface roughness, and surface treatment are among the factors affecting the bond strength and retention of implant-supported cement-retained restorations.[4-9]

The currently used surface treatments include sandblasting with alumina particles, acid etching, silicoating, and anodization. Anodization is one suggested surface treatment for the optimization of titanium surface. It is an electrochemical process that morphologically changes the natural oxide layer and enhances its durability and corrosion resistance. It also increases its wear resistance and enables better adhesion to metals.[10,11] The oxide layer can be created with different thicknesses on the titanium surface using different voltages. Metal surfaces are coated with a thin layer of oxide in contact with air, which is highly porous, has low resistance and mechanical strength, and cannot protect the metal against corrosion.[12,13] Anodic oxidation is a suggested surface treatment that creates a homogenous coating on the surface. This coating can be seen in different colors due to light interferences with the titanium dioxide layer.[14] This process does not change the chemical structure of titanium and only increases the thickness of the oxide layer. The generated color is also stable and will have long durability if covered with a crown or soft tissue.

One major advantage of anodization is the resultant enhancement of surface roughness. Thus, it not only improves esthetics but also enhances cement retention.[15,16] Anodization of titanium and its alloys creates tiny porosities in the first step of anodization, and larger pores in the next step, and subsequently increases the bond strength to polymers.[17] However, many issues regarding anodization remain to be elucidated. For instance, the effects of anodization temperature, duration, and voltage and microstructure and mechanical properties of anodized oxide layer have not been precisely and systematically investigated. These variables play a fundamental role in the clinical application of anodization.[18]

The majority of studies on the bond strength of implant-supported restorations have assessed the effects of different types of surface treatments except for anodization,[12,18,19] and anodization has not been comprehensively addressed in the literature. Accordingly, an information gap exists regarding the effects of anodization particularly on the bond strength of implant-supported all-ceramic restorations. Thus, this study aimed to assess the effect of the anodization of titanium abutments on their tensile bond strength to implant-supported lithium disilicate (LDS) all-ceramic crowns.

MATERIALS AND METHODS

This *in vitro*, experimental study was conducted on 26 straight abutments.

Sample size

The minimum sample size for the assessment of tensile bond strength was calculated to be 12 in each group according to a previous study,[10] assuming $\alpha = 0.05$, $\beta = 0.2$, and bond strength standard deviation = 1.8 MPa to find a significant difference in bond strength equal to 2 MPa using the two-sample *t*-test power analysis of software PASS 11.0.

The minimum sample size for the assessment of surface roughness was calculated to be 7 in each group according to a previous study,[12] assuming $\alpha = 0.05$, $\beta = 0.2$, and surface roughness mean standard deviation = 12 (Ra) to find a significant difference in surface roughness equal to 20 units using the two-sample *t*-test power analysis of PASS 11.0.

Sample preparation

In this study, 10 mm × 4 mm dental implants (Tixos, Leader Italia, Italy) and 26 straight abutments with 4 mm diameter, 6 mm height, and 2 mm gingival height were used.[20] Dental implants were first mounted in acrylic molds fabricated from putty silicon impression material (Speedex, Coltene, Switzerland) with a circular cross-section of 72 mm in diameter and 22 mm in height.[21] The molds were filled with transparent auto-polymerizing acrylic resin (Meliodent, Heraeus Kulzer GmbH, Germany). To prepare the acrylic resin, proper amounts of powder and liquid were mixed as instructed by the manufacturer. A surveyor (JM Ney Co, Bloomfield, CT, USA) was used for the vertical (90°) mounting of dental implants in acrylic molds.[21] Accordingly, after the complete setting of acrylic resin for 24 h, all mounted dental implants were ready for the experiment.[12,18,19,22]

The abutments were cleaned with water vapor for 4 min[21,23] and were then randomly assigned to two groups (*n* = 13) of anodization and control. Thirteen abutments were then subjected to anodization. For this purpose, seven flat 9 V batteries were connected in series to generate 64 V energy.[12,22,24] A glass container was filled with 250 mL of distilled water and 1 g of trisodium phosphate was added to it and allowed to dissolve. An electrolyte solution was prepared as such. The negative electrode (cathode) was connected to a piece of aluminum foil measuring 3 cm × 6 cm and immersed in an electrolyte. The positive electrode (anode) of the red wire was connected to a titanium abutment [Figure 1]. Care was taken to ensure the anode holder was not immersed in the solution and the anode was not in contact with the solution. The current had to enter the solution through the abutment.[24] The voltage was established, and the process was accomplished within 5 s. The process of anodization is self-limited and stops spontaneously. At 60 V voltage, the color turned yellow gold, and then the anode was disconnected and the abutment was inspected under daylight and rinsed with acetone and deionized water.[18,22,24] Seven of the anodized abutments and 7 of the nonanodized abutments were randomly selected for the assessment of surface roughness by a profilometer (Time 3200/3202, Beijing Time Height Technology, China). The surface roughness of each specimen was measured in triplicate, and the mean of the three measurements was calculated and reported as the surface roughness (Ra) of the respective specimen in micrometers. Furthermore, one anodized abutment and one nonanodized abutment underwent surface topography assessment by a scanning electron microscope (SEM; FEI Quanta 200, *10-100000*, FEI, USA). These specimens were then separated from the rest of the specimens. Finally, 12 anodized and 12 nonanodized abutments were placed on the respective dental implants, and the abutment screw was torqued to 25 N/cm by an analog torque meter (Tixsos) as instructed by the manufacturer.[25] For the fabrication of all-ceramic crowns, the abutments were scanned by a laboratory scanner (Up300e; Up3d, China) using powder. Then, inLab SW18 software was used to design full-contour monolithic maxillary central incisor crowns with 1 mm thickness and a 3-mm hole in their incisal surface to ensure correct application of vertical tensile load by a universal testing machine (Z050; Zwick/Roell, Germany). Next, a resin pattern was printed by a computer-aided design/computer-aided manufactured milling machine (CEREC MCXL, Sirona).[23] The printed patterns were then spread and placed in an investment ring, and the flask was filled with investment gypsum (IPS PressVest). After a minimum of 60 min, the specimens were placed at room temperature to allow the complete setting of gypsum. Next, the ring was placed in a wax burnout furnace upside down, heated to 850°C, and kept at this temperature for 60 min. After completion of the preheating phase in the heat-press furnace, the flask was placed in the furnace within 30 s to prevent cooling. Next, a cold ceramic IPS e.max Press ingot (Ivoclar Vivadent, Lichtenstein) was placed over the investment (facing up) and subjected to load by a cold IPS ALOX plunger; the heat pressing process of ceramic was accomplished as such. After cooling at room temperature and removing the ceramic from the investment, the sprue was cut by a disc. Next, the crowns were cleaned in an ultrasonic bath (DTE D3, Woodpecker, China) containing 96% isopropyl alcohol for 5 min. The crowns were seated manually with hand pressure and cemented with TempBond (Kerr, USA) temporary cement according to the manufacturer's instructions. The specimens were incubated in artificial saliva (Kin Hidrat, KIN, Spain) at 37°C for 7 days.[21] Next, they underwent 5000 thermal cycles at 5°C–55°C with a dwell time of 30 s and transfer time of 10 s, corresponding to 6 months of clinical service.[18]

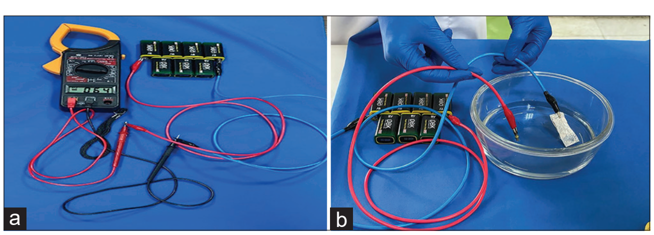

Figure 1: (a) Flat battery used for anodization. (b) Anodization process.

Cyclic loading

Considering the use of temporary cement, the application of thermal cycles, and the possibility of debonding during cyclic loading, 2 specimens underwent cyclic loading as a pilot. They were cemented and subjected to cyclic loading by 10,000 cycles corresponding to 1 year of clinical service.[23] Both crowns were debonded from the abutments after cyclic loading. The previous steps were repeated for these two specimens, and the conduction of cyclic loading was canceled.[23]

Bond strength measurement

A tensile load (50 kg) was applied at a crosshead speed of 0.5 cm/min to each crown in a universal testing machine (Z050; Zwick/Roell, Germany). The load at cement failure (debonding) was recorded in Newtons (N), and the mode of failure was also recorded.[23]

Statistical analysis

Data were analyzed by SPSS, version 25 (IBM company, usa). The Kolmogorov–Smirnov test showed normal distribution of bond strength data. Thus, the parametric Student's *t*-test was used to compare the two groups regarding the bond strength. The Kolmogorov–Smirnov test showed nonnormal distribution of surface roughness data. Thus, the nonparametric Mann–Whitney *U*-test was applied to compare the surface roughness of the two groups. The level of significance was set at 0.05.

RESULTS

Table 1 presents the mean values of bond strength and surface roughness in the two groups. The Student's *t*-test showed that the mean bond strength was significantly higher in the anodized abutment group ($P = 0.003$). The Mann–Whitney *U*-test showed that the mean surface roughness was slightly, but not significantly, higher in the anodized abutment group ($P = 0.11$).

Table 2 presents the frequency of different modes of failure in the two groups. In total, 91.7% of fractures in the nonanodized abutment group and 83.3% of fractures in the anodized abutment group were mixed.

Table 1: Mean values of bond strength and surface roughness in the two groups

Variable	Group	Mean	SD	SEM	P
Bond strength (N)	Nonanodized abutments	19.75	8.66	2.50	0.003
	Anodized abutments	42.63	20.46	5.91	
Surface roughness (μm)	Nonanodized abutments	0.11	0.10	0.04	0.11
	Anodized abutments	0.23	0.14	0.05	

SEM: Standard error of mean, SD: Standard deviation

Table 2: Frequency of different modes of failure in the two groups

Mode of failure Group	Adhesive (%)	Mixed (%)	Total (%)
Nonanodized abutments	1 (8.3)	11 (91.7)	12 (100.0)
Anodized abutments	2 (16.7)	10 (83.3)	12 (100.0)
Total	3 (12.5)	21 (87.5)	24 (100.0)

Figures 2 and 3 present the SEM micrographs of anodized and nonanodized abutment groups, respectively.

DISCUSSION

Anodization is a suggested surface treatment for titanium implant surface which alters the morphology and increases the thickness of the natural oxide layer and enhances its durability and corrosion resistance.[10] This study assessed the effect of the anodization of titanium abutments on their tensile bond strength to implant-supported LDS all-ceramic crowns. The results showed that the anodization of titanium abutments significantly increased their bond strength to LDS crowns compared with the nonanodized group.

Titanium has high chemical reactivity, and a reaction occurs between titanium and gases such as nitrogen, oxygen, and hydrogen at high temperatures. The formation of a thick oxide layer on the titanium surface probably decreases its resistance and toughness and interferes with its bonding capacity.[26] Several studies have assessed the efficacy of different surface treatments for titanium surfaces,[15,27,28] such as thermal oxidation, chemical oxidation, titanium nitride coating, and anodization. However, thermal oxidation complicates the achievement of a homogenous and reproducible color,[29] and chemical oxidation

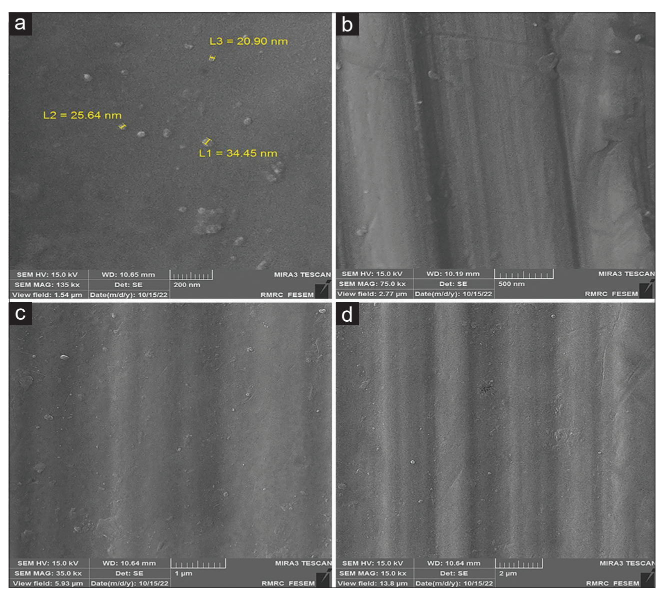

Figure 2: Scanning electron microscope micrographs of anodized abutment group. (a) Magnification 135 kx. (b) Magnification 75 kx. (c) Magnification 35 kx. (d) Magnification 15 kx. SEM: Scanning electron microscope.

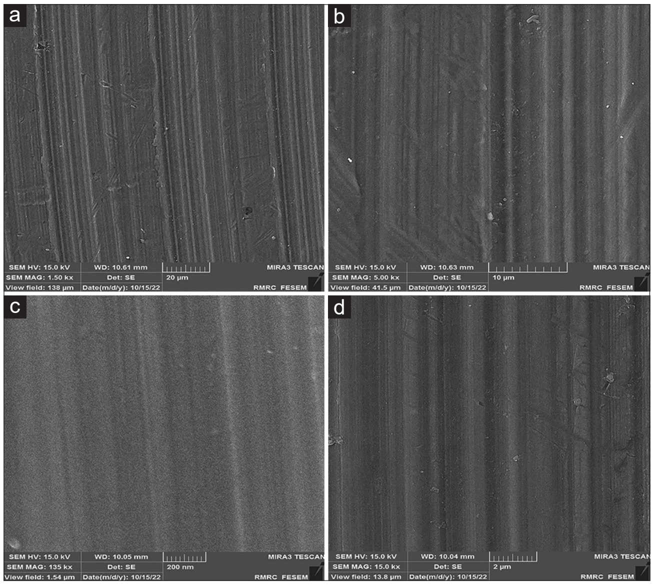

Figure 3: Scanning electron microscope micrographs of nonanodized control group. (a) Magnification 1.50 kx. (b) Magnification 5 kx. (c) Magnification 135 kx. (d) Magnification 15 kx . SEM: Scanning electron microscope.

results in inadequate and short-term corrosion and the formation of a chemical substrate.[30] Titanium nitride coating changes the metallic color to gold and improves the esthetics. However, this coating can cause allergic reactions.[31] Thus, the above-mentioned methods cannot be reliably used in the clinical setting. Anodization is an electrolyte reaction that increases the thickness of the titanium oxide layer. The thickness of anodized oxide layer may vary depending on the voltage and duration of anodization,[32] electrolyte temperature, and type of electrolyte solution.[33] In addition, different colors may be emitted from the surface depending on the light interferences with the superficial oxide layer.[34] Titanium oxide is the main compound on the titanium surface which has a gold color at 60 V voltage. Titanium anodized with gold has shown satisfactory clinical results.[35] Thus, anodic oxidation may serve as a simple, low-cost, and ecologically friendly method with predictable results.[12,36] The oxide film on the titanium surface has 2–7 nm thickness.[12]

Amornwichitwech and Palanuwech[37] measured the shear bond strength of titanium to LDS glass ceramics following different surface treatments and showed that different surface treatments, particularly anodization, enhanced the bond strength of titanium to resin cement-bonded LDS. Similarly, Rudawska *et al*.[22] demonstrated that the anodizing of titanium surfaces enhanced the bond strength of titanium alloy. Moreover, He *et al*.[18] evaluated the microscopic structure of anodized titanium alloy and measured its shear bond strength to epoxy resin and reported that anodizing at 0°C and 25°C temperatures enhanced the shear bond strength of specimens by 217% and 225%, respectively (compared with the control group). The results of the abovementioned studies were in agreement with the findings of the present research.

In contrast to the results of the present study, Acar *et al*.[38] showed that the anodization of titanium casts with sodium hydroxide had no significant effect on the titanium-porcelain bond strength. Differences between their results and those of the present research may be due to different anodizing parameters adopted in the two studies. The type of electrolyte and anodizing parameters can significantly affect the surface morphology, topography, and biocompatibility of the anodic layer.[39] Voltage is an influential parameter in this respect. Increasing the voltage increases the surface porosities, thickness, roughness, wettability, and crystallinity of the titanium surface, which may be due to electrochemical reactions in high voltages and increased thickness and resistance of the oxide layer.[39] Thus, a high potential is required for the fracture of the dielectric layer and the formation of a porous surface, which increases the surface roughness and enhancing the wettability of the oxide layer.[40] On the other hand, by increasing the voltage to over 60 V, as in the present study, the diameter of the pores decreased, which would subsequently affect the titanium surface morphology.[41]

The duration of anodization is another important factor, which was 5 s in the present study. Increasing the duration of anodization results in the formation of areas with anodic layers with high crystallinity.[39] It has been reported that increasing the duration of anodization increases the diameter of pores, surface roughness, and hydrophilic property of the titanium oxide anodic layer.[42,43] Electrolyte concentration, temperature, titanium alloy composition, and simultaneous application of other titanium surface treatments are among other influential factors in this respect.[39]

In the present study, mixed failure was the dominant mode of failure in both groups. However, the frequency of adhesive failure in the anodized group was twice that of the nonanodized group. Similarly, Amornwichitwech and Palanuwech[37] showed a higher frequency of adhesive failure in the anodized group, and He *et al*.[18] reported that mixed failure was the dominant mode of failure in the anodized group.

Profilometric assessments in the present study revealed higher surface roughness in the anodized group, although the difference did not reach statistical significance. This finding was also confirmed by SEM assessment. In contrast to the findings of the present study, Amornwichitwech and Palanuwech[37] revealed that the anodized titanium surface was smoother and had no porosity. The anodized film thickness was reported to be 180 nm.[44]

It has been reported that the anodization of titanium implants can significantly increase their surface roughness height but its effect on the highest peaks is limited since it creates surfaces with higher valleys rather than peaks.[45] Such changes appear to enhance the bond strength of implant-supported LDS crowns as the bond strength of the anodized group was significantly higher than that of the nonanodized abutments in the present study. Consistent with the present study, Wang *et al*.[12] indicated that anodization increased the titanium surface roughness. He *et al*.[18] reported an increase in the surface roughness of titanium after anodization at 0°C and 25°C, but this increase was limited at temperatures over 40°C. Increased surface roughness of abutments also increases biofilm formation and has adverse effects on clinical periodontal parameters.[46] Some other studies have reported a rougher and harder titanium oxide layer in anodized titanium implants,[47,48] which is in line with the results of the present study. Such rougher surfaces increase the durability of restorations.

The shear bond strength of sound teeth is approximately 13.4 MPa.[49] The values obtained in the present study for LDS crowns exceeded the value in natural teeth in both groups. Thus, both methods would yield a bond strength higher than that of natural teeth, indicating a low risk of displacement in the clinical setting.

This study had an *in vitro* design. Although the authors did their best to simulate the clinical setting by thermocycling, the oral environment cannot be perfectly simulated *in vitro*. Thus, generalization of the results to the clinical setting should be done with caution. In addition, future studies are recommended to simulate pH and thermal alterations and perform long-term water storage and dynamic fatigue loading to better mimic the clinical setting and obtain more accurate results regarding the effect of the anodization of titanium abutments on their bond strength. Furthermore, this study only assessed LDS ceramics. Future studies are required to evaluate other ceramic types. Moreover, the effects of anodization should be compared with those of thermal and chemical oxidation and other types of coatings.

CONCLUSION

Within the limitations of the present *in vitro* study, the results showed that the anodization of titanium abutments significantly improved their tensile bond strength to implant-supported LDS all-ceramic crowns. However, it did not significantly increase the surface roughness.

Financial support and sponsorship

Nil.

Conflicts of interest

The authors of this manuscript declare that they have no conflicts of interest, real or perceived, financial or non-financial in this article.

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