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Original article

Influence of thermo-mechanical cycling on porcelain bonding to cobalt–chromium and titanium dental alloys fabricated by casting, milling, and selective laser melting

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ABSTRACT

Purpose: The aim has been to determine the effect of thermo-mechanical cycling on shear-bond-strength (SBS) of dental porcelain to Co–Cr and Ti-based alloys fabricated by casting, computer-numerical-controlled milling, and selective-laser-melting (SLM).

Methods: Seven groups ($n = 22/\text{group}$) of metal cylinders were fabricated by casting (Co–Cr and commercially pure-cpTi), milling (Co–Cr, cpTi, Ti-6Al-4V) or by SLM (Co–Cr and Ti-6Al-4V) and abraded with airborne-particles. The average surface roughness (R_a) was determined for each group. Dental porcelain was applied and each metal–ceramic combination was divided into two subgroups – stored in deionized water (24-h, 37 °C), or subjected to both thermal (6000-cycles, between 5 and 60 °C) and mechanical cycling (10^5 -cycles, 60 N-load). SBS test-values and failure modes were recorded. Metal–ceramic interfaces were analyzed with a focused-ion-beam/scanning-electron-microscope (FIB/SEM) and energy-dispersive-spectroscopy (EDS). The elastic properties of the respective metal and ceramic materials were evaluated by instrumented-indentation-testing. The oxide thickness on intact Ti-based substrates was measured with Auger-electron-spectroscopy (AES). Data were analyzed using ANOVA, Tukey's HSD and t -tests ($\alpha = 0.05$).

Results: The SBS-means differed according to the metal–ceramic combination ($p < 0.0005$) and to the fatigue conditions ($p < 0.0005$). The failure modes and interface analyses suggest better porcelain adherence to Co–Cr than to Ti-based alloys. Values of R_a were dependent on the metal substrate ($p < 0.0005$). Ti-based substrates were not covered with thick oxide layers following digital fabrication. **Conclusions:** Ti-based alloys are more susceptible than Co–Cr to reduction of porcelain bond strength following thermo-mechanical cycling. The porcelain bond strength to Ti-based alloys is affected by the applied metal processing technology.

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1. Introduction

Porcelain-fused-to-metal (PFM) prostheses have been subject over the years to variety of modifications, mainly regarding the materials used and the processing techniques applied. Metal frameworks in PFM prostheses have, traditionally, been fabricated by lost-wax casting. Although the casting technique still prevails in metal processing, its execution is often very complex due to the high melting range of the various metals and their susceptibility to

oxidation at high temperatures [1]. The shortcomings of this technology often result in porous and incomplete castings. Computer-aided design/computer-aided manufacturing (CAD/CAM) has enabled implementation of advanced routes for metal processing as an alternative to the traditional lost-wax casting, improving the fabrication time-efficiency and the precision of the frameworks [2,3]. CAD/CAM is readily used for both designing the prosthetic framework and executing its fabrication, by either subtractive or additive CAM processes. The subtractive method uses the concept of computer numerical controlled (CNC) milling from solid prefabricated blocks of material. It is applied successfully for the fabrication of dental restorations from a wide variety of materials, but has a limited potential for obtaining complex shapes and results in a substantial waste of material [4]. A

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new approach to fabricating metal frameworks has been introduced, that involves additive manufacturing (AM). Selective laser melting (SLM) is an AM technology that is based on building a three-dimensional restoration by laser fusion of multiple layers of powder-material. The respective layers, corresponding to the virtual cross-sections of a CAD model, are being fused locally to the underlying ones. There is almost no material waste, since the remaining powder can be used for further SLM [2].

Different metal processing routes imply exposure to different heating and cooling conditions, resulting in variations in metal microstructure [5–8], mechanical properties [8,9], and surface characteristics [10,11] that could ultimately affect the porcelain bonding. Thus, contemporary metal processing has added a new variable to porcelain bonding. The differences in strength of porcelain bonds to Co–Cr frameworks fabricated by different technologies are currently being studied [7,10–15], given that an adequate metal–ceramic bond is essential for the longevity of the PFM restorations [16].

A range of noble and base dental alloys are used in PFM prostheses, but the increasing cost of noble metals, together with the high allergenic potential of Ni–Cr alloys, have led to wide acceptance of the Co–Cr alloys as materials of choice for fabricating PFM prostheses [17,18]. There are, however, growing concerns about the limited biocompatibility of these alloys [19–21] and efforts are being made to optimize the Co–Cr processing for improved corrosion behavior [7,22,23]. Commercially pure titanium (cpTi) and titanium alloys (such as Ti-6Al-4V) are known for their attractive properties for dental restorations, including high mechanical and corrosion resistance, excellent biocompatibility, low allergenic potential and low cost [24]. However, Ti-based PFM prostheses have been marginalized clinically for decades, given the limitations of conventional titanium casting [25]. The high melting temperature (1672 °C) of titanium, together with its high reactivity at elevated temperatures and poor castability [26], require application of special casting procedures/equipment. CAD/CAM appears therefore to be particularly attractive for the fabrication of titanium prosthetic frameworks. However, the clinical behavior of titanium PFM constructions has been reported to be poor, revealed in frequent porcelain veneer failures [27–29]. Thus, considerable efforts are being made to improve the titanium–ceramic bond [25,30]. In order to prevent the uncontrolled reactivity of titanium frameworks at high temperatures, they are veneered with ultra-low-fusing porcelains with firing temperatures below 800 °C. These porcelains also have a modified coefficient of thermal expansion (CTE) to match the low CTE of titanium ($9.6 \times 10^{-6} \text{ K}^{-1}$), since a great CTE mismatch may affect the metal–ceramic bond adversely [31]. Nevertheless, the strengths of porcelain bonds to titanium alloys have been reported to be lower than those of noble [32–34] and Ni–Cr dental alloys [35,36].

Little is known about the differences in porcelain bonding to Co–Cr and Ti-based alloys. Furthermore, the effects of the digitalized titanium fabrication on porcelain bonding have not been explored sufficiently. The formation of excessively thick oxide layers on the titanium–ceramic interface, which easily delaminate from the bulk metal, is considered to be one of the main reasons for veneer failures [37]. Thus, the absence of an undesirable oxide layer on digitally fabricated titanium surfaces has been regarded as the key advantage of these technologies over casting [28,38,39]. The surface composition of titanium castings has been relatively well examined [40], however knowledge of the surface chemistry of digitally fabricated titanium is limited.

To date, digitally fabricated alloys have not been tested for porcelain bond strength after thermo-mechanical cycling. Thus, the fatigue performance of digitally fabricated metal–ceramic systems has not been assessed in conditions simulating the oral environment where the PFM prostheses are exposed to frequent

thermal changes and repeated loading [33,34,41–44]. Further, there are no reports in which the porcelain bonds to conventionally and digitally fabricated dental alloys of different composition are compared. The aim of this study was therefore to investigate the effect of thermo-mechanical cycling on the shear strength of porcelain bonds to Co–Cr and Ti-based alloys fabricated by casting, CNC milling and SLM. The null hypothesis was that neither thermo-mechanical cycling nor a metal–ceramic combination would affect the shear bond strength.

2. Materials and methods

A total of 154 metal cylinders (4 mm in diameter and 4 mm in height) were fabricated. They were divided into seven groups of 22 each, according to the applied technology for metal processing (c – for casting; m – for CNC milling; and s – for SLM) and to the chemical composition of the metal substrate (C – for Co–Cr; T – for cpTi and TA – for Ti-6Al-4V). Dental porcelain was subsequently built up on each metal substrate, forming metal–ceramic test specimens (Fig. 1a, b). The groups and materials used in this study are presented in Table 1.

2.1. Fabrication of the metal substrates

Two groups of metal cylinders were cast, one from Co–Cr (cC group) and the other from cpTi (cT group). Wax rods (Dentaurum, Ispringen, Germany), 4 mm in diameter, served as casting templates. They were mounted in a silicone ring and embedded in a refractory investment. To prevent excessive reactivity and to compensate for the high degree of shrinkage of titanium, an investment material with high magnesium and alumina content (Rematitan Plus, Dentaurum, Ispringen, Germany) and a vacuum-pressure casting technique (Rematitan Autocast, Dentaurum, Ispringen, Germany) were used for casting the titanium. The Co–Cr specimens were fabricated in a centrifugal casting unit (Ducatron S3 Prisma, Ugin'Dentaire, Seyssins, France), using a conventional phosphate-bonded investment (Fujinvest, GC, Tokyo, Japan). The castings were divested, and airborne-particle abraded for 10 s, with 110 µm aluminum oxide powder (Korox, Bego, Bremen, Germany) under 2 bar pressure, and at 45° angle/2 cm distance. Each rod was then mounted in a precision cutter (IsoMet 1000, Buehler, Lake Bluff, IL, USA) and cross-sectioned in the radial direction to a final height of 4 mm. In order to preserve the metal surface characteristics obtained by casting, only one end of each rod was cross-sectioned, leaving the other end for porcelain application.

One group of Co–Cr cylinders (mC group) and two groups of titanium cylinders (mT and mTA groups) were fabricated by CNC milling. The cylinders were designed using CAD software (Exocad Dental Cad, Exocad, Darmstadt, Germany), exported to CAM software (Sum3d Dental, CIM system, Cinisello Balsamo, Italy) and milled (Arrow Mill Beluga, Dentas, Maribor, Slovenia) from prefabricated milling blanks. The commercial SLM method Laser-CUSING (Concept Laser, Lichtenfels, Germany) was used to fabricate two groups of metal cylinders, from Co–Cr (sC group) and from Ti-6Al-4V (sTA group). The cylinders were designed in Solidworks CAD software (Dassault Systemes, Waltham, MA, USA) and the CAD file transferred to the SLM unit (Mlab-R, Concept Laser, Lichtenfels, Germany) equipped with a 100 W Yb-fiber laser. The longitudinal axes of the specimens were aligned perpendicularly to the building platform. The layer thickness was set to 25 µm in both groups. The particle sizes of the Co–Cr and Ti-6Al-4V powders used were 10–30 µm and 15–45 µm, respectively. SLM was carried out in an atmosphere of argon. In order to eliminate the residual stresses arising from the localized thermal input from the laser and to tailor the microstructure for improved mechanical

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