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The shear strength of three-dimensional capillary-porous titanium coatings for intraosseous implants

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ABSTRACT

A plasma spraying process for the deposition of three-dimensional capillary-porous titanium coatings using a wire has been developed. In this process, two additional dc arcs are discharged between plasmatron and both the wire and the substrate, resulting in additional activation of the substrate and the particles, particularly by increasing their temperature. The shear strength of the titanium coating with 46% porosity is 120.6 MPa. A new procedure for estimating the shear strength of porous coatings has been developed.

1. Introduction

The design of orthopaedic prostheses requires the solution of several complex materials science and engineering problems. High cyclic loads necessitate the application of a metallic base, namely, titanium, that is biologically inactive. It is highly difficult to combine the operation of titanium structure and bone tissue because of the tenfold difference between their elasticity moduli and the absence of strong chemical bonding [1]. In this case, under mechanical loading, deformations with different magnitudes arise at the interphase boundary, leading to the mutual wear of titanium and bone. The use of porous coatings with intermediate values of elasticity modulus decreases the stress concentration at the implant–bone interface [2–5]. The coating porosity increases the interface area and therefore increases the load transmitted from the implant to the bone tissue. Additionally, a hydroxyapatite (HA) layer applied on the surface of a porous titanium coating increases the transmitted load at the expense of the higher HA-bone tissue joining strength of [4,5]. Thus, it is necessary to prepare a bulk composite porous stable interface with a biologically active free surface, for example, by plasma spraying. Coating pore sizes of 300 to 600 μm are recommended (based on biological studies) for the successful growth of the bone tissue and its further operation [6]. We consider the coating–bone interface as the interface in the composite material. The strength of the bone-coating joint is estimated based on its shear strength and taking into account the volume fractions of the joint components. In this case, a method is required for the deposition of porous coating that allows us to control the porosity, pore sizes, coating strength, and adhesion [7].

It can be assumed that traditional powder metallurgy methods for the preparation of porous volume materials, namely, the sintering of a spherical powder were used in the initial development of the methodology for the preparation of porous coatings for intraosseous implants. Plasma coatings with the porous structure were prepared using titanium powders with a particle size of 200 μm, an intermediate powder spraying rate and a relatively low plasmatron power that give rise to the low degree of deformation of the particles deposited on the substrate [2]. The porous structure of the coating consists of weakly deformed particles and exhibits a relatively low strength because of point contacts between the particles (the shear strength of coating with 50% porosity is 18 MPa) and a specific porous structure characterized by expanding and narrowing pores [2]. Improvement of the mechanical properties of coating by titanium wire spraying was realized in [5]; in this case, the deposited particles were in the molten state [5]. However, the porosity of such a titanium coating is 4.8%, whereas the porosity of HA layer on the surface of titanium coating is 5.0% [5].

We have developed an original concept for the organization of volume implant–bone tissue interface through the formation of three-dimensional capillary-porous (3D CP) titanium coatings consisting of ridges and cavities [8–10]. In such a coating, dense (ridges) and porous (cavities) volumes are separated through a new mechanism. The formation mechanism of 3D CP titanium coatings is realized through the existing "shadow effect" [7]. The shadow zone exists behind the solidified particles and is not available for subsequent deposition of the off-angle spray particles. Shadow zones subsequently form cavities. The shadow zone width increases with decreasing spray angle. These

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coatings can be optimized for structure, porosity, and strength. To improve the implant–bone biocompatibility, the 100-μm HA layer is also applied on the surface of the 3D CP titanium coating [8–10]. In this case, coating cavities form the basic volume of pore space, up to 50%, in which a new bone tissue can grow freely and operate. Ridges of the 3D CP titanium coating transmit the main mechanical load between the implants and bone tissue. Owing to this effect, the strength of the formed ridges is comparable with that of monolithic materials. In the present study, we analyse the effect of two additional arc discharges between the plasmatron and both the wire and the substrate on the shear strength of 3D CP titanium coatings.

2. Methodology and materials

Porous titanium coatings with a thickness of 1 mm were deposited using a UPU-3d universal plasma equipment with dc arc plasmatron and additionally supplied with a chamber for spraying in an argon atmosphere. We used VT1/00 titanium containing 0.15% Fe, 0.05% C, 0.08% Si, 0.04% Ni, 0.1% O, and 0.008% H as the substrate for the coating deposition. A substrate in the form of a rod with a diameter and length of 9 and 100 mm, respectively, rotates at a rate of 160 rpm and moves in reciprocative manner at a rate of 0.1 m/s. Abrasive SiC powder with an average particle size of 700 μm was used to clean the substrates and to roughen them. The following operational plasmatron conditions were used for the deposition. The current of main arc is 300 A and the voltage is 30 V; the argon flow rate is 40 l/min. The coating was formed by spraying a VT1/00 titanium wire with a diameter of 1.2 mm; under these operational conditions, the sprayed wire is consumed at the rate of 0.8 mm/min. The use of an additional arc, energized (at the 120-A current and 50-V voltage) between the plasmatron and sprayed wire, increases the wire consumption to 8 m/min. The third arc discharge (at the 120-A current and 50-V voltage) was initiated between the plasmatron and titanium substrate. The third arc discharge was applied for 6, 9, 12, and 15 s. The reference sample was prepared in the absence of the arc discharge. The spray angle is 30°. The average size of sprayed particles was 250 μm, as determined using an NMZ 100H50A instrumental microscope equipped with an electronic system for measuring linear sizes of objects with an accuracy of 0.5 μm. We studied the dependence of the 3D CP titanium coating shear strength fabricated using the arc discharge between the plasmatron and the substrate on the time of substrate activation (Table 1). Parameters of the three applied arc discharges were optimized at the preliminary stage of the study taking into account the technical characteristics of the plasmatron (its power). To plot the ridge- and cavity-width distributions (for three hundred ridges and cavities), the ridge and cavity widths of a 3D CP titanium coating were measured by the secant method using an MBS-10 binocular microscope and a magnification of 100×.

The structural peculiarities of the 3D CP titanium coatings consisting of ridges and cavities necessitate the application of a new technique for estimating their shear strength. When shearing tested according to the traditional method of 3D CP coating ridges are destroyed sequentially; therefore, the traditional procedure for measuring the shear strength cannot be used for such coatings. In choosing the alternative testing procedure, we assumed that the maximum shear strength is reached after the completion of bone tissue growth in the porous space of the 3D CP coating. Such a state of the test sample can be simulated by filling the porous space (cavities) with a substance with mechanical properties that are close to those of bone tissue. Based on preliminary studies, we choose the MultiFast (Struers) bakelite resin (plastics) that is used for hot pressing of metallographic sections and exhibits the shear strength of 96.1 MPa. Test samples with the length of 17 mm were prepared by turning of the substrates with the applied 3D CP titanium coatings (Fig. 1).

The coating retained on the samples was 3 mm long. The prepared sample was mounted into a disk that prevents the bottom of the sample from the ingress of plastics. The sample mounted in the disk was placed into a mould with a diameter of 25 mm. The space around the coating was filled with bakelite resin powder such that after pressing, the height of the plastics is greater than that of the coating. Prior to the shear test, the excess plastic was removed to obtain the height equal to that of the coating. The width of the test coating is 3 mm. The high shear strength makes it impossible to realize the simple shear along the coating–substrate interface for a wider coating. The shear test of the coating was performed by exerting a pressure of the matrix and the punch on the coating (Fig. 2). The outer punch diameter is smaller than that of the coating. In this case, the shearing load is applied along the coating–substrate interface.

3. Results

3D CP titanium coatings consisting of ridges and cavities have been deposited (Fig. 3). The cavity and ridge widths vary from 0.1 to 2 mm. To plot the cavity- and ridge-width distributions, 0.3-mm width ranges (0–0.3, 0.3–0.6, 0.6–0.9, 0.9–1.2, 1.2–1.5, 1.5–1.8 mm) were used (Fig. 4). The average cavity and ridge widths are 0.55 and 0.65 mm, respectively.

Because we assume that the main pore volumes originate from the cavities, we calculate the porosity P (%) of the studied 3D CP titanium coatings as the ratio of the average cavity width to the sum of average

Table 1
Dimensions of coating and its shear strength.

Time of substrate activation (s)	Coating						
	Sample no.	Coating thickness (mm)		Coating area (mm^2)	Load, (kg)	Strength (MPa)	
		Actual	Average[a]			Actual	Average
0	1	0.84	0.94	76.65	450	57.6	71.2
	2	1.11		91.20	715	76.9	
	3	0.84		87.17	710	79.9	
	4	0.99		88.97	640	70.6	
6	5	0.76	0.92	91.99	2110	224.9	115.8
	6	0.74		74.36	400	52.8	
	7	1.25		91.33	650	69.8	
9	8	0.86	0.97	91.43	780	83.7	68.1
	9	0.70		84.92	310	35.8	
	10	1.25		88.88	680	75.0	
	11	1.06		94.26	750	78.0	
12	12	0.83	0.82	90.48	2150	233.0	120.6
	13	0.64		88.12	910	101.3	
	14	1.03		89.91	960	104.7	
	15	0.79		90.38	400	43.4	

[a] Note. Coating thickness averaged over all samples is 0.91 mm.

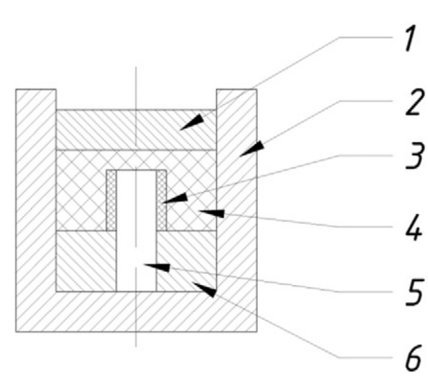

Fig. 1. Sample assembly for shear test: *1* punch; *2* mould; *3* coating; *4* plastics; *5* sample; and *6* limiting disk.