

# The Effect of Different Production Methods and Anodisation Parameters on Resin Cement-Titanium Bonding: An in Vitro Study

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## ABSTRACT

**Objective:** To evaluate the bonding strength between titanium produced by different techniques and resin cement. In addition, the study examines the effects of alumina blasting and anodisation voltage levels on the titanium-resin cement bond.

**Materials and Methods:** The control group comprised titanium samples produced by milling, while the experimental group comprised titanium samples produced by selective laser melting. The experimental group was subdivided into subgroups: sandblasting only with alumina; anodization at 36 V after alumina sandblasting; and anodization at 50 V after alumina sandblasting. The alumina blasting process was conducted at 1.5 atm for 10 seconds. Subsequently, anodisation was performed in 1 M Phosphoric Acid. Subsequent to thermal cycling, the bond strength was analysed using t-tests and Kruskal-Wallis tests at a significance level of  $p < 0.05$ .

**Results:** The bond strength of the titanium group subjected to selective laser melting and only to alumina sandblasting was statistically significantly higher than that of the control group ( $p = 0.040$ ). A statistically significant discrepancy was identified among the experimental group subgroups ( $p = 0.039$ ). The bonding strength of the group subjected to sandblasting alone was significantly higher than that of the group anodised at 36V after sandblasting ( $p = 0.033$ ).

**Conclusion:** Titanium produced by the selective laser melting method demonstrated higher bond strength with resin cement compared to the milling method. Among the surface treatments evaluated, alumina blasting alone provided the highest bond strength, whereas anodisation performed after sandblasting reduced bond strength. Anodisation applications at different voltages yielded similar results, both exhibiting clinically acceptable bond strengths.

## Farklı Üretim Yöntemleri ve Anodizasyon Parametrelerinin Rezin Siman-Titanyum Bağlantısına Etkisi: Bir in Vitro Çalışma

### Makale Bilgisi

#### Makale Geçmişi

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Kayma Dayanımı.

### ÖZET

**Amaç:** Farklı tekniklerle üretilen titanyum ile rezin siman arasındaki bağlanma dayanımını değerlendirmektir. Ayrıca çalışma, alümina kumlama ve anodizasyon voltaj seviyelerinin titanyum-rezin siman bağı üzerindeki etkilerini incelemektedir.

**Gereç ve Yöntemler:** Kontrol grubu frezeleme yöntemiyle üretilen titanyum örneklerinden, deney grubu ise selektif lazer eritme yöntemiyle üretilen titanyum örneklerinden oluşturulmuştur. Deney grubu şu alt gruplara ayrılmıştır: sadece alümina ile kumlama; alümina kumlamadan sonra 36 V'ta anodizasyon; ve alümina kumlamadan sonra 50 V'ta anodizasyon. Alümina kumlama işlemi 1,5 atm basınçta 10 saniye süreyle uygulanmıştır. Takiben, 1 M Fosforik Asit içerisinde anodizasyon işlemi gerçekleştirilmiştir. Termal siklus (ısısal döngü) sonrası bağlanma dayanımı,  $p < 0.05$  anlamlılık düzeyinde t-testi ve Kruskal-Wallis testleri kullanılarak analiz edilmiştir.

**Bulgular:** Selektif lazer eritme uygulanan ve sadece alümina ile kumlanan titanyum grubunun bağlanma dayanımı, kontrol grubundan istatistiksel olarak anlamlı derecede yüksek bulunmuştur ( $p = 0.040$ ). Deney grubunun alt grupları arasında istatistiksel olarak anlamlı bir fark saptanmıştır ( $p = 0.039$ ). Sadece kumlama uygulanan grubun bağlanma dayanımı, kumlama sonrası 36 V'ta anotlanan gruba göre anlamlı derecede yüksek çıkmıştır ( $p = 0.033$ ).

**Sonuç:** Selektif lazer eritme yöntemiyle üretilen titanyum, frezeleme yöntemine kıyasla rezin siman ile daha yüksek bağlanma dayanımı göstermiştir. Değerlendirilen yüzey işlemleri arasında, yalnızca alümina kumlama en yüksek bağlanma dayanımını sağlarken; kumlama sonrası yapılan anodizasyon bağlanma dayanımını düşürmüştür. Farklı voltajlardaki anodizasyon uygulamaları benzer sonuçlar vermiş olup, her iki uygulama da klinik olarak kabul edilebilir bağlanma dayanımı sergilemiştir.

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## INTRODUCTION

The achievement of optimal aesthetics in implant-supported anterior restorations is contingent on the appearance of the soft tissue in individuals exhibiting a high smile line and a thin gingival biotype. Despite the fact that titanium is extensively utilised in dental implants and abutments due to its exceptional biocompatibility, in certain instances, its greyish hue is reflected in the gingival area, leading to an aesthetically displeasing outcome, particularly in anterior restorations.<sup>1</sup> In order to overcome this problem, it may be possible to increase the thickness or opacity of the restorative material, or alternatively to use abutments made of materials other than titanium. For instance, the fabrication of abutments from gold, titanium nitride, alumina, zirconia, or composite resin-coated titanium has been utilised with a view to improving aesthetics. Another method for improving the aesthetic appearance of dentures is to alter the colour of the abutments by means of anodising. Anodising is an electrochemical process that thickens and changes the appearance of the natural oxide layer on the titanium surface without altering the chemical composition of the titanium itself. The formation of this layer is achieved through the exposure of titanium to an electrolyte solution at varying voltages (ranging from 5 to 90 V). The resultant layer exhibits a spectrum of colours that demonstrate stability over an extended period.<sup>2-4</sup> The utilisation of anodised yellow or pink abutments confers aesthetic advantages over non-anodised titanium abutments, which exhibit a greyish appearance in the anterior region.<sup>5,6</sup>

In addition to achieving an aesthetically acceptable outcome, adequate retention between the abutment and the restoration is an essential factor in the success of implant-supported restorations. The creation of micro retentive grooves and ridges is achieved through a variety of techniques, including but not limited to acid etching, electroerosion, and milling. The employment of these techniques, either individually or in

combination, is undertaken to achieve specific objectives such as enhancing bonding, increasing the surface area of the material, and consequently improving the retention of cemented restorations. In addition to the aforementioned methods, sandblasting and anodising techniques have been demonstrated to create rough, thick, porous oxide films on the titanium surface, thereby producing high-strength adhesive bonds.<sup>7,8</sup>

In the prosthetic treatment field, Computer-Aided Design-Computer-Aided Manufacturing (CAD-CAM) technologies have provided alternative methods for titanium fabrication, utilising both subtractive and additive manufacturing techniques. Subtractive manufacturing is performed by milling solid blocks, whereas additive manufacturing is performed by melt deposition, stereolithography, selective laser sintering and selective laser melting (SLM). SLM is an additive manufacturing technology that relies on the laser melting of multiple layers of powdered material in order to form a three-dimensional structure.<sup>9,10-12</sup>

In the extant literature, a plethora of studies have been conducted investigating the bonding between ceramic materials and titanium produced using various manufacturing techniques. The focus of these studies has been on shear bond strength, bonding performance after thermo-mechanical ageing, and the characteristics of the oxide layer formed during porcelain firing.<sup>13-15</sup> Furthermore, there are studies that evaluate the resin cement–titanium bond after different surface treatments of titanium. Nevertheless, the number of these studies remains limited compared with those focusing on ceramic–titanium bonding.<sup>16,17</sup> The objective of the current study is to compare the bond strength of titanium produced by different methods and subjected to various surface treatments with resin cement. The null hypotheses ( $H_0$ ) of this study are as follows: (1) there is no

discrepancy in the bond strength with resin cement between titanium specimens manufactured by additive and subtractive methods, and (2) there is no variation in the bond strength with resin cement among titanium specimens produced by additive manufacturing and anodised at varying voltage levels post-sandblasting.

## **MATERIALS AND METHODS**

The present study does not require ethics committee approval as it does not involve the use of human or animal material.

### **Material**

The study formed two primary groups of materials: the subtractive manufacturing titanium group (GM, control) and the additive manufacturing titanium group (GSLM, experimental). The GSLM group was further divided into three subgroups: The experimental design comprised three distinct groups: GSLM-1, which involved  $Al_2O_3$  blasting only; GSLM-2, which involved  $Al_2O_3$  blasting followed by 36V anodising; and GSLM-3, which involved  $Al_2O_3$  blasting followed by 55V anodising.

### **Preparation of the control group (GM)**

First of all, disk-shaped specimens with a diameter of 8 mm and a height of 3 mm were designed and transferred to a Standard Tessellation Language (STL) file. Next, thirteen specimens were produced from titanium blocks (KERA Ti5-DISC, Eisenbacher Dentalwaren ED GmbH, Eisenbach, Germany) using a milling machine (Dentium Rainbow Mill-Metal, Dentium Co., Seoul, South Korea). Finally, the surface of the prepared specimens was roughened with 110 microns of  $Al_2O_3$  particles at a distance of 10 mm, at a pressure of 1.52 bar for 10 seconds at an angle of 45 degrees, and then, cleaned in an ultrasonic bath.

### **Preparation of the GSLM1 group**

Initially, thirteen samples were fabricated utilising Ti6Al4V powder (ERMAK A252, Ermaksan Makina Sanayi ve Ticaret A.Ş., Bursa) with a laser power of 190 W at a working distance of 30 microns in the SLM machine (ENAVISION 250, Ermaksan Additive Manufacturing Technologies, Bursa, Turkey) by designing disk-shaped samples with a diameter of 8 mm and a height of 3 mm, and then transferring them to the STL file. The surface of the prepared samples was roughened with 110 microns of  $Al_2O_3$  sand at a distance of 10 mm, at a pressure of 1.52 bar for 10 seconds at an angle of 45 degrees. The samples were then cleaned in an ultrasonic bath (BK-3550 Ultrasonic Cleaner, BAKU Electronics Co., Ltd., Shenzhen, China) (Figure 1a).

### **Preparation of GSLM2 and GSLM3 Groups**

In accordance with the established protocol for the preparation of samples belonging to the GSLM1 group, the samples were subjected to anodisation in a temperature-controlled bath, utilising 1M  $H_3PO_4$  as the electrolyte for a duration of 30 seconds. The samples were subjected to anodisation at 36 V to obtain a blue colour, thus, forming the GSLM2 group ( $n = 13$ ) (Figure 1b), and at 50 V to obtain a yellow colour, thereby forming the GSLM3 group ( $n = 13$ ) (Figure 1c). Subsequently, the samples were subjected to a cleaning process involving deionised water at 100 °C for a duration of 30 minutes. Subsequently, the samples were sealed and dried in a cold air stream.

### **Scanning Electron Microscope (SEM) Analysis**

A scanning electron microscope (SEM) (LEO 1430 VP Scanning Electron Microscope, Carl Zeiss SMT Ltd., Oberkochen, Germany) analysis was conducted to examine a selected sample from each group in detail (See Figure 2).

### Preparation of Composite Samples

The composite samples were prepared and then affixed to the surface of the titanium alloy using a resin cement. This process was carried out to standardise the bond strength between the titanium alloy and the resin cement. For this purpose, a brass mould was utilised. This mould comprised a 4 mm diameter cavity and a 4 mm diameter cylindrical rod, which fitted into the cavity. The rod was secured using a 1.5 mm thick brass mould, and composite resin (Filtek Z250 Nanohybrid Composite Resin, 3M ESPE AG, Seefeld, Germany) was applied incrementally using the layering technique. The composite resin was then polymerised for 20 seconds using an LED light-curing device (Hilux LED 550 Light-Curing Unit, Benlioğlu Dental, Ankara, Turkey). A total of 52 composite samples were produced.

### Cementation Process and Thermal Ageing Application

The prepared titanium samples were embedded in acrylic moulds within plastic tubes, and composite samples were cemented onto the titanium surfaces using a self-adhesive dual-cure resin cement (RelyX™ U100, 3M ESPE, Seefeld, Germany), followed by polymerisation. The specimen preparation procedure and the experimental setup are schematically illustrated in Figure 3 to facilitate a clearer understanding of the methodology applied in the study. All specimens with titanium-composite bonding underwent thermal ageing in a thermal cycling device (MOD Dental, İstanbul, Turkey) with a temperature range of 5-55°C, a dwell time of 30 seconds, and 6000 cycles.

### Shear Bond Test and Fracture Evaluation

Following the conclusion of the thermal ageing process, the specimens were loaded in a universal testing machine (MOD Dental, İstanbul, Turkey) at a crosshead speed of 1 mm/min until failure occurred. The force at failure was recorded and calculated in MPa. The types of failure were classified as adhesive, cohesive or mixed.

### Statistical Analysis

All statistical analyses were performed using IBM SPSS Statistics for Windows, Version 25.0 (Armonk, NY: IBM Corp.). The descriptive statistics were presented as means and standard deviations. The assumption of normality was assessed using the Shapiro-Wilk test, while homogeneity of variance was evaluated using Levene's test. In instances where a comparison was to be made between two independent groups, an independent samples t-test was employed if the data followed a normal distribution. In cases where this was not the case, the Mann-Whitney U test was utilised. For comparisons involving more than two groups, one-way ANOVA was conducted for normally distributed data, while the Kruskal-Wallis test was used for non-normally distributed data. A p-value of <0.05 was considered statistically significant. It was considered that a p-value of less than 0.05 was statistically significant.

### RESULTS

As indicated in Table 1, the mean shear bond strength values of the groups and their statistical comparisons are presented. The lowest mean value was observed in the GSLM2 group, whereas the highest value was obtained in the GSLM1 group.

**Table 1:** Shear bond strength values and statistical relationships of the groups

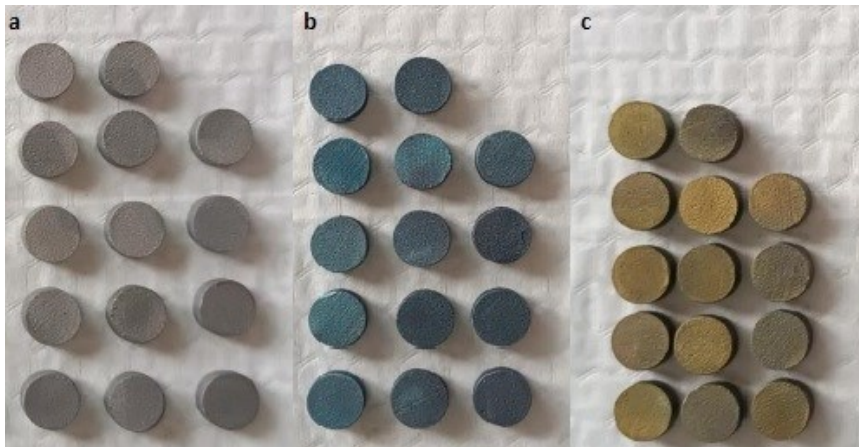
	Mean±SD (MPa)	P	
GM	11.37±4.01	GM-GSLM1*	0.040***
GSLM1	16.91±8.05	GSLM1-GSLM2**	0.033***
GSLM2	9.92±3.25	GSLM1-GSLM3**	0.460
GSLM3	12.30±5.07	GSLM2-GSLM3**	0.791

\*Independent samples t-test was applied, \*\*Kruskal-Wallis test was applied. \*\*\* p<0.05: indicates statistical significance. GM: Titanium group produced by the milling method, GSLM1: Titanium group produced by the selective laser melting method without anodization, GSLM2: Titanium group produced by the selective laser melting method and anodised at 36 V, GSLM3: Titanium cluster produced by additive manufacturing and anodised at 50 V.

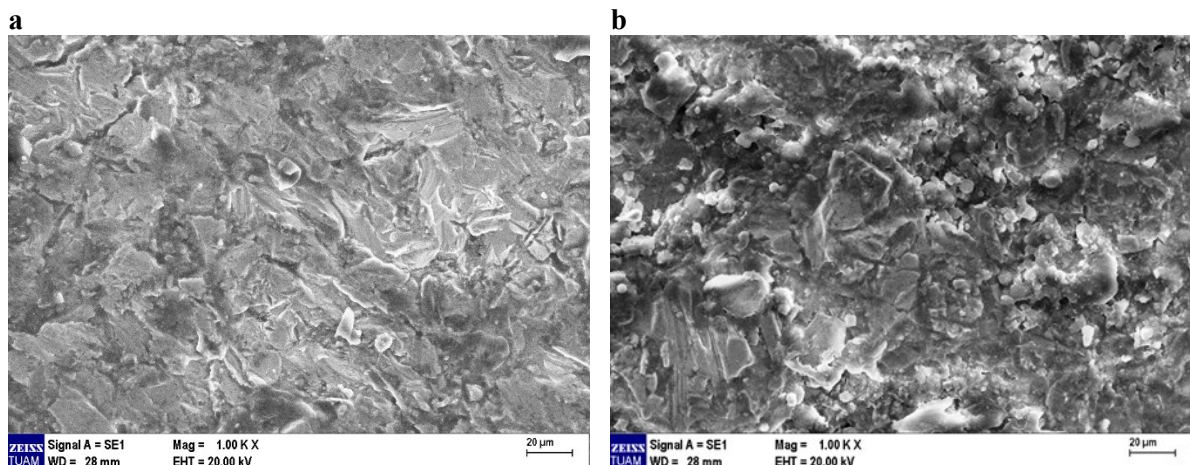
According to the independent samples t-test, the shear bond strength of the GSLM1 group was significantly higher than that of the GM group ( $p = 0.040$ ). The Kruskal–Wallis test applied to the subgroups of the GSLM group revealed a statistically significant difference ( $p = 0.039$ ). Pairwise comparisons indicated that the GSLM1 group had significantly higher shear bond strength than the GSLM2 group ( $p = 0.033$ ). However, no statistically significant differences were detected between GSLM1 and GSLM3 ( $p = 0.460$ ) or between GSLM2 and GSLM3 ( $p = 0.791$ ).

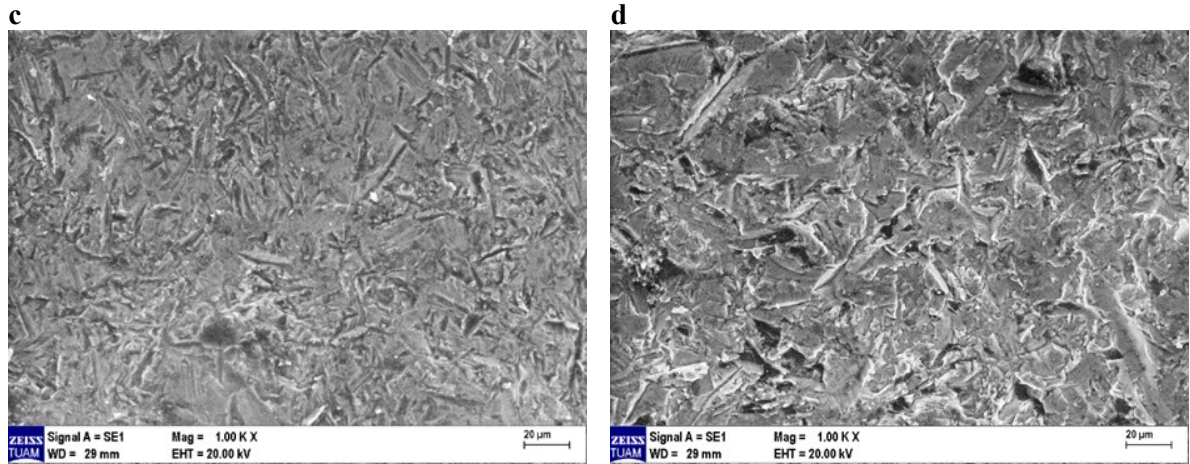
The use of SEM images revealed that the GM group exhibited a relatively homogeneous and smooth morphology. Despite the presence of distinct cracks and stratifications on the surface, the overall structure appeared to be orderly (see Figure 2a). Conversely, the GSLM1 group exhibited a more irregular, porous, and rough surface morphology (see Figure 2b). In the GSLM2 and GSLM3 groups, anodization resulted in more regular, homogeneous, and smoother surface morphologies (see Figures 2c and 2d). Stereomicroscopic examination revealed adhesive failure in all specimens. On the other hand, representative stereomicroscope images from each group are presented in Figure 4.

**Figure 1.** Photographs of the experimental group specimens: (a) GSLM-1, (b) GSLM-2, and (c) GSLM-3.

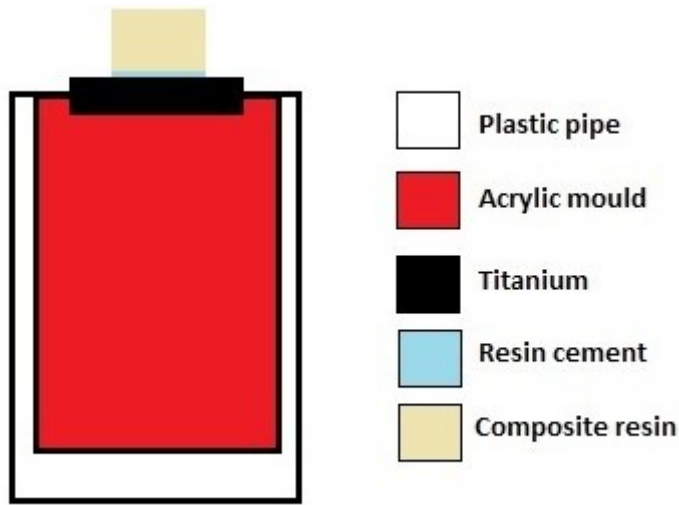


**Figure 2.** SEM images of a sample from different groups. a. Titanium sample made by milling method (GM), b. Titanium sample made by the selective laser melting method without anodizing process (GSLM1), c. Titanium sample prepared by selective laser melting and anodised at 36 V (GSLM2), d. Titanium sample prepared by selective laser melting and anodised at 50 V (GSLM3).

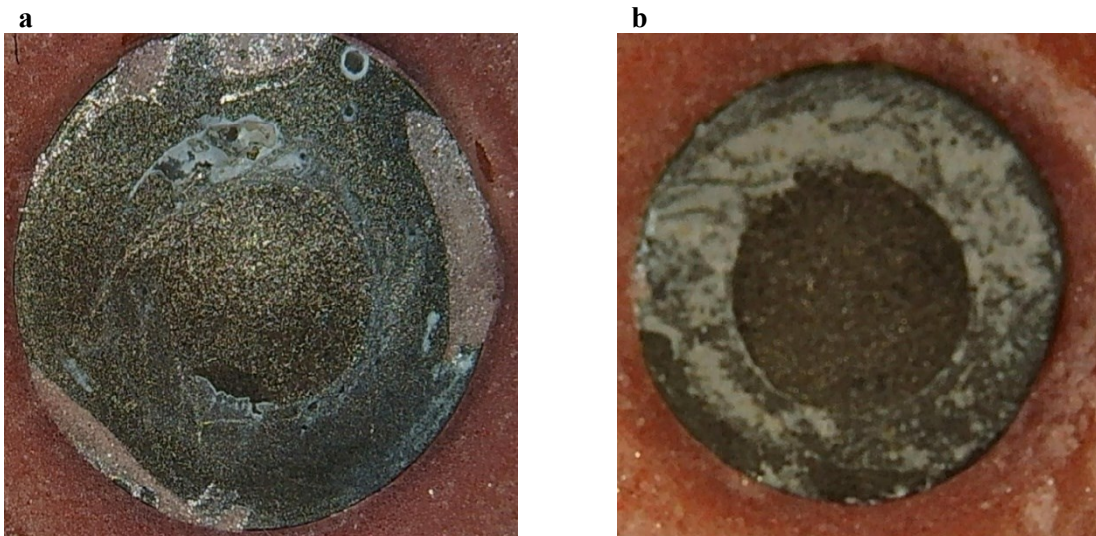


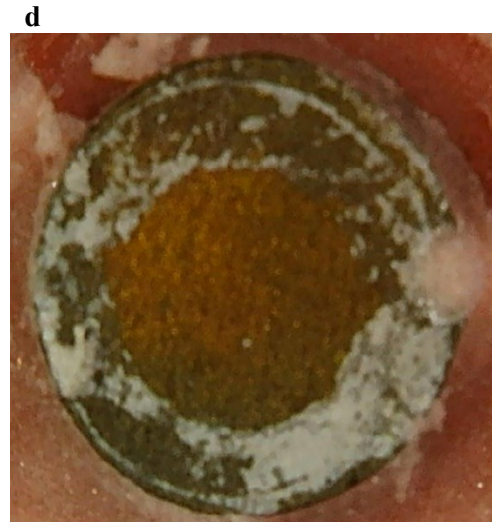
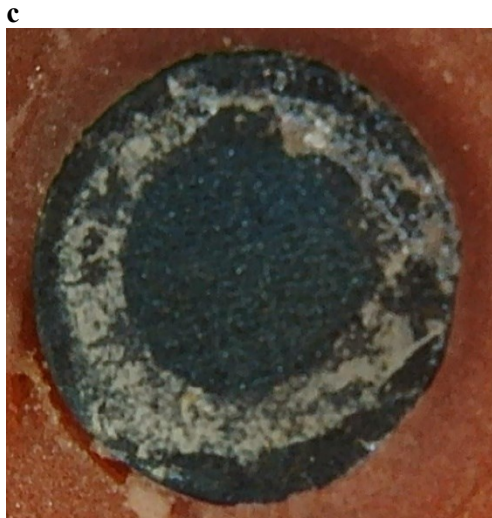


**Figure 3.** Schematic illustration of the specimen preparation procedure.



**Figure 4.** Stereomicroscope images of representative samples from each group. a. Titanium sample made by the milling method (GM), b. Titanium sample made by the selective laser melting method without anodizing process (GSLM1), c. Titanium sample prepared by selective laser melting and anodised at 36 V (GSLM2), d. Titanium sample prepared by selective laser melting and anodised at 50 V (GSLM3).





## DISCUSSION

The present study constitutes an evaluation of the bond strength of titanium produced by additive and subtractive methods with resin cement. Furthermore, the resin cement bond of titanium samples obtained by sandblasting and anodising at different voltages was examined. In light of the research findings, the null hypotheses formulated at the study's inception were refuted, signifying that disparate production techniques and surface treatments applied to titanium produced by additive manufacturing exert a statistically significant influence on the bond between the resin cement and titanium.

Titanium produced by subtractive processes had a smoother surface, while titanium produced by additive processes had a rough and irregular surface structure. This noticeable roughness is attributable to the so-called balling phenomenon, which occurs when isolated powder particles partially melt along the laser scanning path. The presence of partially melted particles on the surface of the alloy was shown to increase surface roughness.<sup>9,13</sup> The hypothesis that the roughness of the alloy surface, characterised by micro-retentive grooves and ridges, promotes mechanical interlocking and increases the effective surface area for

chemical bonding is one that has gained general acceptance.<sup>7,13</sup> A thorough examination of the SEM images of the specimens belonging to the GM and GSLM1 groups revealed that microporous regions were observed with greater frequency in the GSLM1 group. In terms of resin-cement bond strength, a significant difference was identified between the GSLM1 and GM groups. The former exhibited higher bond strength. The enhanced bond strength exhibited by the GSLM1 group can be ascribed to the augmented density of microporous regions that have formed on the surface of the titanium specimens produced by the SLM method, resulting in an enlarged surface area. Despite the paucity of studies comparing the bond strength of titanium produced by the additive and subtractive methods with resin cement, there are studies evaluating the bond strength of these production methods between titanium and ceramics. The results of these studies suggest that titanium produced by the milling method has low bond strength with ceramics due to its surfaces containing less microporosity.<sup>14,15</sup>

The present study evaluated the effect of Al<sub>2</sub>O<sub>3</sub> sandblasting and anodising at varying voltage levels on the bond strength between titanium and resin cement on the surface of titanium samples produced by an additive manufacturing technique. Acar et al.

investigated the effect of Al<sub>2</sub>O<sub>3</sub> sandblasting and sodium hydroxide anodising methods applied to the surface of pure titanium on the bond strength between titanium and ceramic. The study concluded that the SEM examinations of the anodised and sandblasted surfaces demonstrated that the anodised group exhibited a flatter surface topography and reduced porosity, while the Al<sub>2</sub>O<sub>3</sub> sandblasting method exhibited a more favourable effect on the titanium-ceramic bond.<sup>8</sup> In the study conducted by Amornwichtwech and Palanuwech, it was reported that the formation of a flat and smooth film layer on the anodised titanium surface resulted in a compromise to the integrity of the titanium-ceramic bond. The bond strength exhibited by the anodised titanium surface was found to be lower in comparison to the bond strength observed in the alumina sandblasted group.<sup>16</sup> In the study by Bömicke et al., the bond strength between resin cement and titanium was evaluated. The study concluded that anodisation could be applied without weakening the bond strength between titanium resin cement and titanium.<sup>17</sup> In the current study, the highest bond strength was observed in the GSLM1 group, which was sandblasted only, while the shear bond strength was lower in the GSLM2 and GSLM3 groups, which were anodised after sandblasting. The reduced bond strength can be attributed to the process of anodising that follows sandblasting, a procedure which results in a diminution of the microporous areas thus created. This hypothesis is further substantiated by the results obtained from the SEM images, which demonstrate that the regions of porosity in the sample from the GSLM1 group exhibit a greater intensity compared to those observed in the GSLM2 and GSLM3 groups. Conversely, the mean bond strength for the GSLM2 and GSLM3 groups was 9.92 and 12.30 MPa, respectively, which approaches the clinically acceptable threshold of 10 MPa.<sup>17</sup> These findings suggest that anodised titanium abutments can rapidly enhance esthetics without compromising clinical outcomes.

The thickness of the oxide layer formed during titanium anodising increases in direct proportion to the applied voltage. This process results in the formation of a compact dielectric barrier that hinders the movement of ions through the oxide. However, when the dielectric breakdown voltage of the oxide is exceeded, this barrier becomes ineffective, and the oxide continues to grow. This process, known as dielectric breakdown, results in the formation of porous titanium oxide films on the titanium surface. The dielectric breakdown voltages in sulfuric acid electrolytes are approximately 110 V, while those in phosphoric acid electrolytes are approximately 200 V.<sup>18-20</sup> In the present study, phosphoric acid was utilised as the electrolyte, and the shear bond strength of the GSLM3 group anodised at 50 V was found to be higher than that of the GSLM2 group anodised at 36 V, although this difference was not found to be statistically significant. The enhanced bond strength exhibited by the GSLM3 group may be attributable to the anodising voltage that approaches the dielectric breakdown point, resulting in the formation of a more porous surface structure. This, in turn, leads to an increased resin cement retention. This finding is further substantiated by the higher density of porous areas observed in the SEM image of the GSLM3 group sample compared to the GSLM2 group.

The present study is subject to several limitations. Firstly, the sample size was limited and only one type of resin cement was utilised in the study. A further significant limitation is that merely two voltage values were utilised for the anodising process. As the GM group was selected as the control group, it is important to note that no anodising process was applied to this group. Furthermore, no additional surface treatments were conducted beyond sandblasting and anodising, and surface roughness was not evaluated. Despite the utilisation of thermal cycling as the prevailing ageing technique, the employment of advanced methodologies

that more precisely replicate the intraoral environment was not undertaken. It is recommended that comprehensive studies be conducted in the future, utilising a range of techniques and materials, and employing larger sample sizes.

### CONCLUSION

Within the limitations of the study, it was concluded that the resin-cement-titanium bond produced by the additive method was higher than that of titanium produced by the subtractive method and that only Al<sub>2</sub>O<sub>3</sub> sandblasting contributed more positively to the titanium-resin-cement-bond produced by the additive method than anodization applied at 36 and 50 V after sandblasting. However, the findings of the study, based on the examination of manufacturing techniques and surface treatments, concluded that a resin-cement-titanium bond with clinically acceptable strength was observed. The anodisation process, a technique employed to enhance the aesthetic appearance, can be safely utilised in a clinical setting.

### Ethical Approval

An ethics statement was not required for this study type and no human or animal subjects or materials were used.

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The authors declare that this study received no financial support.

### Conflict of Interest

The authors deny any conflicts of interest related to this study.

### Author Contributions

Design: NU, Data collection or access: NU, SP, Analysis and comments: NU, Öİ, AE, Literature search: NU, Writing: NU, Öİ.

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