Original Article / Özgün Araştırma

The Comparison of Different Surface Preparation Methods in Terms of Shear Bond Strength of Tri-ceram Porcelain-titanium Alloy

Tri-ceram Porselen ve Titanyum Alaşımı Arasındaki Makaslama Bağlantı Direnci Üzerine Farklı Yüzey Uygulamalarının Etkisi

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Abstract

Objective: Several studies have been made to evaluate effects of the surface preparation on shear bond strength (SBS) of titanium-porcelain complex. However, a completed picture has not been obtained yet. Therefore, such studies appear frequently on recent literature. The purpose of this study is to determine a simple method providing strong SBS and using fewer parameters.

Materials and Methods: Sixty titanium samples were equally divided into five groups. Group 1: Control (C). Group 2: Airborne-particle abrasion with 250 μ Al_2O_3 (250 μ AbPA). Group 3: Etching with 10% HCl (10% HCl). Group 4: Etching with Nd:YAG Laser at 6W (Nd6). Group 5: Etching with Nd:YAG laser at 7 W (Nd7).

Results: Mean SBS value of C (10.69 MPa) was close to that of 250 μ AbPA (10.57 MPa). The mean value of 10% HCl (19.37 MPa) was nearly twice higher than C, whereas those of laser groups (8.89 MPa and 8.77 MPa) were smaller than C. There was no overlap between SBS values of samples etched with 10% HCl and those of other samples. Multiple comparisons indicated a significant difference between acid group and others (p=0.00). Laser groups were different from control, too. The failure mode of % HCl group was 67% adhesive and 33% mix, while those of other groups were cohesive or mix.

Conclusion: Titanium surfaces etched with 10% HCl provides significantly strong SBS values. This method requires only the use of aqueous solution of 10% HCl, and boiling process for 30 minutes. Such a simplicity suggests that etching with 10% HCl provides a very simple surface preparation method which involves in use of fewer parameters.

Öz

Amaç: Yüzey pürüzlendirme işlemlerinin titanyum porselen bağlantısının kopma direnci üzerine etkilerini değerlendiren birçok çalışma yapılmıştır. Buna rağmen henüz, bu alandaki yöntemlerin standardizasyonu sağlanamamıştır. Bu nedenle son yıllarda bu konuyla ilgili çalışmalara sıkça rastlanmaktadır. Bu çalışmanın amacı, yüzey uygulama metodları ve doğru parametreleri kullanarak kopma direnci sonuçlarını değerlendirmektir.

Keywords
Shear bond strength, titanium alloy, surface preparation, Tri-ceram porcelain

Anahtar Kelimeler
Makaslama kopma direnci, titanyum alaşımı, yüzey pürüzlendirme, Tri-ceram porselen

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Gereç ve Yöntemler: Altmış titanyum örnek 5 gruba eşit sayıda bölündü. Grup 1: Kontrol, grup 2: Al2O3 (250 µm) ile kumlama, grup 3: Asit (%10 HCl) ile pürüzlendirme, grup 4: 6 W lazer (Nd:YAG) uygulama, grup 5: 7 W lazer (Nd:YAG) uygulama.
Bulgular: Kontrol grubunun ortalama değerleri (10,69 MPa), kumlama grubuyla (10,57 MPa) yakın bulundu. Asit grubunun ortalama değerleri (19,37 MPa) önemli ölçüde kontrol grubundan fazla bulunurken, lazer grupları değerleri (8,89 MPa ve 8,77 MPa) kontrol grubundan düşük bulundu. Çoklu karşılaştırma testi sonucunda asit grubuyla diğer gruplar arasında anlamlı bir fark bulundu ($p<<0,05$). Altı W'luk lazer uygulaması dışındaki gruplarla kontrol grubu arasında anlamlı bir fark bulunmadı. Asitle pürüzlendirme grubunda %75 adeziv ve %25 miks kopma görülürken diğer gruplarda kohesiv ve miks kopma görüldü. Adeziv kopma tipinin ortalama makaslama bağlanma direnci değeri (20,31 MPa), kohesiv (9,48 MPa) ve miks (10,90 MPa) tipleri değerlerinden yüksek elde edildi. Adesiv kopmanın değerleri istatistiksel olarak diğerlerinden farklı bulundu ($p<<0,05$).
Sonuç: Titanyum yüzeyinin %10 HCl asit ile pürüzlendirilmesi güçlü bir makaslama kopma direnci göstermektedir. Bununla, titanyum yüzeyinin asitle pürüzlendirilmesinde uygun bir konsantrasyon olduğunu önerilmektedir.

Introduction

Several studies have been carried out to investigate the effects of surface preparation on shear bond strength (SBS) of metal-porcelain complex. The SBS values for titanium-porcelain complex have been evaluated by several methods such as acid etching (1-17), alumina airborne-particle abrasion (7-14,17-30), laser irradiation (6,15,16,22,27,30), fluoride etchant application (8,9,12,13), nanotechnology (10), machining/milling/thermic treatments (8,9,13,23,26,31) and cooperative use of simple surface preparation methods (2-4,9). The SBS has also been investigated for the influence of various parameters such as acid types and concentrations (2-4,9), treatment with and without vacuum (3), laser types (6,16,30), radiation power of laser irradiation (15,27;30), size of Al_2O_3 particles (22,23,25,26,28), resin cements/bonding agents (3,16,17,21,25,26,32-35), surface coating (20,24,36,37), interfacial oxidation (38-40), ceramic types (5,7,15,41,42). Duration and temperature for surface preparation (2,5,8,9,13,15), firing temperature (20,43), storage in water and water loading (7,9,11,16,26,27,41,44), thermocycling (8,13,16,18,25, 26), distance in airborne abrasion (13), area fraction of adherent porcelain (19) and crosshead speed of universal testing machine (13,14,22,24). In addition, several review papers have been published in this field (42,45-47)

Different materials, diferent methods, different experimental conditions, various parameter settings, and combination of several methods for the surface preparation have yielded different values of the SBS. Depending on experimental conditions and chosen parameters, even contradictive results have been obtained in some studies (1,6,13,14,16,17,22,27-29). In the other words, a completed picture has not been obtained yet. Therefore, the studies on the SBS of titanium-ceramic complex are still interest of clinical research, and such studies appear frequently on the recent literature (5,10,11,13,14,18,28-30,35,42). For these reasons, a new study done by using the simple surface preparation methods and specific parameters may contribute to complementary studies in this field.

The aim of this *in vitro* study was to reveal simple surface preparation methods providing strong SBS for Tri-ceram porcelain-titanium complex**.** The hypothesis of the study was to obtain confirmative results contributing to the studies on the strong SBS For these purposes, the SBS values between titanium Tri-ceram porcelain complex were obtained by simple surface preparation methods such as acid etching, alumina airborne-particle abrasion and laser etching. Some particular parameters for each method were also chosen. The effects of these methods on the SBS between a titanium and porcelain were compared.

Materials and Methods

Sample Preparation and Measurements

Titanium bars (ASTM F67-00; Titanium Industries, Inc, Rockaway, NJ) were sectioned with a lathe (computer numerical control auto lathe type SA-12 S/N 0910; Star Micronics Co, Ltd, Shizuoka, Japan) into 60 specimens, with 2.2 mm length and 5.7 mm in diameter. All specimens were machine cut from long metal rods to the same specified dimensions. No specific surface treatment was performed for the machined surface group, which served as the control group (C) . The samples were equally divided into five groups for surface preparation (12 samples for each group).

The airborne-particle-abraded surface specimens (12 samples) were abraded with alumina particles

(250 μm) with a dental airborne-particle-abrasion unit (Micro-blaster; Daedong Industrial Co, Ltd, Daegu, Korea). The air pressure was set at 2 bar, and the distance between the nozzle tip and the specimen surface was maintained at 15 mm, during the airborne-particle abrasion, for 20 seconds (10 scans in 20 seconds, at the rate of 1 scan every 2 seconds.

The acid-etched surface specimens were subjected to chemical surface treatment by submerging the specimens in a 10%-by weight aqueous solution of HCl (DC Chemical Co, Ltd, Seoul, Korea) in a heat-resistant glass container and boiling for 30 minutes, taking care to avoid contact between specimens.

The laser-etched surface specimens were treated using a custom-made pulsed Nd:YAG laser (Jenoptic Laser Optik Systeme GmbH, Jena, Germany). The titanium surfaces of the specimens were irradiated by the linear movement of a glass fiber of the Nd:YAG laser at a power setting of 7 W and 6 W, representing energy and frequency levels of 120 mJ with 50-Hz frequency

The groups were as follows:

Group 1 (C): Control (no treatment).

Group 2 (250 m AbPA): Airborne-particle abrasion with Al2O3 particles (250 m).

Group 3 (%10 HCl) : Surfaces etched with %10 HCl

Group 4 (Nd6): Surfaces etched with Laser (Nd:YAG laser) at 6 W irradiation power.

Group 5 (Nd7): Surfaces etched with Laser (Nd:YAG laser) at 7 W irradiation power.

Before application of porcelain, the samples were replaced in ultrasonic cleaning apparatus at 80 °C for 10 minutes. Then, they were washed with distilled water. Low-fusing porcelain (Tri-ceram; Esprident GmbH, Ispringen, Germany) was used in this investigation. Firing temperature and times were in accordance with the manufacturer's specifications and directions. Heat pretreatment of the specimens was performed immediately after cleaning procedures in a dental porcelain furnace (Austromat 3001; Dekema GmbH, Freilassing, Germany). Opaque porcelain was mixed as a powder and liquid until it reached a creamy consistency, then applied in 2 uniform coats with a brush on each treated porcelain-bearing surface. After the opaque porcelain firing cycle (795 °C), the dentin porcelain was subsequently formed on the opaque layer, using a specially designed silicone mold, and fired at 500 °C to 755 °C with a heat rate of 55 °C/min under a vacuum of 72 cm/Hg. The firing shrinkage was compensated for with a second body porcelain application, until an approximately 4-mm thick porcelain layer was obtained. A glazing procedure was not performed. Porcelain application for all of the specimens was performed by a single dental technician. The Tri-ceram were then adhered to the surface treated samples using Clearfil SE Protect (Kuraray).

The following treatments were applied to the prepared samples:

1- The samples were mechanically loaded (20.000 cycles; 50 N load; distilled water at 37 °C).

2- The samples were thermocycled (3.000 cycles; 5-55 °C, dwell time: 30 sec).

To evaluate the bond strength of the interface between the metal and ceramic, the shear bond test, which has been described by other investigators, was performed. For the shear bond test, a special stainless steel device was fabricated. This device enabled the specimen to be held firmly during the shear bond test. The device containing the metal ceramic specimen was placed in a tensile testing machine (micro 500, type U4000, Maywood Instruments. Limited Basingstoke Hants. England). The power loading point was 5 mm far from metal porcelain connection, while the speed of loading was 5 mm/minute The load was applied until fracture of the metal-porcelain interface occurred, and the maximum load at fracture was expressed in megapascals (MPa). After fracture, scanning electron microscope (SEM) (JSM-6700F; JEOL Ltd, Tokyo, Japan) observation was once more performed to evaluate the nature of the fractured surfaces. Three photomicrographs with x2000 magnification were made of different regions of the treated surface and the fractured surface of each specimen.

Statistical Analysis

Results were presented as the mean + standard deviations (SD). One-way ANOVA followed by Games-Howell post hoc test (α=0.05) was used for comparisons. $P<0.05$ was considered as statistically significant.

Results

Mean SBS values and SD of all groups were given in Table 1. The mean values of C are close to that of alumina airborne-particle abrasion. The mean value of acid group is almost twice higher than control, whereas those of laser groups are smaller than control. There is no overlap between maximum SBS values for 250 μ AAbPA and minimum SBS values for acid etching. There is large overlap among the SBS of other groups.

Multiple comparisons of groups are given in Table 2. It is seen that the SBS of 10% HCl etching group is highly significantly different than those of other groups (p=0.00). There is no significant difference between 250 m AbPA and control, and also between 250 m AbPA and Nd6 (p>0.05). 250 m AbPA is different from Nd7 (p=0.05). In addition, C is different from both laser groups (p<0.05).

The numbers of adhesive, cohesive and mix failure modes, determined by SEM, were 8, 21 and 31 respectively. Adhesive failure mode was only observed in 8 of 12 samples etched with acid, but cohesive mode was found in all groups other than 10% HCl. Mixed failure mode was exist in all groups.

SEM Images Obtained From Surface Preparations

SEM images of C, 250 μ AbPA, 10% HCL and Nd7 are shown in Figures 1,2,3 and 4, respectively The

Table 1. The mean values of shear bond strength together with standard deviation for investigated groups

Groups	Number	Mean SBS values ± SD (MPa)	min-max
Control (C)	12	10.69+1.40	7.20-12.10
250 μAbPA	12	10.57+1.47	8.40-12.90
10% HCl	12	19.37+2.99	14.40-25.40
Nd6	12	8.89+1.11	7.30-10.90
Nd7	12	8.88+2.34	6.60-13.80

SBS: Shear bond strength, SD: Standard deviation, MPa: Megapascal, min: Minimum, max: Maximum

Table 2. Statistical significances between groups

Comparison between groups		Significance (p)
10% HCl	C 250 μAAbPA Nd6 Nd7	0.000 0.000 0.000 0.000
250 mA AbPA	C Nd6 Nd7	0.998 0.075 0.050
C	Nd6 Nd7	0.049 0.032
Nd6	Nd7	0.990

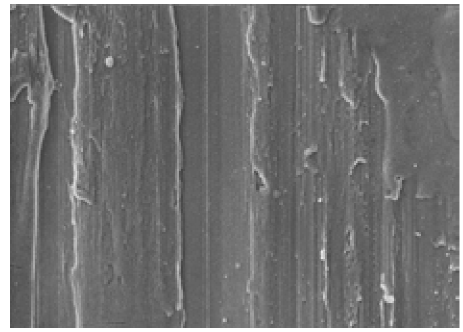

Figure 1. Scanning electron microscope image of control group

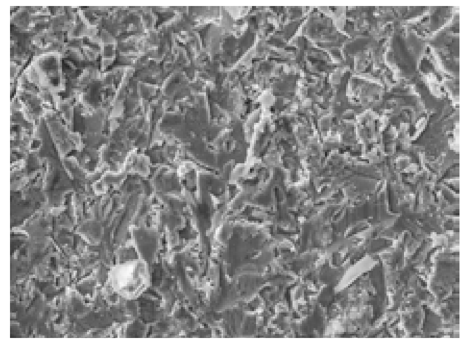

Figure 2. Scanning electron microscope image of airborne-particle ablated with alumina particle group

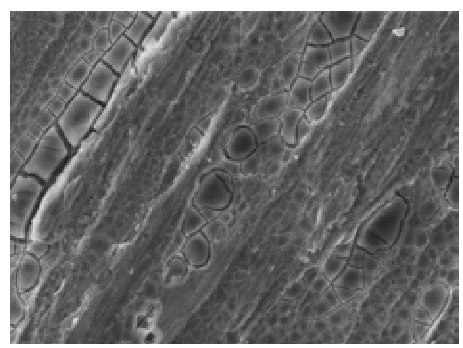

Figure 3. Scanning electron microscope image of acid etched group

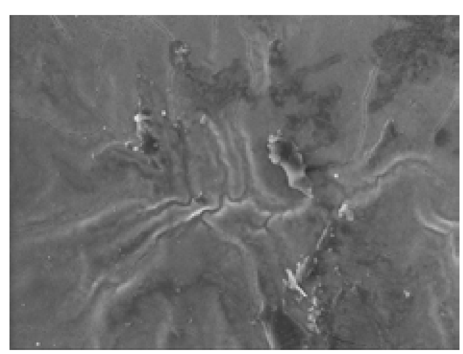

Figure 4. Scanning electron microscope image of laser group at 7 Watt

image for Nd6 was not given for the sake of simplicity. Smooth surface was observed for both C and Nd6. The roughest surface was observed in 250 μ AbPA. In addition to these, laser irradiated samples at Nd7 had a smoother surface. Nevertheless some detonation points and black areas were found on SEM images of the Nd7.

Discussion

The success of the porcelain-fused-alloy restoration depends widely on the strong bonding between porcelain and the titanium. In the current work, the titanium surface treatment with 10% HCl has provided strong SBS values, and acid etching was found to be more effective than alumina airborne-particle abrasion, laser etching and C. In fact, acid etching has been indicated as mostly quite effective method for increasing SBS in titanium-porcelain system (1-4,12,16,18). Therefore, the present results obtained with 10% HCl are in consistent with the previous studies related to surface acid treatment. However, in the previous studies, the surface acid etching has been reinforced by other treatment methods (2,4,16). Also titanium surfaces have been treated by using highly concentrated acids (e.g. 48% H_2SO_4) different than HCl (3,12,16). On the contrary, even 1 N (about 3%) HCl is known to erodes the titanium surface effectively and to provide increasing SBS values (18).

Airborne-particle abrasion has been noted to weaken the metal ceramic bonding in some studies (1,6,24). Alternative methods to the airborne-particle abrasion have also presented (1,3,6,10,12,27). However, airborne-particle abrasion with certain size of Al2O3 (e.g. 110 and 250 m Al2O3) is known to increase bonding in the metal ceramic system (6,13,18,29). It was noted that SB increased as particle size increased (29). Despite this, the combined use of alumina airborne-particle abrasion with other surface preparation materials and methods has mostly been used to obtain strong SBS (2,8,9,14,17,19,21,23,26). The diversity of SBS values including the current value for 250 μA AbPA should be related to parameter settings and experimental conditions. In fact, the strong SBS in the airborne-particle abrasion is dependent on many factors such as particle size of Al_2O_3, bonding agent, water storage, etching times, laser welding, metal conditioners, vacuum firing, cooperative use of simple surface preparation methods, thermic treatment, order of cooperative treatment, pressure and angle used (3,7, 13,14,17,18,21,22,24,26,28,31).

The Nd:YAG laser etching has been found found to be effective improving bond strength of titanium-ceramic system in some studies (6,15,27), whereas lower SBS was obtained by laser irradiation in other studies (16). High variability was observed in adhesion values obtained by laser etching (42). In fact, the efficiency of laser irradiation is dependent on various parameters such as irradiation power, laser type etc. (15,30). For example, Er:YAG and Nd:YAG lasers applied with certain power have yielded the stronger SBS (27,30). However, Nd:YAG laser was found to be more successful than Er:YAG and Ho:YAG lasers for bonding low fusion porcelain to metal alloy (30). In addition to these, laser applications or other surface preparation methods may be more successful when applied to ceramics or other type of metal alloys. The SBS obtained with laser etching or without laser have been found to be strong for the ceramic-Ni-Cr or ceramic- Co-Cr alloys (48-51).

Adhesion is the tendency of dissimilar surfaces to stick to one another. There are many types of forces that can occur when surfaces come in close contact (48,49). Rather than inter molecular forces between dislike molecules, mechanical and chemical forces provide binding of metal to ceramic where failure mode is adhesive. In mechanical binding, a strong bond is formed between the substrate and the adhesive (48,49). Also, Chemical binding is usually the strongest form of adhesion (48,49). Since adhesive failure is dominant on the surfaces treated with 10% HCl, the current strong mean SBS obtained for titanium-ceramic system should be related to ontrol mechanical or chemical binding.

Smooth surface appearance obtained by SEM for laser irradiation groups are in agreement with the small SBS and cohesive failure mode of these groups. The SEM appearance with relatively rough surface in acid etching group is in consistent with strong adhesive bonding that is likely produced through mechanical or chemical interactions. The roughness in the alumina airborne-particle abrasion may be related to long-distance adhesive interactions since cohesive failure mode is dominant for this case. In addition to these, the detonation points and black areas on SEM images of laser group etched at 7 W power express that increasing energy creates burning areas. The connection between titanium and porcelain becomes weaker in this area. Therefore, the use of a laser at 6 W energy should be more convenient.

As mentioned earlier, variability of the SBS values obtained from titanium-ceramic complex is dependent on surface preparation methods, experimental conditions and parameter settings. Multitude of parameters and conditions requires elimination of some of them which are not providing simple method and strong SBS. In our case, etching titanium surface by using acid requires only the use of aqueous solution of 10% HCL, and boiling process for 30 minutes. Therefore, it is a very simple method providing significantly strong SBS. Thus the aim or hypothesis of our study was fulfilled.

Conclusion

Etching titanium surface with 10% HCl has provided a strong bonding for titanium-ceramic complex. This suggests that etching with 10% HCl provides very simple method for surface preparation of titanium.

Ethics

Ethics Committee Approval: The study were approved by the Dicle University of Local Ethics Committee (protocol number: 2018/7).

Informed Consent: The authors confirm that this article content has no conflict of interest.

Peer-review: Internally peer-reviewed.

Authorship Contributions

Concept: B.D.Y., A.M., E.Ö., Design: B.D.Y., A.M., E.K., Data Collection or Processing: E.K., Analysis or Interpretation: A.M., Literature Search: E.K., Writing: B.D.Y., A.M., E.K.

Conflict of interest: The authors confirm that this article content has no conflict of interest.

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