



## Mechanical Properties of Cast Commercially Pure Titanium Simulating Ceramic Firing Cycles

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### ABSTRACT

**Aim:** To evaluate the mechanical properties (ultimate tensile strength, elongation and hardness) of the commercially pure titanium (cp Ti) as casting and after ceramic firing cycles.

**Materials and methods:** Dumbbell-shaped specimens were prepared for the tensile strength testing. Disk-shaped cast specimens were used for microhardness testing. The ceramic firing cycles were made simulating a low fusion ceramic application. Tensile testing was conducted in a universal testing machine at a crosshead speed of 1 mm/min until failure. Ultimate tensile strength and elongation were recorded. The fracture mode was analyzed by scanning electron microscopy. Vickers hardness was measured in a hardness tester. The data from the tensile and hardness tests were subjected to a one-way analysis of variance and Tukey's test ( $\alpha = 0.05$ ).

**Results:** The mean values of tensile strength were not changed by the ceramic firing cycles. Lower hardness was observed for cp Ti as casting compared with Ti cast after the firing cycles.

**Clinical significance:** The ceramic firing cycles did not show any considerable prejudicial effects on the mechanical properties of the cp Ti.

**Keywords:** Titanium, Tensile strength, Laboratory research, Hardness.

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### INTRODUCTION

The applications of titanium for medical and dental purposes have been dramatically increased due to its high resistance to corrosion, which contributes to excellent biocompatibility, high strength-to-weight ratio, and low modulus of elasticity.<sup>1</sup> Commercially pure titanium (cp Ti) is frequently used for

dental implants and, more recently, for crowns, partial and complete denture frameworks.

The increase of the misfit marginal has been found for metal copings after simulating the ceramic firing cycles.<sup>2,3</sup> Moreover, the ceramic firing cycles involve high temperatures; this may cause structural changes and, consequently, modifications in the mechanical properties of metal ceramic prostheses, especially in the partial and complete denture. Properties such as tensile strength, elongation and hardness of the metals and metal alloys used to make the framework of metal ceramic dentures have a relationship with the stresses in the bond between ceramic and metal, which has an influence on the system behavior,<sup>4</sup> so these properties have been the focus of many recent studies.<sup>5-8</sup> The literature contains limited information about the effects of the ceramic firing cycles on the mechanical properties of cp Ti.

The aim of this study was to investigate the tensile strength (ultimate tensile strength and elongation) and hardness of the cp Ti as casting and after the ceramic firing cycles, under the hypothesis that the firing cycles can change these properties. The fractured surfaces were characterized after the tensile testing using scanning electronic microscope (SEM).

### MATERIALS AND METHODS

#### Tensile Testing

Fifty cylinder-shaped wax patterns (5-mm diameter and 45-mm length) were prepared for casting in grade II cp Ti (Tritan; Dentaurnum JP Winkelstroeter KG, Pforzheim, Germany). The cp Ti was cast using investment material (Rematitan Plus; Dentaurnum) and an argon arc centrifugal titanium cast machine (Rematitan; Dentaurnum). The burnout schedules for the investment materials and casting

procedures followed the manufacturers' instructions. After casting, the investment blocks were immediately quenched in cold water following the manufacturers' instructions. The castings were retrieved with 100 µm glass particles airborne.

The cylinders were machined using a mechanical lathe under refrigeration and with rotation speed controlled, obtaining dumbbell-shaped specimens (3-mm diameter in the center area). The specimens were evaluated using film radiographic to verify the porous presence in the framework. The porous specimens were replaced with new specimens whenever porosity was found. The sequential shaping and finishing was completed in the surfaces of the specimens as recommended for the veneering ceramic application (Vitatitank ceramic, Vita Zahnfabrik, Bad Säckingen, Germany). The surfaces were ground with tungsten carbide burs and air-abraded with 150 µm Al<sub>2</sub>O<sub>3</sub> powder. The firing cycles were simulated without the application of ceramic,<sup>2</sup> using an adjustable furnace (Austromat M, Dekema Austromat-Keramiköfen, Freilassing, Germany). A recommended low-fusion ceramic firing protocol was followed (Table 1). The specimens were cooled to room temperature after each cycle.

**Table 1:** Firing protocol for veneering ceramic\*

Firing cycle	Initial temperature (°C)	Final temperature (°C)	Heating rate (°C/min)	Vacuum
Bonder	400	800	60	Yes
Opaque	400	790	110	Yes
Dentin	400	770	50	Yes
Glaze	400	770	50	No

\*Recommended protocol was followed for the veneering ceramic (Vitatitank ceramic, Vita Zahnfabrik)

Tensile testing was conducted using a universal testing machine (MTS 810; MTS System Corporation, Minnesota, USA) at a crosshead speed of 1 mm/min until failure. Ultimate tensile strength (MPa) and elongation were recorded by software (Test Work IV for Test Star II; MTS System Corporation, Minnesota, USA) after the rupture. The means and standard deviations of these properties were calculated. Data (n = 10) were statistically analyzed by one-way analysis of variance (ANOVA); (p < 0.05). After tensile testing, the fractured surfaces of representative specimens were observed using a SEM (LEO EVO 40; Carl Zeiss, Germany).

### Vickers Hardness

Fifty disk-shaped wax patterns (6-mm diameter and 3-mm height) were prepared for casting, using grade II cp Ti (Tritan; Dentaureum). The specimens were invested, casted,

and divested as described previously for the tensile specimens, as well as the sequential finishing in one of the disk plan surfaces. The firing cycles also were simulated as described previously.

The specimens were embedded in acrylic resin. Water-cooled sandpapers (Carbimet Paper Disks, Buehler) with 180, 320, 400, 600 and 1200 grits were used in the polishing machine (Metaserv 2000; Buehler, Coventry, West Midlands, England) to grind and polish the disk surfaces. Vickers hardness was measured using a loading of 9.807 N during 5 seconds in a hardness tester (HMV-2; Shimadzu, Tokyo, Japan). Four measurements were averaged for each specimen. The data were statistically analyzed using one-way ANOVA and Tukey's test (p < 0.05).

## RESULTS

### Tensile Testing

The results of the tensile testing are summarized in Table 2. The ANOVA did not present statistical difference in the ultimate tensile strength or the elongation between the groups (p > 0.05).

### Fracture Analysis

Representative specimens in the different groups were characterized using SEM. Figures 1A and B represents the fracture surface of the specimens of cp Ti as casting. The figure shows the presence of porosity with considerable dimension, which was not found by radiographic examination. Some similar porosity also was found in specimens from other groups. The cp Ti as casting showed ductile dimple fracture in a predominant area. Figures 2A and B is representative of the groups submitted to the ceramic firing cycles, suggesting a mix of cleavage (brittle) and ductile dimple fractures.

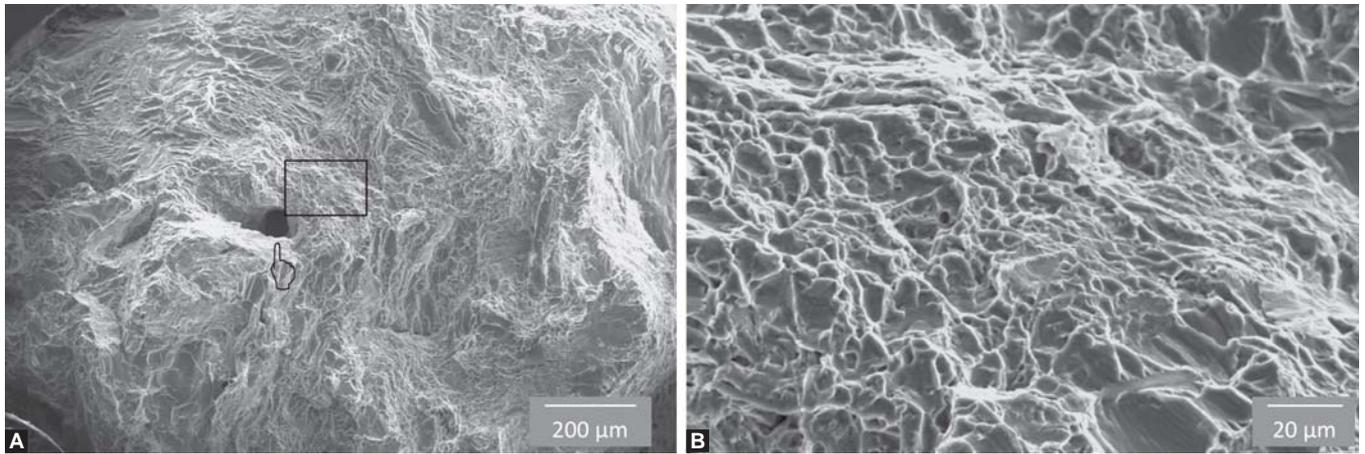
### Vickers Hardness

The results of the hardness testing are summarized in Table 2. The ANOVA presented statistical difference on the hardness between the groups (p < 0.05). Tukey's test

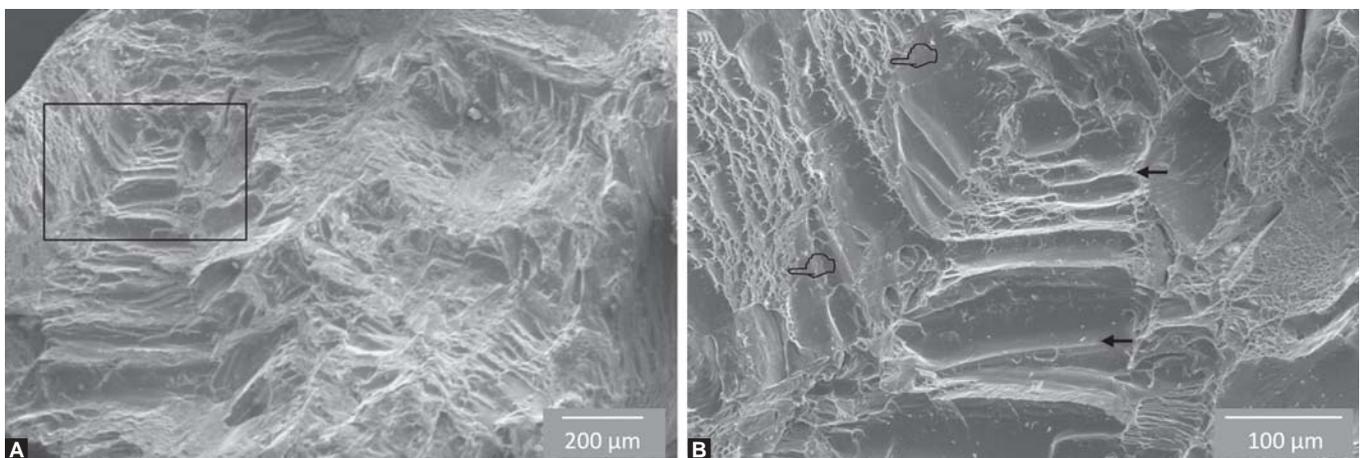
**Table 2:** Mechanical properties of cp Ti cast in each ceramic firing cycle

Firing cycles	Ultimate tensile strength (MPa)	Elongation (mm)	Hardness (VHN)
Initial	494 (54)	3.1 (1.0)	187 (6) b
Bonder	511 (52)	3.0 (0.9)	206 (3) a
Opaque	505 (45)	2.7 (0.4)	200 (3) a
Dentin	482 (69)	3.0 (0.9)	202 (2) a
Glaze	493 (33)	2.7 (0.9)	207 (7) a

Identical letters in same column indicate no statistical differences (p > 0.05) using Tukey's test for hardness test



**Figs 1A and B:** (A) SEM of the fracture surface for cp Ti as casting specimen, presenting ductile dimple fracture in predominant areas and the presence of porosity (pointer), (B) higher magnification of the Figure 1A (delimited area), representing areas of ductile dimple fracture



**Figs 2A and B:** (A) SEM of the fracture surface for cp Ti specimen submitted to the ceramic firing cycles, (B) higher magnification of the Figure 2A (delimited area), representing areas of cleavage (arrows) and ductile dimple (pointers) fractures

showed lower hardness for cp Ti as casting compared with cp Ti after the firing cycles ( $p < 0.05$ ). No statistical difference was found between the firing cycles ( $p > 0.05$ ).

## DISCUSSION

No significant difference was found in the mean values of elongation or the ultimate tensile strengths between cp Ti as casting and after being submitted to ceramic firing cycles. The temperatures used in the ceramic firing cycles in the present study had values up to 800°C, as recommended by Kimura et al.<sup>9</sup> Titanium has two crystal modifications: The close-packed hexagonal structure, up to 882°C; and above this temperature the body-centered cubic structure, making the framework more fragile.<sup>10</sup> Therefore, this issue could explain why the ceramic firing cycles had no influence on the tensile strength.

The reaction between molten titanium and investment material during casting increase the hardness in the surface<sup>1,8</sup> and dimensional change had an influence on the metal mechanical properties.<sup>11</sup> The specimens used in the tensile

test had the surface layer removed during fabrication. While the Ti present oxidation again during the firing cycles, this oxidation layer had no influence on the tensile strength, and there was no significant difference between the groups after the ceramic firing cycles.

The SEM analysis showed areas with the presence of dimples for cp Ti as casting. Azevedo and Santos<sup>12</sup> found that fibrous surfaces containing dimples might be produced by a ductile overload mechanism (unstable fracture). The specimens submitted to ceramic firing cycles presented mixed areas of cleavage (brittle) and dimple fractures, suggesting behavior with lower ductility compared to cp Ti as casting. Fragile fracture is most damaging only to the metal framework, if it occurs quickly without the presence of plastic deformation. Ductile fracture is preceded by plastic deformation, which allows detection of an eminent occurrence of ductile fracture. However, in metal-ceramic prostheses, all deformation of the metal framework higher than ceramic strength induces a fracture without metal fracture.

The cp Ti as casting presented hardness mean values of 187 (VHN) in agreement with other studies.<sup>5-7</sup> Hardness evaluation in metals or alloys may be related, in the same indirect mode, with factors influencing the clinical performance. The metal alloys can contact the opposing teeth in some cases, such as with a resin-bonded fixed partial denture. The hardness of the alloy must be sufficient to resist occlusal forces but not inflict wear on opposing teeth. Generally, alloys with Vickers hardness between 125 and 340 kg/mm<sup>2</sup> will avoid these conditions.<sup>13</sup>

The cp Ti as casting presented lower mean values of hardness than that found after the ceramic firing cycles. This finding is in agreement with a simulation of the firing cycles to other ceramics.<sup>14</sup> The alpha case layer was probably partially removed when the finishing was accomplished for the hardness test. This layer found in the cp Ti cast surfaces has greater hardness than in the bulk of the same specimens.<sup>5,6</sup> This fact occurs by oxygenic diffusion between grains, which changes the hardness of the cp Ti.<sup>8</sup> Despite the fact that the vacuum was not absolute in the furnace, the oxidation that may have occurred presented no considerable effect on the surface hardness of the cp Ti for these simulated firing cycles. The results of the current study suggest a change to a higher stiffness of the cp Ti after the ceramic firing cycles. However, this stiffness should not present a problem to the framework of the ceramic-veneered titanium restorations.

Clinically, the principal problem of the ceramic-veneered titanium restorations has been the ceramic chipping, principally in the fixed partial dentures.<sup>15</sup> A recent study evaluating the clinical performances of CAD/CAM titanium ceramic crowns over 3 years were acceptable, with no biologic complications and a high cumulative survival rate. However, ceramic chipping occurred frequently, and some restorations were not repairable and needed to be remade.<sup>16</sup> The use of titanium in metal-ceramic prostheses may be an option for treatment. However, further studies are required in order to improve the bond between metal and ceramic, and clinical studies are needed to confirm this improvement.

## CONCLUSION

The ceramic firing cycles showed no influence on the tensile strength of the commercially pure titanium, considering ultimate tensile strength, as well as elongation. The commercially pure titanium as casting presented fractured surfaces with predominant ductile dimple fractures. After the ceramic firing cycles, the fractured surfaces presented more mixed areas with cleavage and ductile dimple fractures. Hardness increased after the firing cycles,

although the effect was similar between the firing cycles. Overall, the firing cycles presented no negative effects on the mechanical properties evaluated of the commercially pure titanium concerning a metal-ceramic restoration.

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