

## Effect of the ceramic mould composition on the surface quality of As-cast titanium alloy

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Titanium has received growing attention from researchers and clinic professionals in dentistry as a consequence of its excellent biocompatibility, chemical, physical and mechanical properties [1]. This combination of properties makes titanium and its alloys one of the most widely used material for dental implants and partial denture prosthesis. Casting processes for commercially pure titanium (cp Ti) and its alloys, such as the Ti-Al-V grades, have increased in quality during the last years, stimulating its clinical use. However, the surface quality of the parts is process sensitive, and surface damage associated with porosity, inclusions, surface microstructure, adhesion, and roughness may be present in different levels. In the last years a growing number of papers have been published regarding the influence of the casting parameters on the titanium dental part quality. In order to achieve the best process conditions to increase the soundness of the cast surface, important parameters have been reported as: investment mould oxide composition [2, 3], casting temperature [2], mould temperature [4], type of atmosphere and partial pressure of the gases [5], process pressure [6].

The high titanium reactivity is well known as is its capability to reduce some oxides normally used for the investment mould production and crucible materials. The practical results of this strong reaction with the mould material is the formation of the  $\alpha$ -case, composed of titanium oxide, titanium silicates and solid solutions [7]. Considering the mould composition, more stable oxides have been selected such as: calcium [8], magnesium [1], zirconium [9], aluminium [7] and Yttrium [10]. These oxides are used in substitution of silica or as a coating diffusion barrier on the mould surface. Castings for dentistry require the production of serial parts with complex shape and tight tissue toler-

ances. Therefore, considering the cast parts fabrication, the  $\alpha$ -case formation and the surface quality are very important parameters to be controlled, because of the interaction between the liquid metal and the ceramic mould. To best understand the metal/mould interaction reactions during the casting process, we must consider the mould composition and the thermodynamic aspects present at the liquid/solid interface during the casting.

In the present work three types of mould ceramic materials were selected. Materials were received as powders to test the reactivity with molten titanium. The selected commercial investment systems were: Rematitan-Plus<sup>®</sup> (R-Plus), Rematitan-Ultra<sup>®</sup> (R-Ultra), and Ticoat-Manfredi<sup>®</sup> (T-Manfredi). The chemical composition for the refractory ceramic powders is shown in Table I. Wax moulds for castings were prepared according to the procedure described by [11]. Metallic rings were used as inclusions. Castings were carried out using an argon-casting machine, type Easy Ti<sup>®</sup>, with induction heating and a centrifugal liquid metal injection system.

Surface quality was examined first by visual methods. Scanning Electron Microscopy was performed on a LEO 440I microscope coupled with an X-ray Energy Dispersive Electron Spectrometry (EDS) microanalysis Li/Si system from Oxford. The samples were analysed without mounting or coating using secondary and backscattered electron detectors. The chemical composition of the ceramic powders was determined by instrumental methods.

According to the chemical composition of the ceramic powders shown in Table I, we observe different oxide constitution among the materials analysed. The R-Plus powder is based on the SiO<sub>2</sub>/Al<sub>2</sub>O<sub>3</sub> oxide system, the R-Ultra on MgO, and the T-Manfredi on SiO<sub>2</sub>/ZrO<sub>2</sub>. Considering the stability of the different

TABLE 1 Chemical composition of the ceramic powders (mass %)

Mould material	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	MgO	P <sub>2</sub> O <sub>5</sub>	ZrO <sub>2</sub>	CaO	Total	Fire loss	Others
R-Plus	62.89	20.80	7.68	4.80	0.01	0.17	96.35	3.43	0.22
R-Ultra	0.69	16.12	71.91	0.02	5.64	0.70	95.08	4.24	0.68
T-Manfredi	69.40	0.12	6.50	5.51	12.32	0.17	94.02	5.42	0.56

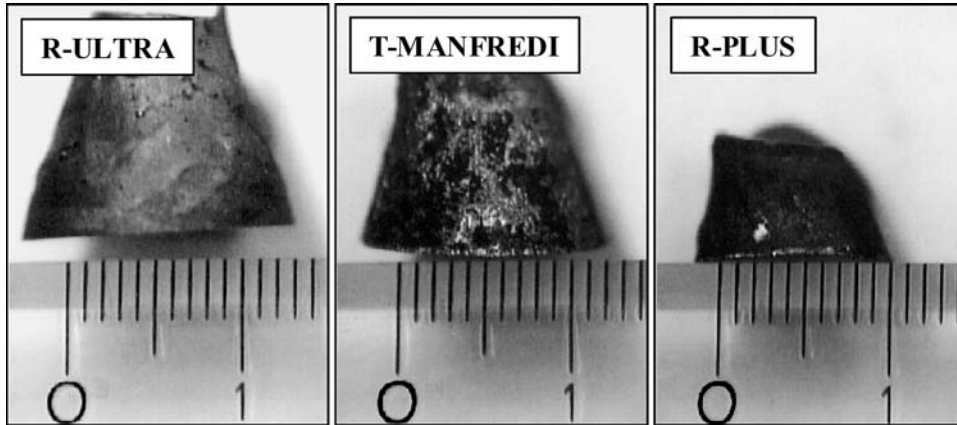


Figure 1 Surface quality of the as-cast Ti-alloy parts for the different ceramic mould materials.

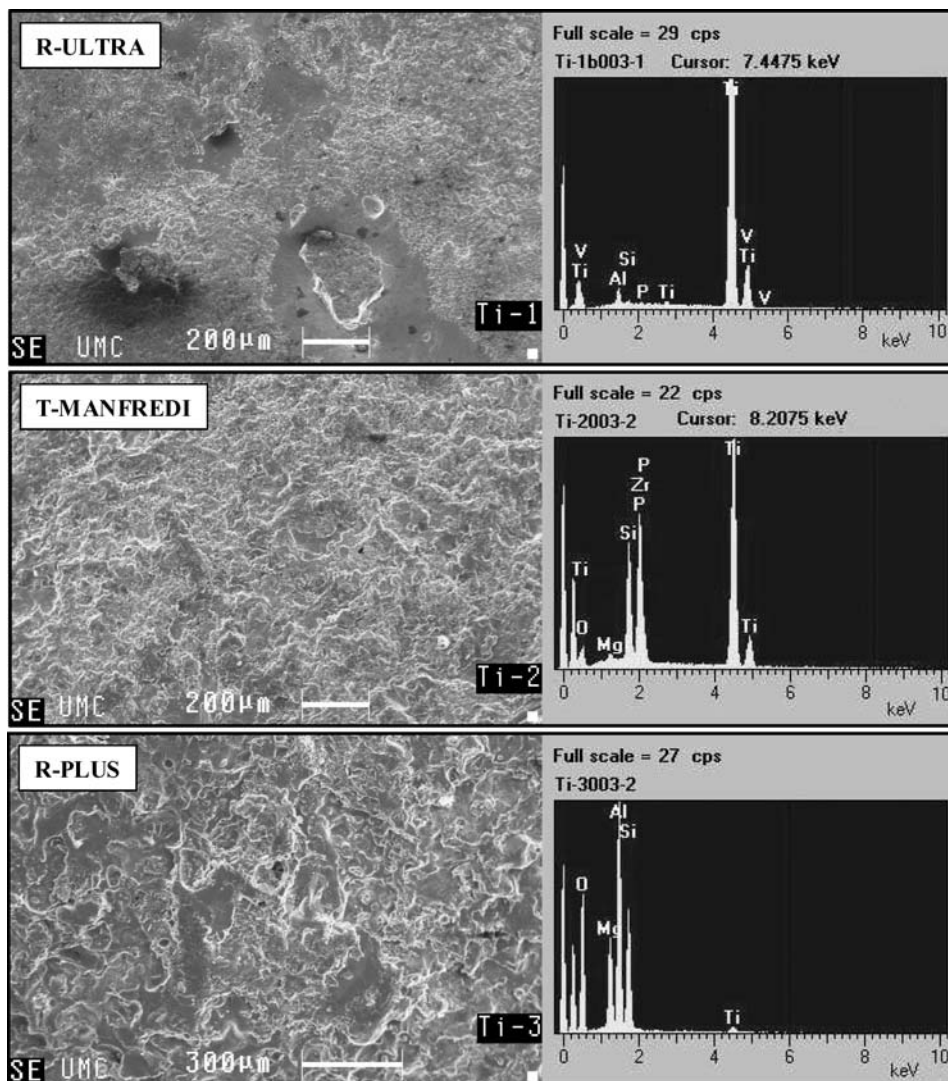


Figure 2 Scanning Electro Microscopy of the as-cast surfaces, and EDS microprobe analysis.

oxides present on the ceramic materials, related to the Gibbs Free Energy for oxide formation, different reactions should be expected between the mould and the liquid titanium during the casting process [12]. Fig. 1 shows the Ti-alloy parts after casting. It is noticeable that there is a difference in the surface quality depending on the different ceramic materials used on the moulds. The best result was obtained with the R-Ultra/MgO based system, with minimum reaction products on the cast surface. For the systems containing SiO<sub>2</sub> the reaction intensity increases from the T-Manfredi to the R-Plus.

To best understand the surface reactions on casting additional SEM and EDS analyses were performed. Fig. 2 shows the surface MEV topography by secondary electron detector and the EDS microprobe analysis for the samples. Fig. 2a shows the lower interaction between the Ti-alloy and the R-Ultra mould. In this case, the EDS spectrum shows only peaks related to Ti and the alloying elements, Al and V. No peaks from oxide ceramic mould reaction were found. For the ceramic T-Manfredi the surface is rougher than the former and the EDS spectrum shows peaks related to the elements P, Zr, Si and Mg, incorporated to the surface from the reduction reactions with Ti, Fig. 2b. A strong interaction between metal/mould was found for the material R-Plus, Fig. 2c. Not only has the surface roughness increased compared to the former mould materials, but the EDS spectrum shows peaks for Mg, Si, Al, Ca and the Ti peak from the metallic alloy appears with a very low intensity.

From these results it is possible to conclude that the casting of Ti and Ti-alloy is very sensitive to the ceramic mould chemical composition. Titanium/oxide equilibrium has four types of oxide, TiO, Ti<sub>2</sub>O<sub>3</sub>, Ti<sub>3</sub>O<sub>5</sub>, and TiO<sub>2</sub> [12]. However, according to [13], the most stable oxide is the TiO<sub>2</sub>. Based on this, the metal must reduce all the oxides with Gibbs free energy higher than

that for the TiO<sub>2</sub> during the casting process. The best mould material is that for which the selected elements have Gibbs free energy for oxide formation,  $Me + 1/2 O_2 \rightleftharpoons MeO$ , lower than that of TiO<sub>2</sub>, as predicted by the Ellingham Diagram. In the present work this material is the R-Ultra, based on the MgO oxide. The others mould materials, T-Manfredi and R-Plus, have elements which interact with titanium during the casting process, such as SiO<sub>2</sub>, and impair the surface quality of the as-cast Ti part.

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