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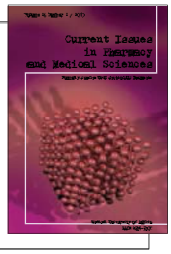

Effects of intermediate gold layer on bond strength of dental porcelain to titanium

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ABSTRACT

The demand for metal dental implants triggers search for innovative biomaterials, which are most importantly, characterised by good biotolerance, are corrosion resistant, possess high mechanical resistance and cosmetic durability. Up until today, it has been impossible to achieve a satisfactory level of high mechanical resistance of titanium prosthetic apparatus with permanent dental ceramic veneers and, for this reason, research in this respect still continues. The purpose of the research was to shape the surface layer of titanium biomaterials by creating a multilayer system comprising an inner gold layer and an outer dental porcelain layer so as to achieve a permanent bond of metal-ceramic. Commercially pure titanium samples (ASTM grade 2) subjected to conventional sandblasting (with 110 μm Al_2O_3 particles) uncoated and coated with gold, were examined. The metal–ceramic bond strength was investigated according to ISO 9693 standards using the three-point flexure bond test. An improvement in the adhesion of dental porcelain to titanium was obtained by coating the metal substrate with gold, which is of clinical importance.

Keywords: titanium, bond strength, dental prosthetics, porcelain, gold layer, sputtering

INTRODUCTION

Dental prosthetics, which is largely based on common metal alloys, more and more intensively seeks produce made of titanium and titanium alloys. This state of affairs raises the need for effective research in the area of material engineering regarding obtaining a permanent bond in the boundary between dental porcelain and the surface of metal prosthetic. Up until today, it has been impossible to achieve a satisfactory level of high mechanical resistance of titanium prosthetic apparatus with permanent dental ceramic veneers (porcelain) and, for this reason, research in this respect still continues.

Laboratory scale production of titanium prosthetic apparatus permanently coated with porcelain is a difficult process owing to differences regarding the type of chemical bonds in the two materials. The nature of chemical bonds changes abruptly at the metal-ceramic boundary, when the metal lattice turns into an ion-atomic one. During laboratory formation of metal prosthetic structures, the metal surface undergoes sand blasting and is veneered by porcelain [3,4]. The bond between the porcelain and a metal base is achieved thanks to an oxides layer, which is formed during the porcelain fusion process. This is when an intermediate layer between the ceramic porcelain and metal is formed. It is responsible for adhesion between porcelain and the metal base. However, this layer is not always formed in an even manner on the whole surface. Then the prosthetic structure has insufficient mechanical resistance and the porcelain may chip off [8]. Clinical tests prove 16% failure rate after only three-year usage [14].

Literature data [1,10] indicate that porcelain-titanium bonds are influenced by the following factors: during porcelain cutting titanium and its alloy undergo constant oxidisation, accumulated internal stress during oxides accrual prevents the oxide layer from shivering and the fact that internal stress develops in the area between particular phases due to ill-adjusted thermal expansion rates. Moreover, it has been concluded that the most beneficial conditions appear when the thermal expansion rate for porcelain is slightly lower that the rate for the metal. Admissible difference in the α rate should be approximately $0.5 \times 10\text{-}6/°C$. If the difference is significantly greater, it causes residual negative stress, which may lead to the metal-ceramic bond being damaged [10].

As for the titanium-ceramic bond, literature [2,9] indicates that it is not always possible to obtain a satisfactory

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level of adhesion between those two components in accordance with ISO 9693 norm standards. Porcelain surface damage (chipping) is a serious clinical problem as it produces a large aesthetic defect. It very often results in the need to replace the whole prosthetic filling, which increase the patient's discomfort.

At present, scientists continue to search for new methods, which could raise the quality of the metal-ceramic bond. Literature knows cases where the durability of titanium biomaterials was increased by means of digestion with acids, alkali and their salts, fusion of new types of low-melting porcelain in vacuum conditions or in argon atmosphere or through the application of intermediate oxide layers using the sol-gel method [3,4,8]. However, these methods neither proved fully successful, nor are widely applied in prosthetic workshops and are still being researched.

There is an interesting alternative, which involves using intermediate coatings with large content of gold. Bonds with large content of gold (so-called Gold Bonder) were used in Tholey's and other research [13] and lead to a seven-fold increase in porcelain adherence to the titanium base, compared to the sand blasting method which is currently used in prosthetic workshops. Suansuwan and Swain [12] obtained similar positive results of applying Gold Bonder in their resistance tests. Literature data [5,11] have indicated that beneficial mechanical properties of the titanium-ceramic system may be obtained by means of intermediate gold layers overlain with ion dusting.

The purpose of this study is to shape the surface layer of titanium biomaterials by creating a multilayer system comprising an inner gold layer and an outer dental porcelain layer so as to achieve a permanent bond.

MATERIALS AND METHODS

Commercially pure titanium (ASTM – grade II) was used as the metal substrate (Daido Steel Co. Ltd.). The samples had the figure of plates with dimensions 25 × 3 × 0.5 mm in agreement with requirements of norm PN-EN ISO 9693 [15]. The surface of all titanium samples were subjected to conventional sandblasting. Sandblasting was performed using Al_2O_3 particles of 110 µm at the pressure of 0.4 MPa (Korostar, Bego) and the distance between the surface and nozzle was 20 mm. Next, the specimens were rinsed with water vapor at the pressure of 0.4 MPa (Star, Reitel). Eight of the titanium samples were then coated with pure gold using a sputter coater (BS100 Unitra Unima, Poland) operating at 10 A for 10 seconds. The remaining 8 titanium samples were not coated.

Next, in the middle of the specimens (8×3×1 mm) Triceram (Dentaurum, Germany) low-fusing dental porcelain was fired. The dental porcelain was deposited in the following order: bonder, opaque (1), opaque (2), dentin, glaze according to the manufacturer's recommendations (Table 1). The parameters for porcelain firing were controlled automatically with a dental porcelain furnace (Programat X1, Ivoclar Vivadent).

Three-point bending test was performed on a Zwick Z100 universal testing machine equipped with a 500 N measuring head. The distance between the supports was 20 mm, and the diameter of the rollers supporting the sample was 2 mm. The load was applied at a crosshead speed of 1.5 mm/min until a disruption of the load deflection-curve occurred, which was the indication of bond failure. The bond strength (?b) was calculated using the equation [15]

$$\tau_b = k \cdot F_{fail} \quad (1)$$

where the coefficient k is a function of the thickness of the metal substrate and the value of Young's modulus. Then F_{fail} is fracture force causing the loss of joint of metal-porcelain.

The microstructure of samples after three-point bending test was examined using the scanning electron microscope Phenom G2 pro.

Table 1. Firing parameters of the Triceram low-fusing porcelain

	Base tempe-rature (°C)	Pre-drying time (min)	Heating rate (°C/min)	Vacuum on at (°C)	Vacuum off at (°C)	Final tempe-rature (°C)	Holding time (under vacuum) (min)
Bonder	600	2	65	600	795	795	1
Opaque 1	600	2	65	600	795	795	1
Opaque 2	600	2	65	600	795	795	1
Dentine	600	2	55	600	755	755	1
Glaze	600	2	55	–	–	755	1 without vacuum

RESEARCH METHODOLOGY

The results of the bond strength of metal substrate--intermediate layers - dental porcelain are listed in Table 2 and Fig. 1. Average results significantly exceed the minimal value determined in ISO 9693 (25MPa). In the case of Ti/Al_2O_3 abrasion/gold coating/Triceram porcelain system, 30% increase of bond strength has been achieved in comparison with Ti/Al_2O_3 abrasion/Triceram porcelain system without intermediate gold coatings. Data were analyzed using Mann – Whitney U Test to compare differences between the groups (level of significance p=0.05). The statistical analysis revealed the existence significant differences in the bond strength values between these groups (p=0.0086).

Table 2. The bond strength test results of metal-ceramic systems for two experimental groups (n=8)

Groups	Bond strength (MPa)	
	Mean	Standard Deviation
Ti/Al2O3 abrasion/ Triceram porcelain	27.51	7.23
Ti/Al2O3 abrasion/ gold coating/Triceram porcelain	35.78	4.29

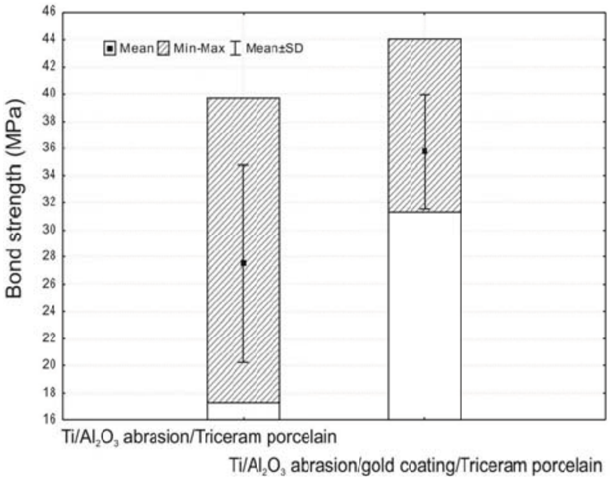

Fig. 1. The mean bond strength of metal-ceramic systems with different surface titanium modification

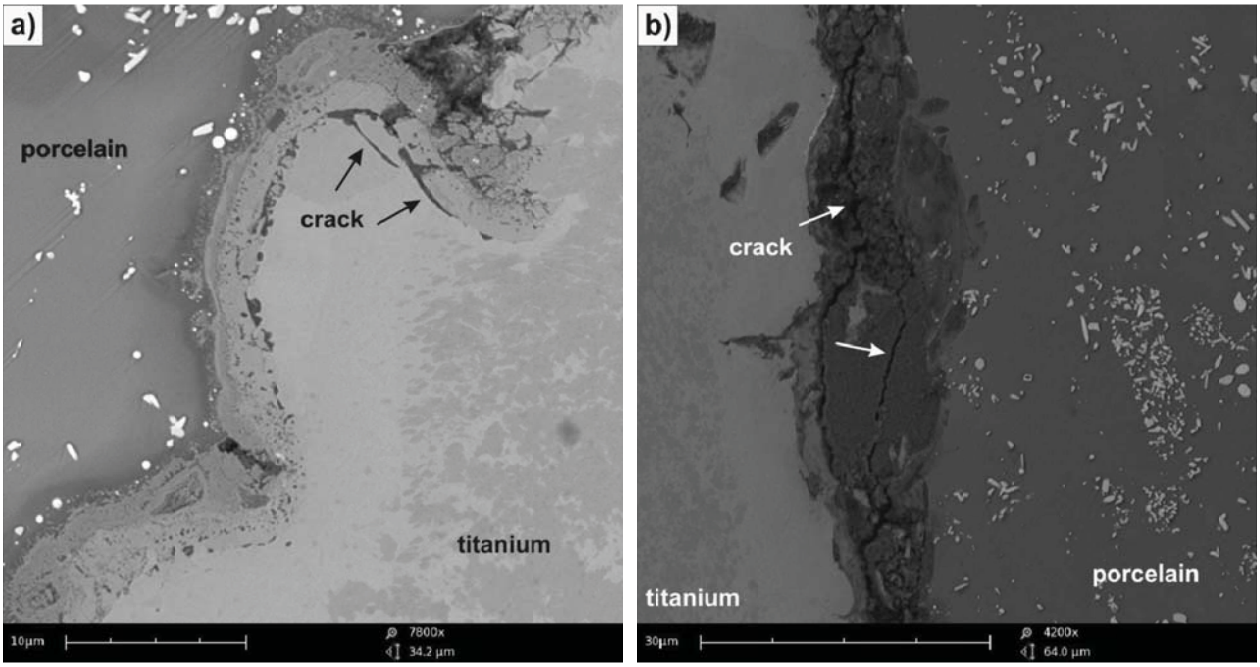

Fig. 2. SEM microphotograph of the cross-section of the metal-ceramic systems after debonding: (a) Ti/Al2O3 abrasion/Triceram porcelain and (b) Ti/Al2O3 abrasion/gold coating/Triceram

The SEM microphotographs of the cross-section of the materials examined after the bond test are presented in Fig. 2, where good bonding of porcelain to metal substrate with the intermediate gold layer is demonstrated. In the case of Fig 2a, the loss of bond (cracks) was observed on titanium oxide – porcelain interface. Whereas Fig. 2b presents a characteristic cracks through the porcelain area.

Fig. 3 shows fracture images of the representative specimens on the titanium side after three-point bending test. The image (Fig. 3 a,b) shows that the remains of porcelain adhere to the titanium fracture surface. The type of fracture for Ti/Al_2O_3 abrasion/ gold coating/Triceram porcelain group was cohesive-adhesive. However, for Ti/Al_2O_3 abrasion/ Triceram porcelain group only adhesive type of fracture was observed.

According to research conducted by Martinez [7], Ti diffuses inside the gold coating and constitutes an effective diffusion barrier for TiO_2 layers. Whereas, according to conclusions made by Lee [6], titanium ions, which diffuse inside the coating, do not create an effective barrier for the development of the oxide layer formed during porcelain fusion. A new Au_2Ti phase is formed. This provides ground for research in the area of modifying titanium-ceramic systems by means of an intermediate gold layer.

DISCUSSION

The application of intermediate gold coatings to titanium substrate, produced by sputtering technique, significantly improves the bond strength of metal-porcelain systems in comparison to the metal substrate after

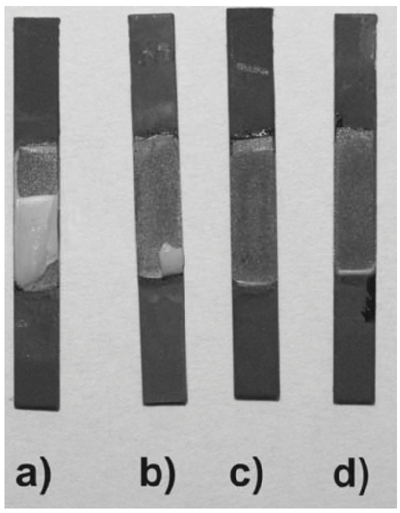

Fig. 3. Representative fracture images of the specimens on the titanium side after three-point bending test: (a, b) Ti/Al_2O_3 abrasion/gold coating/Triceram porcelain and (c, d) Ti/Al_2O_3 abrasion/Triceram porcelain.

sandblasting only. The failure in all systems was adhesive-cohesive, mainly adhesive, observed at the interface between titanium substrate and porcelain. The obtained metal – intermediate gold layer – porcelain system met the qualitative demands of dental implants (according to ISO 9693) enabling increasing their quality, durability and strength and may be used in the dental prosthetics.

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