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Evaluation of Titanium Nitride (TiN) and Titanium Aluminum Nitride ((Ti,Al)N) Surface Coating on Bond Strength and Microleakage at Metal-Ceramic Interfaces

Seniha Baba[1], Şafak Külünk[2], Duygu Saraç[2] and Tolga Külünk[2]*

[1]Department of Prosthodontics, Oral and Dental Health Centre, Izmir, Turkey

[2]Department of Prosthodontics, Faculty of Dentistry, University of Ondokuz Mayıs, Samsun, Turkey

Abstract

Statement of Problem: Base metals are still widely used in dentistry. However, biological and aesthetic disadvantages related to base metal alloy supported restorations are reported. There are not enough studies about surface coating processes that improve the mechanical, aesthetic and biological properties of the surface they are applied to.

Purpose: The purpose of this study was to evaluate the effect of surface coatings of metal frameworks on early and long-term bond strength and microleakage at metal-ceramic interfaces.

Material and Methods: A total of 72 metal frameworks were abraded with 50-µm aluminum oxide (Al2O3) particles and randomly divided into three main groups (n=24). Group C: no coating was applied (Control); Group TN: specimens were coated by titanium nitride; Group TAN: specimens were coated by titanium aluminum nitride. Surface morphology was examined under a Scanning Electron Microscope (SEM). Veneering porcelain was applied to all metal frameworks. Half of the specimens in each group were subjected to an early bond strength test. The remaining specimens were subjected to early microleakage analysis, thermocycled for 6,000 cycles, and thereafter subjected to long-term microleakage analysis and a bond strength test, respectively. Data were statistically analyzed by a one-way Analysis of Variance (ANOVA), independent-samples t-test, and paired-samples t-test (α=0.05).

Results: The group TN showed significantly higher values in the early flexural bond strength test, but no significant difference (P=0.068) was found among the groups in the long-term test results. In early microleakage analysis, no significant difference (P=0.481) was found among the groups, whereas the group TN was significantly different (P<0.001) from other groups in the long-term analysis.

Conclusions: Both surface coating methods have shown no superior microleakage results compared with the control group in early and long-term analyses. Titanium Nitride (TiN) and Titanium Aluminum Nitride ((Ti,Al)N) coating of metal frameworks are not effective methods to increase porcelain bond strength.

Keywords: Metal ceramic restorations; Bond strength; Surface coating; Thermal aging; Microleakage

***Correspondence:**
*Şafak Külünk, Department of Prosthodontics, University of Ondokuz Mayıs Faculty of Dentistry, 55139, Atakum, Samsun, Turkey, Tel: 00903624576032;
E-mail: skulunk@omu.edu.tr*

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Clinical Implications

The TiN and (Ti,Al)N coating process with the magnetron sputtering technique has no effect on microleakage and bonding strength of metal supported porcelain restorations. Different results may be obtained with other types of dental alloys, different surface coating methods and coating materials.

Introduction

Dental porcelain, that has been used for years in construction of prosthetic restorations such as crowns, onlays, inlays and veneer crowns, is fragile and prone to premature failure under high stresses [1,2]. To increase the strength of the porcelain material, a common method is use of metal based frameworks, which meets mechanical and aesthetic requirements [3]. Noble metal alloys are preferred in construction of metal-ceramic restorations, due to their good thermal compatibility and the success of chemical bond. However, these alloys have a high cost of financing, and the need for precise technical processing leads to use of base-metal alloys in metal frameworks [4,5]. Base-metal alloys are economical alternatives to expensive gold alloys [6]. In dentistry, nickel-based and cobalt based alloys have exhibited an acceptable combination of strength, hardness, and in vivo wear resistance [5,7-9]. Moreover, these alloys allow fabricating thinner metal frameworks, because they have greater rigidity, which is related to the modulus of elasticity [10], and also provides low cost compared with noble alloys.

Despite their advantages, laboratory processes such as casting, machining and polishing of base metal alloys are challenging procedures. Moreover, the grayish color of the metal framework cannot be masked successfully, so the color of the restoration is affected negatively [11,12]. Several layers of dental porcelain are fired onto facial side of the metal substrate to mask the metal color and fabricate natural tooth-colored metal ceramic restoration. Furthermore, in some patients, the vanadium, nickel and cobalt elements of these alloys have been associated with sensitivity reactions [7,13-15].

To eliminate the negative properties of base metal alloys, the physical masking of metal surface using biocompatible barriers, or coatings, has been given considerable attention. The procedure involves using an intermediate layer deposited on the alloy surface prior to the application of the dental porcelain. Such a layer must be a biocompatible material and act as a barrier to the diffusion components, and be strongly adherent to the metal substrate. In dentistry, various surface coating methods including tin oxide, Titanium Aluminum Nitride ((Ti,Al)N), Titanium Nitride (TiN), Aluminum (Al), Aluminum Nitride (AlN), gold, silicon nitride, calcium phosphates, glass composites, bioactive phosphor silicate glasses, glass ceramics, and hydroxyapatite have been recommended [1,7,16-24]. Of these, several studies have focused on TiN and (Ti,Al)N coatings on alloy surfaces. These film layers improve the tribological properties of the surface to which they are applied, reduce the friction coefficient of the alloys, and increase the corrosion resistance, wear resistance and hardness [7,16,25]. Moreover, they have several biological advantages as a prosthetic material, including excellent biocompatibility, reduction of bacteria, and its suitability for use in patients who has a metal allergy to vanadium, nickel and cobalt. Although the surface coating method, used mostly in the engineering field, is not yet widely used as one of the alternative surface treatments, it is possible to use these methods in routine treatments because of their low cost and easy application [7,11,16,25,26].

The durability and strength of restorations is closely related to the quality of the adhesion between metal and ceramic, as well as mechanical properties of metal frameworks [3,27]. The adhesion mechanism between the metal and ceramic has not been completely defined, but it is believed to generally result from suitable oxidation of the metal and interdiffusion of ions to ceramic substrate [28-31]. Moreover, stress concentrations during ceramic cooling can result in ceramic chipping with either immediate or delayed response. Chipping and delaminating of veneer ceramics are critical problem for both base metal and noble metal alloys in fabricating metal ceramic restorations. The primary requirement for the success of a metal ceramic restoration is the development of reliable bond between the veneering ceramic and alloy [32-36].

Bond strength is determined by several factors: the strength of chemical bonds, mechanical interlocking, the type and concentration of defects at the interface, wetting properties, and the degree of compressive stress in the veneer layer due to differences in the coefficients of thermal expansion between the metal and veneering ceramic [5,37,38]. To increase the bond strength between the metal alloy and ceramic, various mechanical and chemical treatments are applied to the metal surface, such as air abrasion with aluminum oxide (Al_2O_3) particles in different sizes and/or treating with various acids. However, even with all these methods, there are still some connection failures between metal and ceramic.

One of the important criterions affecting the success of prosthetic treatments is microleakage [39], which is identified as the passage of oral fluids, bacteria, molecules or ions from oral environment [40]. This passage has two interfaces that may be between two different restorative materials or restorative material and tooth, and may lead to corrosion, cervical discoloration, and detachment of the veneer material [41]. Thermal aging is frequently used in evaluation of microleakage to simulate clinical conditions and thermal effects of oral environment as much as possible [42].

The purpose of this study was to evaluate the effect of two different surface coating methods (TiN and (Ti,Al)N)) on early and long-term flexural bond strength and microleakage at metal-ceramic interfaces. In the present study, two hypotheses were tested: H1: both surface coating methods would increase the early and long-term flexural bond strength; H2: both surface coating methods would decrease early and long-term microleakage values.

Materials and Methods

Metal framework preparation

A total of 72 wax patterns (sculpting wax FC; BEGO) were prepared using a polytetrafluoroethylene mold that have rectangular spaces with dimensions of 25 mm × 3 mm × 1 mm. Wax patterns were invested in a phosphate-bonded investment (Multi-Vest, Dentsply International). A nickel-based dental alloy (Wiron 99, BEGO) was used to casting procedure. The elemental composition of the alloy was as follows: Ni 65%, Cr 22.5%, Mo (Molybdenum) 9.5%, Nb (Niobium) 1%, Si (Silicon) 1%, Fe (Iron) 0.5%. The casting procedure was performed using an induction-casting machine (Fornax, BEGO) according to manufacturers' recommendations. The completed castings were divested, and the sprues were removed by air abrasion with 110 μm Aluminum Oxide (Al_2O_3) particles (Korox 110, BEGO), then all specimens were ultrasonically cleaned (Eurosonic Energy, Euronda) for 15 min in distilled water. Two sides of metal surfaces were finished by grinding respectively with a 400, 600, 1,200 grits of silicon carbide abrasive papers (3M ESPE) for 20 seconds at 300 rpm on a grinding machine (Buehler Metaserv) under running water, ultrasonically cleaned for 10 min in distilled water, and then air-dried. The thickness of metal specimens was controlled using a digital caliper (Digimatic Caliper, Mitutoyo). All specimens were air abraded with 50 μm Al_2O_3 particles, then ultrasonically cleaned for 15 min in distilled water.

Surface coating

Specimens were randomly divided into 3 main groups (n=24 in each group) for surface treatment procedures. First group served as a control and surfaces were left untreated. Other specimens in the second and third groups were coated with titanium nitride (TiN) and titanium aluminum nitride ((Ti,Al)N), respectively. The surface coating process was performed by physical vaporization (PVD) using a reactive magnetic field sputtering system. During the coating process, the radiofrequency power was fixed at 200 W,

Table 1: Firing procedures of opaque, dentin, and enamel porcelain.

Porcelain	Initial Temperature	Thermal Increase	Final Temperature	Wait Duration at Last Temperature
Opaque	500°C - 2 min	80°C	950°C	1 min
Dentin	500°C - 6 min	55°C	930°C	1 min
Enamel	500°C - 6 min	55°C	920°C	1 min

Table 2: Early and long-term flexural bond strength test results.

Groups	N	Test Results (MPa)	
		Early	Long-term
C	12	117.67 ± 23.50^{ab} A	116.70 ± 38.52^{a} A
TN	12	127.10 ± 24.50^{b} A	121.92 ± 23.32^{a} A
TAN	12	98.92 ± 16.80^{a} A	94.42 ± 24.40^{a} A

Superscript letters in a column and capital letters in a row show differences between groups. No significant differences were found between groups with the same letter.

and the working pressure was set at 10 mTorr. A 9 cm distance was left between the target and the lamp. The substrates came across the compound target once in every rotation. The deposition time was 120 min. The thickness of the coatings deposited on the substrates was measured by ball-cratering method. One specimen from each group was also examined under a scanning electron microscope (SEM) (Leo Stereoscan S440, Leica) at 20 kV. SEM photomicrographs were recorded at a magnification of X500 for visual inspection. The thickness of the TIN and (Ti, Al)N coating was also measured at a magnification of X20,000.

All metal specimens were cleaned for 1 min and air dried for 5 min prior to porcelain veneer application. Porcelain firings are presented in Table 1. The VMK 68 dental porcelain system (Vita VMK Master, VITA Zahnfabrik) was used to fabricate porcelain veneers. A thin and uniform layer of opaque porcelain, approximately 0.2 mm in thick, was applied to central 8.0 mm × 3.0 mm area of each specimen, using a standardized two-piece alignment jig to guide porcelain application, and then specimens were vacuum fired (Programat P90, Ivoclar Vivadent). Body and enamel porcelain application was standardized by the height of the alignment jig. Completed porcelain veneer buildups were vacuum fired according to the manufacturer's instructions. Specimens were stored in distilled water at 37°C for 24 h before the early flexural bond strength test. Half of each group (n=12) was separated for early flexural bond strength test. The other half of each group (n=12) was separated for early and long-term microleakage analyses and for long-term flexural bond strength test after thermal aging.

Flexural strength and microleakage testing

The early flexural bond strength between the metal-ceramic interfaces was evaluated by using a universal testing machine (Lloyd LRX, Lloyd Instruments) that has 3-point bending fixture. Each metal-ceramic specimen was positioned on the supports of the fixture by turning the porcelain veneer surface down. A compressive load was applied to the midpoint of the metal framework at a crosshead speed of 1mm/minute. The compressive load was sustained until a distortion in the load-deflection curve was noted, which indicates bond failure. The following formula was used to convert the data recorded in Newton (N) into megapascals (MPa): $\Sigma=3Pl/2bd^2$ (P: Maximum force (N), b: Specimen width (mm), d: Specimen thickness (mm) l: Distance between bases (mm), Σ=Flexural bond strength (MPa)).

The other halves of the specimens were firstly subjected to early microleakage analysis. The microleakage, between metal and porcelain after surface coating, was analyzed by gamma camera (Spect, Siemens, and Erlangen, Germany) using radioisotope method. The specimens were incubated for 24 h in 2% thallium solution (Monrol, Eczacıbaşi), then cleaned by brushing under running water. The radioisotopes attached to the metal-ceramic interface were imaged with a gamma camera and total counts were made. The counts were performed at the same pixel value for each specimen and the results were recorded.

Then, specimens were subjected to 6,000 cycles of aging at 5-55°C ± 2°C on a thermal cycler (Dentester Solubris Technica). Specimens were checked in every 500 cycles and aging continued without waiting. Immersion time for both baths was 30 s; the transfer time between the bathrooms was set at 10 s. The long-term microleakage analysis of the specimens after aging was carried out as described in the first analysis. Results were recorded, including total and average values. For long-term flexural bond strength evaluation, specimens were subjected 3-point bending test as previously described in early flexural bond strength evaluation, and the results were recorded in the same manner. The failure types observed at the metal-ceramic interfaces of each specimen tested for early and long-term evaluation were examined under light microscope (Leica MS5, Leica). Data were statistically analyzed by 1-way ANOVA (IBM SPSS Statistics v21.0; IBM Corp), Tukey HSD test, Tamhane T2 test, independent-samples T test, and paired-samples T tests. The significance level was set at.05.

Results

The mean flexural bond strength values and standard deviations of early and long-term flexural bond strength tests results are presented in Table 2. In early flexural bond strength test, 1-way ANOVA results showed a significant difference (df=35; F=5.17; P=0.011) among the groups. The highest mean early flexural bond strength value was obtained from the group TN. In multiple comparisons (Tukey HSD test), only significant difference (P=0.009) was found between the group TN and group TAN. In long-term flexural bond strength test, no significant difference (df=35; F=2.92; P=0.068) was found among the groups according to 1-way ANOVA test results. The highest mean long-term flexural bond strength value was obtained from the group TN. The significance of thermal aging in each group was analyzed using independent-samples T test, and no significant difference was found between the early and long-term flexural bond strength results in each group. Failure types observed at metal-ceramic interfaces are presented in Table 3. Adhesive failures were occurred between the metal and metal oxide layers, whereas cohesive failures occurred between the metal oxide and metal oxide layers (Figure 1). The mean microleakage values and standard deviations of early and long-term microleakage analyses results are presented in Table 4. In early microleakage analysis, 1-way ANOVA results showed no significant difference (df=35; F=0.75; P=0.481) among the groups. The highest mean early microleakage value was obtained from the group TN. In long-term microleakage analysis, a significant difference (df=35; F=9.72; P<0.001) was found among the groups according to 1-way ANOVA results. In multiple comparisons (Tamhane T2 test), no significant difference (P=0.199) was found between the group C and

Table 3: Type of bond failures observed at groups.

Groups	N	Early			Long-term		
		Adhesive	Cohesive	Mixed	Adhesive	Cohesive	Mixed
C	12	7	3	1	9	2	1
TN	12	5	7	-	4	8	-
TAN	12	4	8	-	4	7	-

Table 4: Early and long-term microleakage analyses results.

Groups	N	Test Results (x-ray counts)	
		Early	Long-term
C	12	38.7 ± 4.1^{a} A	9.4 ± 2.2^{a} B
TN	12	41.6 ± 9.9^{a} A	11.7 ± 2.5^{b} B
TAN	12	38.4 ± 5.7^{a} A	8.3 ± 0.7^{a} B

Superscript letters in a column and capital letters in a row show differences between groups. No significant differences were found between groups with the same letter.

TAN, whereas the group TN was significantly different from other groups. The significance of thermal aging in each group was analyzed using paired-samples T test, and there was a significant difference ($P<0.001$) between the early and long-term microleakage results in each group. In SEM analyses, alterations in the surface morphology were examined at a magnification of X500 (Figure 2). All specimens showed similar surface morphologies. The coating thickness was measured at a magnification of X20,000 (Figure 3). SEM analysis showed that the TiN coating had a thickness of approximately 1.55 µm, whereas (Ti, Al)N coating had a thickness of approximately 1.49 µm.

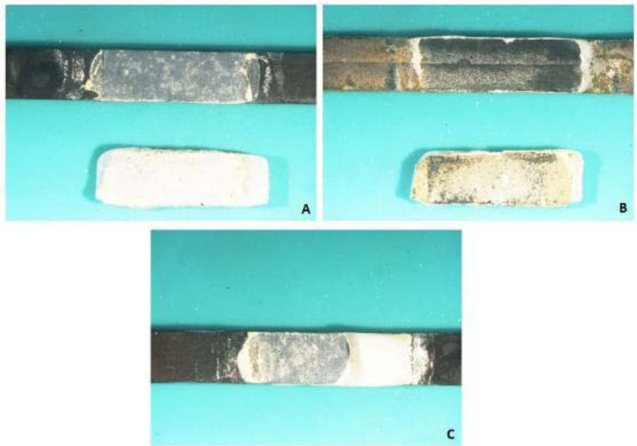

Figure 1: Failure types. A) Adhesive; B) Cohesive; C) Mixed.

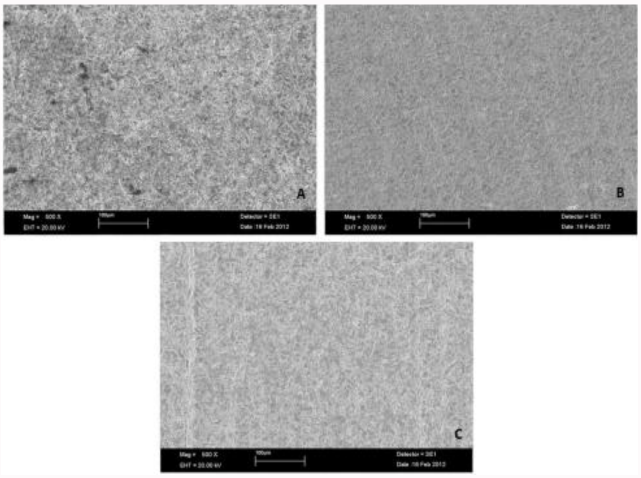

Figure 2: SEM microphotographs recorded at magnification of X500. A) Control group; B) TiN group; C) (TiAl) N group.

Discussion

In the direction of obtained results, it was observed that both coating methods have neither significantly increased the early and long-term flexural bonds strength and nor significantly decreased the early and long-term microleakage values. Therefore, both hypotheses were rejected. One of the most important criterions determining the long-term success of metal ceramic restorations is the formation of a reliable bond strength between the metal and porcelain [31,43-46]. This bond mechanism is primarily depending on the formation metal oxides, which acts as a bridge between the metal and opaque porcelain. However, an uncontrolled increase in thickness of this oxide layer may weaken the metal-ceramic bond strength. Nickel and cobalt based metal alloys are more likely to form thicker oxide layer compared with noble alloys [47]. It has been stated that air abrasion of the metal surface increases the surface area and reduces the surface tension, thus promotes the metal-ceramic bond strength. Moreover, air abrasion of metal surface provides micromechanical interlocking at metal-ceramic interfaces [48]. Lenz et al. [49] have reported that air abrasion of metal frameworks with Al_2O_3 particles show higher porcelain bond strength values compared with those polished specimens. Fischer et al. [50] have also reported similar results.

In addition to conventional surface treatment methods, alternative surface coating techniques have been suggested to increase the metal-ceramic bond strength. Tek et al. [25] have coated Ni-Cr alloy by TiN using PVD method and observed that coated specimens show higher wear resistance and hardness compared with uncoated ones. In another study, gold-palladium-silver (Au-Pd-Ag) alloys coated by TiN have been reported to increase the metal-resin bond strength and exhibit excellent biocompatibility as well as corrosion resistance. (Ti,Al)N material is also used for coating by PVD method. Liu et al. [11] have evaluated the mechanical behaviors and corrosion resistance of Ni-Cr and Au alloys coated by (Ti,Al)N and stated that (Ti,Al)N coating increases the wear and corrosion resistance. Chung et al. [7] have investigated the porcelain bond strength of Ni-Cr alloys coated by (Ti,Al)N and reported that surface coating provides a suitable oxide formation, and thus increases the metal-ceramic bond strength.

In the present study, no significant bond strength difference was found between the control and coating groups in early and long-term bond strength evaluation. According to ISO 9693-1 standard, bond strength values <25 MPa are not acceptable [51]. No specimen in the present study has showed a bond strength value lower than 25 MPa, and therefore all methods were found successful in terms of metal-ceramic bond strength. Moreover, similar surface morphologies were evaluated in SEM images recorded at magnification of X500. It is

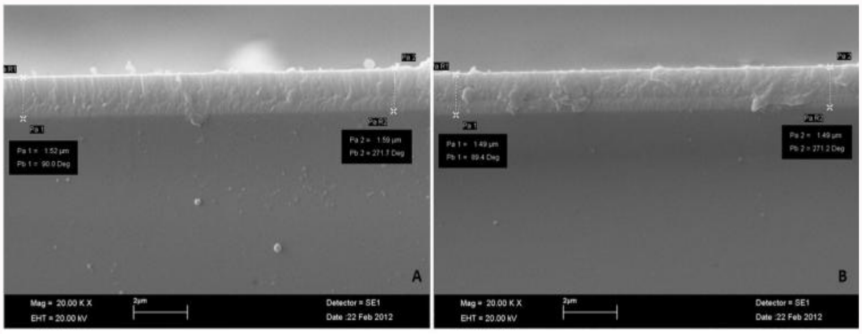

Figure 3: Measuring of coating thickness at magnification of X20,000. A) Specimen coated by TiN; B) Specimen coated by (TiAl) N.

thought that the coating by TiN may increase metal-ceramic bond strength in a small amount, but may not increase it to such an extent that a statistical difference will occur. Furthermore, the specimens coated by TiN have showed significantly higher bond strength values compared with the (Ti,Al)N group, however there was no significant bond strength difference between two coating groups after thermal aging. This may be explained by long-term microleakage results, which were significantly higher in the TiN group. In addition, adhesive failures between the metal and metal oxide occurred mostly in control group, while cohesive failures within the oxide layer occurred mostly in coated groups. It is thought that, coating the metal surface by TiN and (Ti,Al)N may be inadequate to control the thickness of oxide layer. To determine the clinical reliability of restorations, they must be subjected to tests under laboratory conditions. However, no specific test method is available to measure the metal-ceramic bond strength [52-55]. One of the most used test designs measuring metal-ceramic bond strength is shear test. On the other hand, this test design has been associated with unfavorable force distribution within the structure [53]. Based on ISO 9693-1 standard [53], 3-point bending test was used to measure the metal-ceramic bond strength in the present study. This test design provides several advantages including the possibility of comparing bond strength of materials with different elasticity moduli, and simulating the bending force which is a common stress observed at fixed partial restorations. Furthermore, various studies, which have used 3-point bending test to evaluate metal-ceramic bond strength, are available to compare the obtained results [3,31,56-59]. Early and long-term microleakage analyses revealed that a complete impermeability is not provided. No significant difference was found among the groups in early evaluation, however, the specimens coated by TiN showed significantly higher microleakage values in long-term evaluation. This may be associated with the surface characteristics of coating material, which is one of the limitations of the present study. Moreover, there are studies, which have observed increased microleakage values after thermal aging [60,61]. On the other hand, some studies have reported lower microleakage values after thermal aging, and attributed this result to that hygroscopic expansion resulting from water absorption decreases marginal discrepancy and thus microleakage [62,63]. Similar results have been obtained in the present study. The microleakage values in each group have significantly decreased after thermal aging. It is though that water absorption may cause the distance between the metal and ceramic to get narrow, and decrease the microleakage. In available literature, there was no study that evaluates the microleakage between metal and porcelain after coating process. In the present study, surface characteristics of coating materials were not evaluated. Different results may be obtained with other types of dental alloys.

Conclusions

Within the limitations of this study, the following conclusions were drawn; TiN and (Ti,Al)N coating of metal frameworks are not effective methods to increase porcelain bond strength. Thermal aging has not affected the metal-ceramic bond strength but has significantly decreased the microleakage at metal-ceramic interfaces.

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