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SEARCHING THE EFFECT OF ANODIZATION PROCESS AS A SURFACE TREATMENT

Yüzey İşlemi Olarak Uygulanan Anodizasyonun Etkilerinin Araştırılması

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ABSTRACT

Objective: The aim of this study was to compare the effect of anodization and air-particle abrasion surface treatments on shear bond strength between Ni-Cr and ceramics.

Material and Methods: 30 cylindrical Ni-Cr specimens (7x10 mm) were divided into three groups according to the surface treatments [control group (no-treatment), air-particle abrasion group (110 µm Al_2O_3 at 75 psi from 20 cm for 30 seconds), and anodization group]. One sample from each group was evaluated by using SEM. Ceramics with a dimension (5x3 mm) built-up on specimens and shear bond strength tests were performed by using universal testing machine with a 1 mm/min crosshead speed. Stereomicroscope was used to evaluate the failure mode of specimens. One-way ANOVA with post-hoc Tukey's test was used to analyze the differences in shear bond strength values.

Results: The shear bond strength of air-particle abrasion (21.35∓4.64) was higher than anodization group (20.92∓4.85) however, no statistically significant difference was detected (p=0.893). Both groups showed higher bond strength than control group (8.02∓1.47) ($p<.05$).

Conclusion: The evaluation of the surface treatment methods showed that the anodization process can be used in order to increase the metal-ceramic bond strength.

Keywords: Air-particle abrasion, anodization, Ni-Cr, platinum, shear-bond strength.

ÖZ

Amaç: Bu çalışmanın amacı, anodizasyon ve kumlama yüzey işlemlerinin Ni-Cr ve seramik arasındaki makaslama bağlantı dayanımı üzerine etkilerini karşılaştırmaktır.

Gereç ve Yöntem: 30 adet silindir şeklinde Ni-Cr örnek (7x10 mm) uygulanan yüzey işlemlerine göre üç gruba ayrıldı [kontrol group (hiç işlem görmedi), kumlama grubu (110 µm Al_2O_3, 75 psi basınçla, 20 cm uzaklıktan, 30 saniye süresince) ve anodizasyon grubu]. Her gruptan birer örnek alınarak SEM analizleri gerçekleştirildi. Hazırlanan örneklerin üzerine 5x3 mm boyutlarında seramikler pişirildi ve makaslama bağlantı dayanım testleri universal test cihazında kafa hızı 1mm/dk olacak şekilde gerçekleştirildi. Örneklerin kopma şekillerinin tespitinde stereomikroskop kullanıldı. Makaslama bağlantı dayanım değerlerinin analizinde One-way ANOVA ve post-hoc Tukey's testleri kullanıldı.

Bulgular: Kumlama grubunun makaslama bağlantı dayanımı (21,35∓4,64) anodizasyon grubuna (20,92∓4,85) göre yüksek olarak bulunmasına rağmen her iki grup arasındaki farklılık istatistiksel olarak önemli bulunmamıştır (p=0.893). Her iki grupta kontrol grubuna göre (8,02∓1,47) yüksek bağlantı dayanımına sahip olduğu tespit edilmiştir ($p<0,05$).

Sonuç: Yüzey uygulama metotlarının değerlendirilmesi sonucunda anodizasyon uygulamasının metal-seramik bağlantısının artırılması için kullanılabilecek bir yöntem olduğunu tespit edilmiştir.

Anahtar kelimeler: Kumlama; anodizasyon; Ni-Cr; platin; makaslama bağlantı dayanımı.

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INTRODUCTION

In prosthetic restorations, in order to mimic the natural appearance of the tooth, all ceramic restorations have been using with an increasing demand.[1] Although more aesthetic results can be obtained with full ceramic applications, due to the high production costs, the sensitive cementation procedure, and unsuitability to apply in every case, metal-ceramic restorations still the most frequently used fixed partial denture which have long term clinical success. Noble metal alloys have good biocompatibility, superior bond strength with ceramic and mechanical properties, besides the increased cost of this material limits the usage.[2] Despite the difficulty of manipulation because of the harder structure and need for higher temperature for casting, Ni-Cr and Cr-Co are the most preferred alloys due to its cost-effectiveness.[3]

The most susceptible area of metal-ceramic fixed partial denture to fracture is the metal ceramic interface, which plays a curial role for life span of the restoration.[4] In order to improve the bond strength between metal-ceramic, air-particle abrasion (APA), laser, and acid-etching applications have been using.[5–7] APA is the most common method among them, which increases surface roughness of substructure by creating micropores and undercuts to boost mechanical interlocking.[8] It is easy to perform and the obtained metal-ceramic bond strength is sufficient which is mentioned 25 MPa according to the ISO standard.[9] The increased particle size of Al_2O_3 with an increasing pressure and application time has potential to increase the surface roughness however, bond strength increase is not parallel to surface roughness increase.[10] Besides, Gilbert *et al.*[11] mentioned that the particles embedded into the metal surface could contaminate the metal substructures and deteriorate the metal-ceramic bond strength and could cause allergic reactions and decrease the corrosion resistance.

The anodization process, an electronic passivation technique, attracted a noteworthy interest due to its ease of application and the reproducibility of the obtained results.[12] In recent years anodization has been using as a surface treatment for titanium implants in order to make rougher surface to enhance osteointegration.[13] In this process, the material used as an anode dissolve in an electrolytic medium and encapsulate the material used as a cathode. After covering the material, its surface properties change and acts as the material used in coating process (anode). Besides, this technique forms an oxide layer on metal surface in a controlled way. The oxide layer on metal surface plays a crucial role on metal ceramic bond strength which forms chemical adhesion with oxides of ceramics[14] while the excessive amount of this layer decreased the metal ceramic bond strength.[15] The anodization process is used to increase material’s the corrosion resistance and surface roughness however, its effect on metal-ceramic bond strength is unclear.

The purpose of the present study was to compare the effects of anodization and APA on shear bond strength (SBS) between Ni-Cr substructure and ceramic. The present study is a novel approach in prosthetic dentistry. The null hypothesis tested was that the anodization would not be as effective as sandblasting on metal-ceramic bond strength.

MATERIAL AND METHODS

30 Ni-Cr metal cylinders (System KN, Adentatec, Köln, Germany) with a diameter 7 mm and height of 10 mm were used for testing as they were produced. The composition of the metal is depicted in Table 1. The specimens’ surfaces were polished with P0001-220 silicone polisher (NTI silicone, Kerr, CA, USA). They were randomly divided into three groups according to the surface treatments [control group (no treatment), air-particle abrasion (APA) with 110 µm Al_2O_3 (Metoxides, Dortmund, Germany) at 75 psi from 20 cm for

30 seconds and anodization] (n:10). In APA group, specimens were ultrasonically cleaned with distilled water for 10 min than dried at room temperature after surface treatment. One specimen from each group was examined under scanning electron microscopy (SEM) (LEO 440, Zeiss, Jane, Germany).

Table 1. The composition of metal alloy (%)

Ni	**Cr**	**Mo**	**Si**	others
61.9	25	11.5	1.4	<0.1

In anodization group, one side polished 10 Ni-Cr samples were ultrasonically degreased consecutively in acetone, 2-propanol and deionized water for 30 min, and then dried in a nitrogen stream. The samples were etched in ethylene glycol containing 0.4 wt.% NH_4F (Sigma Aldrich, Darmstadt, Germany) and 5 wt % water content by using a two-electrode electrochemical cell Ni-Cr as a cathode and a platinum gauze as a counter electrode (anode). 30 V voltage was applied, and the electrolyte temperature was at 25 ^{0}C. After anodization, the prepared samples were thoroughly washed with a large amount of distilled water and methanol to remove precipitations.

Shear bond strength tests

Ceramic application (Ceramco3, Denstply, Hanau-Wolfgang, Germany) was performed by using a custom-made metal mold which has cylindrical holes (diameter 5, thickness 3 mm) than fired (Multimat Easy, Denstply, Hanau Wolfgang, Germany) according to manufacturers' instructions which is shown in Table 2. All specimens were stored in distilled water for 24 h at 37 C^{0}. Shear bond strength tests were performed by using universal testing machine (Lloyd LF Plus, Ametek Inc, Leicester, UK) with a 1 mm/min crosshead speed. Stereomicroscope (Stemi DV4, Zeiss, Jane, Germany) at x30 magnification was used to evaluate the failure mode of specimens. The failure modes were classified as follows:

Type A: Adhesive failure (on the interface)

Type C: Cohesive failure (within ceramics)

Type AC: Combined failure.

Table 2. Firing schedule

	Pre-drying		**Firing**	
	Temperature (^{0}C)	Time (min)	Heating rate (^{0}C/min)	Firing temperature (^{0}C)
Opaque	650	3	70	970
Dentin	650	3	50	950

Statistical analysis was performed using the SPSS 22.0 (SPSS Inc., Illinois, USA). One-way ANOVA with post-hoc Tukey's test was used to analyze the differences in shear bond strength values. The significance level was set at $p<.05$ for statistical procedures.

RESULTS

The mean shear bond strengths, standard deviations and failure modes for all groups are depicted in Table 3 and Figure 1. In terms of bond strength there was no significant difference between the surface treatment groups (p=0.893).

Table 3. Shear bond strength, standard deviation and failure mode

	SBS (MPa) and SD	**Adeshive**	**Cohesive**	**Mix**
Control	$8,02 \mp 1,47^{a}$	8		
Sandblasting	$21,35 \mp 4,64^{b}$	3	2	3
Anodization	$20,92 \mp 4,85^{b}$	6	2	

Shear bond strength (SBS); standard deviation (SD). $^{(a)(b)}$The values with same superscript mean no statistically significant difference (p=0.893)

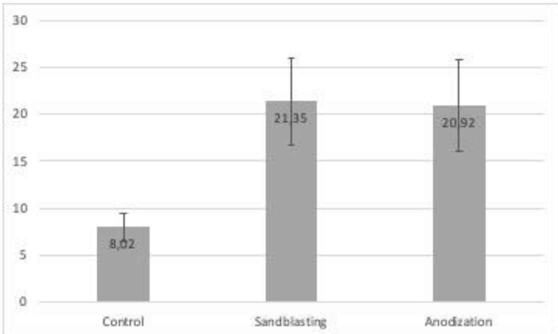

Figure 1. Shear bond strength and standard deviation.

An analysis of the failure mode, adhesive and cohesive failures were observed in both groups however combined failure mode was only observed in sandblasting group.

The scanning electron microscopic (SEM) images of untreated, sandblasted and anodized samples are presented in Figure 2. According to the SEM images, the anodization process appears to be more effective on the surface.

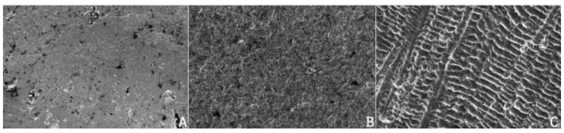

Figure 2. SEM images of the samples (A) non-treated surface. (B) air-particle abraded surface (C) anodized surface

DISCUSSION

This study aims to compare the effects of sandblasting which is the most common and scientifically proven method and anodization which has not been investigated on metal-ceramic bond strength. The results obtained from the tests suggested that the difference between sandblasting and anodization groups was not significantly different ($p>.05$). Therefore, the hypothesis which advocate the application anodization would not enhance metal ceramic bond strength, was not accepted.

This research was conducted on nickel-based alloy which rarely shows allergic reactions. This decision was made according to the research of Sipahi *et al.*[4] in which shear bond strength between different metals and different ceramics were investigated and Ni-Cr gave the highest bond strength value. Furthermore, supporting the Sipahi' s results, Akova *et al.*[16] investigated the bond strength of cast and laser sintered Ni-Cr and Co-Cr metals, they mentioned that the highest metal-ceramic bond strength was obtained with cast Ni-Cr but no significant different was detected between cast Co-Cr. Although different results were obtained in different studies,[17-18] Ni-Cr was preferred due to consistency of data in terms of standard deviation in Sipahi's research.[4]

The effect of airborne-particle abrasion on metal surface in order to achieve mechanical interlocking to obtain higher metal-ceramic bond strength was proven in many research.[19-20] The application of APA increases the surface energy and wettability of the metal substructures, which means the improvement of the adhesion between metal and ceramic.[21] While the benefits of sandblasting were mentioned in so many publications, the parameters preferred in each study were different.[3,19,22] In this research, the APA parameters were determined from our previous research in which all sandblasting parameters were tested and mentioned the most effective parameters on surface roughness was 110µm Al_2O_3 at 75 psi from a distance of 20 mm for 30 min.[10]

The platinum was used as an anode to cover the Ni-Cr because of the corrosion resistance and biocompatibility. Producing platinum framework by casting is difficult because of the high melting point and the need of argon atmosphere. However, by anodization process using the platinum it is aimed to get rid of allergic properties and corrosion tendencies of nickel-containing metals.

There is no publication on the investigation of anodization on metal-ceramic bond strength. The results evaluation of recent study with other studies which investigated the metal-ceramic bond strength is difficult, since different methods were used as surface treatments. Most studies were focused on the effect of sandblasting,[24] chemical etching[25] or acid etching[20,26] on metal-ceramic bond strength. Working mechanism of all these surface treatments to obtain surface roughness is removing the material on the surface of the applied sample. However, in anodization process roughness was obtained by adding (encapsulating) materials on the samples, not removing.

In literature, different bond strength values after the application of APA were obtained in different studies.[17,22,19] Such a result may be attributed to the use of different grain size of Al_2O_3 or the difference of the surface treatments' application time. Fonseca *et al.*[19] who evaluated the effect of different surface treatments on the bond strength between resin cement and a base metal alloy performed the APA procedure after polishing the surface of the specimens with 150, 400, 600 grid silicon carbide papers. However, Yurdanur *et al.*[27] applied the surface treatment after the casting process without any polishing procedure and then investigated the bond strength with the ceramic. However, the obtained results did not only indicate the effect of surface treatment on bond strength but also includes the effect of roughness of casting process. In this study, like Fonseca, the surface of the samples was polished in order to compare only the effects of the applied surface treatments.

The structural compatibility means a strong and durable bond between metal and ceramic. Initially, the chemical bond forms between oxide and metal which than concluded a chemical adherent oxide bond between ceramic and oxide layer. However, the overproduction of the oxide layer on metal surface forms poor metal-ceramic bond.[23] Adhesive failure modes suggesting the weaker bond strength at metal-opaque interface. In anodization group of this study, the vast majority of the specimens were failed in adhesive mode whereas in the sandblasting group, the great majority of the specimens were failed in mix and cohesive mode. Such a result can be interpreted to the weakness of the connection of Ni-Cr and platinum oxide layer after anodization.

CONCLUSION

Within the limitations of this study, it can be concluded that the anodization is a viable method for increasing the metal ceramic bond strength.

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