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Application of the sol-gel coatings in dental prosthetics*

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The paper presents the study of intermediate sol-gel layers (SiO_2 , $\text{SiO}_2\text{-TiO}_2$) and dental porcelain coatings on titanium Ti (cp) and titanium alloy Ti6Al4VELI. The microstructural characteristics and wear tests by pin-on-disc method were investigated. The analysis revealed: (1) compact, homogenous Ti6Al4V/ SiO_2 and Ti/ $\text{SiO}_2\text{-TiO}_2$ coatings, and (2) a good bonding between coatings and metal and (3) that intermediate layers may provide durable joint between metal and porcelain and (4) that dental porcelain on SiO_2 and TiO_2 coatings shows high wear resistance.

1. INTRODUCTION

Titanium and its alloys are attractive materials for biomedical applications [1]. Their advantageous properties such as: corrosion resistance, low density, good mechanical properties and biocompatibility are a base for applications in dental prosthetics [2,3]. Nowadays, these are the most popular materials for production of implants, crowns and bridgeworks [3,4].

The latest studies show that between dental porcelain and metal, the intermediate layers are formed. These layers are responsible for adhesion of porcelain to metal base. Sometimes the bonding of porcelain with orthodontic metal construction possesses insufficient strength. As a result, the porcelain separates [5].

One of the new methods of producing intermediate coatings used in biomaterials is sol-gel process. These coatings are characterised by lower thickness, higher homogeneity and mechanical and chemical stability [5-10]. In the presented work silica or silica titania sol-gel coatings were used for the creation of the new metal surface and new interface joints between commercially pure titanium or titanium alloy and low melting dental porcelain.

2. EXPERIMENTAL PROCEDURE

Commercially pure titanium (ASTM-grade 2) and Ti6Al4VELI alloy (ASTM-grade 5) were used. Ti (cp) was forged and annealed. Titanium alloy was hot-rolled and solution treated. Table 1 shows the chemical compositions of these materials.

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Table 1
The chemical compositions of Ti (cp) and Ti6Al4V alloy (wt.%)

	Fe max	O max	N max	C max	H max	Al	V	Ti
Ti (cp)	0.3	0.25	0,03	0.08	0.015	—	—	bal.
Ti6Al4VELI	0.25	0.13	0.05	0.08	0.015	5.5-6.75	3.5-4.5	bal.

SiO₂ on Ti6Al4V alloy and SiO₂-TiO₂ on Ti (cp) coatings were deposited using sol-gel method. Silica sol was prepared by hydrolysis of tetraethoxysilane (Si(OC₂H₅)₄; abbreviated as TEOS) diluted in ethanol with addition of HCl as a catalyst. Titania-silica sol was prepared by hydrolysis of titanium propoxide Ti(OC₃H₇)₄ and TEOS with addition of HCl as a catalyst. The deposition of layers consisted of withdrawing the metal discs from sol solution with constant speed 20 cm/min. Thickness of the deposit was controlled by multiple dipping. After the completion of deposition, the as-deposited coatings were carefully dried and annealed in an argon atmosphere. The heating removed water and residual organic substances, densified the layer and increased its extent of bonding with the substrate. The Vita Omega dental porcelain was deposited on intermediate layers.

The wear resistance of the dental ceramics was determined using a pin-on-disc method. A load (normal force) of 0.78 N was applied on the ball (0.5 mm diameter WC-6%Co). The ball was rubbing against the sample fixed on a rotating disc. The samples had shape disc about diameter 25 mm and thickness 0.8 mm. The sliding speeds between the rubbing surface was from 53 mm/s. The test were performed for 16000 cycles. The samples wear subjected to day sliding wear test in the room temperature (about 25°C) and 27% atmospheric humidity. The wear test were evaluated from the cross-sectional profiles of the wear tracks measured by means of a Taylor Hobson Profilometer.

The microstructure of coatings and wear after pin-on-disc test were studied using a scanning electron microscope LEO 1430VP with EDX-Roentec.

3. RESULTS AND DISCUSSION

Figure 1 presents the microstructures of Ti/SiO₂-TiO₂ and Ti6Al4V/SiO₂ coatings. The compact, homogenous microstructure of these coatings without structural discontinuity was observed. In both cases the joint between coatings and metal is sufficient. The thickness of ceramic layers is about 3 μm. The Ti/SiO₂-TiO₂ coating consists of a mixture of SiO₂ and TiO₂ formed during the synthesis of coating and its heat treatment. In the microstructure of Ti6Al4V/SiO₂ coating numerous cracks were observed. These cracks are a result of the influence of high temperature of soaking during the sol-gel process [7,11]

The EDX analysis confirms that Si, O and Ti are present in the coatings. According to literature data [7,12] TiO₂ is crystalline whereas SiO₂ is amorphous glassy phase.

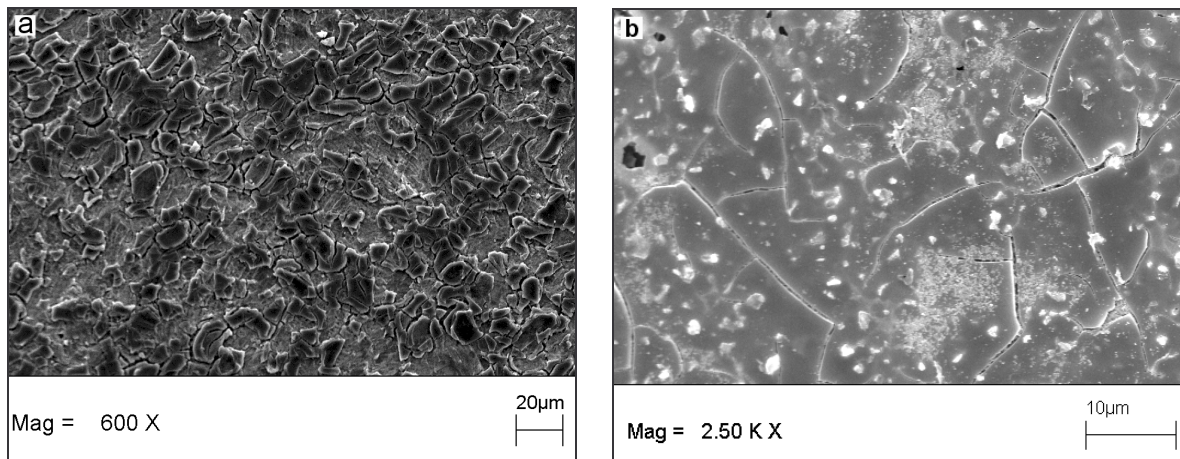


Figure 1. SEM microphotographs of surfaces of different coatings: (a) Ti/SiO₂-TiO₂ and (b) Ti6Al4V/SiO₂.

The microstructure of porcelain coatings with intermediate layers is shown in Fig. 2a. Quite good bonding between porcelain and titanium was observed. The roughness of SiO₂ and SiO₂-TiO₂ averages $R_a = 0.63$ and $R_a = 0.82$ µm may contribute to the improvement of adhesion. In addition, the cracks in the Ti6Al4V/SiO₂ surface also provide the increase of mechanical interlocking between intermediate titanium layers and porcelain [2].

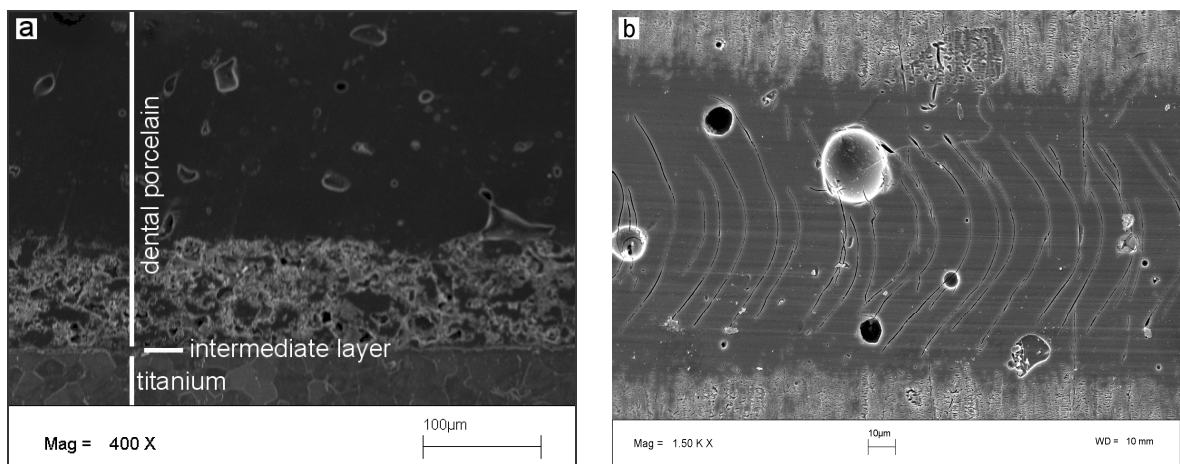


Figure 2. SEM microphotographs: (a) the microstructure of dental porcelain with intermediate layer and (b) worn surfaces of dental porcelain after 16000 cycles of the test.

The pin-on-disc is one of the methods to study wear tests of ceramic coatings [13-15]. A measure of wear of coatings is the transverse field section of wiping trace on the sample [16,17]. The pin-on-disc tests confirmed that wear was ranging between 151.48 and 175.28 µm². The wear of sol-gel porcelain layers didn't take place. The analysis of wear track (Fig. 2b) showed characteristic cracks. The cracks aren't deep and they don't influence adhesion of dental porcelain. Preliminary studies showed good adhesion of ceramic sol-gel layers but there is still a great need for further investigation.

4. SUMMARY

Intermediate layers Ti/SiO₂-TiO₂ and Ti₆Al₄V/SiO₂ deposited by sol-gel method are characterised by low thickness and high structural homogeneity. From the first studies of porcelain coatings it results that the intermediate layers may provide durable joint between metal and porcelain. To sum up, it may be concluded that dental porcelain on SiO₂ and TiO₂ coatings shows high wear resistance. The coatings produced by sol-gel method will find a wide range of applications in stomatology.

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