

Research Article

Development of Osseointegrative Ceramic Coatings Based on ZPTA—Mechanical Characterization and Influence on the Substrate

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Abstract In this article, the mechanical characterization of a monolithic cup made of BIOLOX[®]delta with a porous ceramic coating for osseointegration is presented. During the coating process, a ceramic slurry is deposited on a green substrate, both slurry and substrate are based on BIOLOX[®]delta. To achieve a porous coating, organic pore-forming agents were used which are pyrolyzed in the following sintering process. Porosities of 18 to 47% could be reached depending on the layer thickness. Extensive mechanical investigations were applied on the fabricated cups to verify the suitability of the produced porous layers for biomechanical demands of the interface between bone and dense substrate. The test results showed that coated ceramic cups require comparable push out forces for the disconnection from artificial bone to established metal shells with plasma sprayed titanium coatings.

Keywords porous ceramic coating; Zirconia Platelet Toughened Alumina; osteo-integration; push-out test

1 Introduction

Three different material groups are commonly used in joint replacements today: polyethylene, metals and ceramics [14]. The supposed aseptic loosening caused by polyethylene wear debris [10] and metal ion release by alloys [7, 11] are reasons for the application of ceramics in joint replacements. Ceramic-ceramic hard couplings are showing low wear rates [6] and high bioinertness due to the inorganic character and high bonding forces in the material [9].

Nowadays metal shells with osseointegrative coatings are necessary for a cementless fixation of ceramic inserts in the acetabular region of the human pelvis. These modular systems require relatively large space due to their wall thickness. One possibility to combine the excellent wear behavior of ceramics and a direct implantation of the ceramic cups without metal back is a structured, ideally osseointegrative ceramic coating. Such coatings would also be attractive for knee and spine joint replacements. Different

strategies were developed to produce osseointegrative surfaces and bulk materials. For example, foaming of ZTA slurries with egg white protein leads to interconnective porosity in ceramic blocks after sintering [8]. Another study describes the compositions of an alumina matrix with embedded hydroxy apatite (HA), whereby HA is the bioactive component [13]. Anodic deposition is another route to produce nano porous alumina on dense substrates [12]. Due to the fabrication processes, mechanical properties of porous materials are directly linked to pore size and pore distribution of coatings or bulk materials.

A direct implantation of ceramic components without metal backing leads to high requirements on monolithic ceramic components. Dense bulk materials have to absorb the largest amount of forces during use. Coatings as the porous component, on one hand, have to be stable against shear and pressure loads and, on the other hand, have to allow bone ingrowth. Excellent mechanical properties are required for direct integrated load bearing applications. Zirconia platelet toughened alumina (ZTPA) is a ceramic material group combining the low wear rate of alumina with the high strength and fracture resistance caused by transformation toughening of zirconia and, additionally, crack deflection by platelets. In comparison to medical grade high purity alumina, ZPTA shows a two times higher bending strength and crack resistance [2]. In addition, impact tests have shown that porous coated ZPTA based cups are more stable than established uncoated ceramic cup-inserts (high purity Al₂O₃) with metal backing [5]. To combine the good tribological behavior of a ZPTA-ceramic and the possibility of direct fixation in the bone, a porous ceramic layer on a dense thin walled ceramic substrate was developed [4].

The fabrication of structured porous layers based on ZPTA, the mechanical influences on the substrate, the biomechanical behavior of coated cups for hip joint replacements and the biological response on the structured coatings are main interests of the current project.

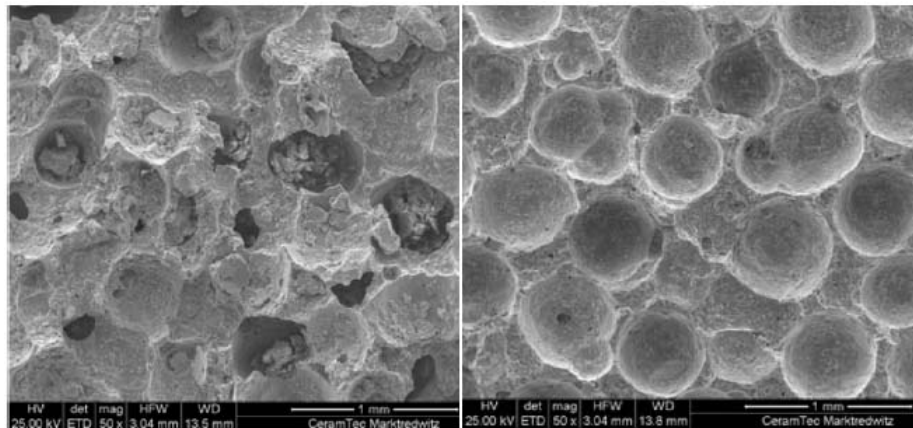


Figure 1: As-fired coating (left); finished coating surface (right).

2 Material and methods

Axially pressed and green-machined bodies were coated with a ceramic slurry in a spraying process. The green body (substrate) and the slurry are based on the above-mentioned ZPTA. Nearly spherical organic pore forming agents were embedded in the low-viscous slurry layer. Already 10 seconds after integration of the pore forming agents, the samples could be handled without damaging the coating. All deposition steps were performed in an automated process that takes into account the surface and geometry of different samples to ensure identical layer characteristics. The suitability of pore size and pore distribution was investigated in a cell study [1].

In a first sintering process, the green body is transferred to a solid ceramic part. Hot isostatic pressing eliminates porous defects in the dense ceramic substrate. A finishing process removes unstable parts from the surface (Figure 1). The interface between the coating and the substrate shows a homogenous connection (Figure 2).

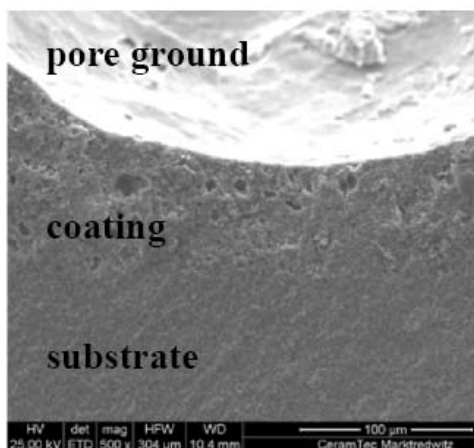


Figure 2: Cross-section of the coating.

The formed pores are a result of the size distribution of the pore forming agents and the shrinkage of the ceramic body. A wide range of pore diameters and distributions were investigated in biological studies to verify the optimal parameters for bone ingrowth [1]. According to this study, the applied range of pore forming agents from 520 to 660 μm diameter leads to main pore sizes from 200 to 500 μm diameter (Figure 3). The topography of the coating was determined by using Laser Scanning Microscopy (LSM), SEM and Light-optical Microscopy (LM) for calculations of the pore quantity on the surface (Figures 3 and 4). LM and LSM measurements show an average height of 0.3 to 0.4 mm and a surface porosity of 47%.

Investigations of the coatings influence on the mechanical properties of the substrate were done by 4-point bending tests in accordance with EN 643-1 and measurements of the torque strength (using cylindrical test specimens). The coating was loaded with hardened steel bolts (5 mm in diameter) to evaluate its pressure strength. Resin embedded coated ceramic cups were loaded by ball heads with different angles (90° and 45°). The primary stability of coated cups was tested by measuring push out forces to disconnect structured ceramic cups from artificial bone. The results were compared with those of established metal cups [5]. For this, polyurethane foam blocks were used (30 PCF, Sawbones, Sweden). Two different press-fits (-2 mm and -1 mm) and oversized cavities of 0 mm and $+1\text{ mm}$ as worst case scenarios were tested. The frontal faces of the cups were loaded with repetitive impacts by steel balls to provoke chip-off [5].

3 Results and discussion

Coated and hard-machined test pieces reach values of 50 to 75% of the four-point bending strength of uncoated as fired reference samples (884 MPa, $n = 30$). Micro pores creating high local stresses in the interface during testing are most likely causing the reduction of bending strength.

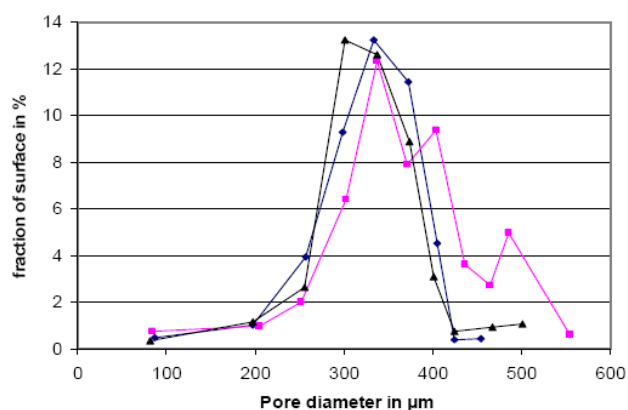


Figure 3: Pore size distribution on the coatings surface (measurements of three different samples).

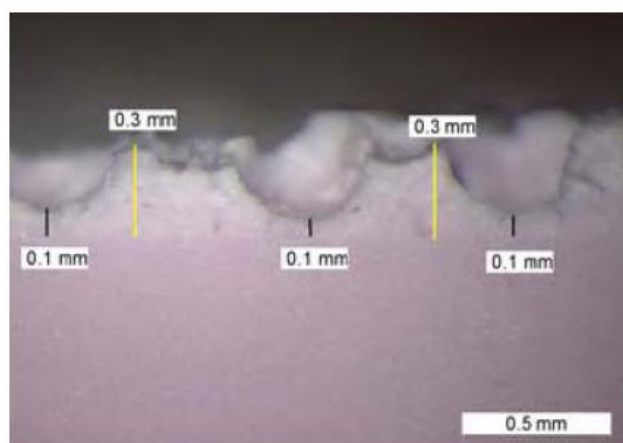


Figure 4: Macroscopic cross-section of the coating.

The average torque strength is in the range of 75 to 85% compared with uncoated reference samples (79 Nm). Coated cups survived axial loadings of 200 kN by compatible ball heads (equivalent ~ 20 tons). The seven axial tested cups failed at an average force of 112 kN under a loading angle of 45° (standard deviation 25 kN). The acceptance criteria for the burst strength of regular ceramic cups with uncoated surfaces is 46 kN. A pressure force of 19 kN was applied on the coating by using a 5 mm diameter steel bolt. The samples show undamaged coating areas after testing (Figure 5) whereas the steel bolts have been deformed during testing.

Coated ceramic cups require comparable push out forces for the disconnection from artificial bone to established metal shells with plasma sprayed titanium coatings (Plasmacup, Aesculap Tuttlingen) (Figure 6). Twenty-five impacts with a maximum energy of 20 Joule per impact were performed without damaging the coated ceramic cups. Lower impact energies and cycle numbers lead to chip-off on high purity alumina cups in metal shells (Figure 7) [5].

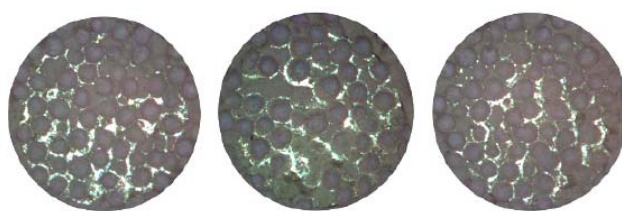


Figure 5: Pressure test samples with marks of the hardened steel bolt after 19 kN axial loading.

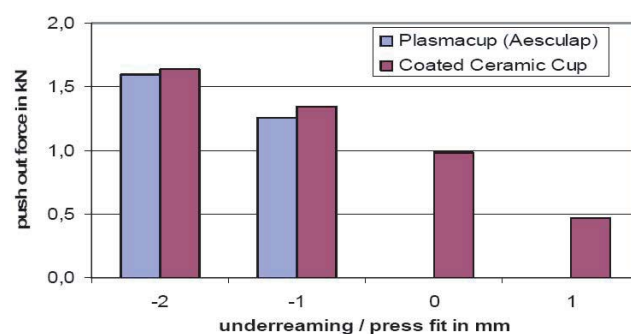


Figure 6: Push out forces of ceramic prototype cups and established metal cups in Sawbone 30 PCF.



Figure 7: Setup for impact testing.

4 Conclusions

A decrease in mechanical properties of coated ceramic cups could not be observed, whereas the bending and torsion strength of idealized test samples was reduced due to micro pores at the substrate-coating interface. Different test setups were applied to simulate severe conditions during implantation and live time of coated ceramic cups. The primary stability is comparable to established metal shells with plasma sprayed titanium coatings. The face area of coated cups can withstand impacts comparable to heavy traffic accidents without damages [5].

First histological and mechanical results of a current animal study with porous coated cylinders have exhibited the in vivo suitability of the coating. The bone structure

is directly attached to the porous surface which leads to higher push-out forces compared to unstructured test specimens [3].

The gathered mechanical, biomechanical and histological results proof the high potential of ZPTA-based porous ceramic coating on dense substrates for high load bearing applications. Optimizing the deposition process to reduce micro pores on the substrate-coating interface and the prototype design for following animal studies are steps in the near future.

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