The Effect of Coating Patterns with Spinel-Based Investment on the Castability and Porosity of Titanium Cast into Three Phosphate-Bonded Investments

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Abstract

Purpose: This study evaluated the effect of pattern coating with spinel-based investment Rematitan Ultra (RU) on the castability and internal porosity of commercially pure (CP) titanium invested into phosphate-bonded investments. The apparent porosity of the investment was also measured.
Materials and Methods: Square patterns ($15 \times 15 \times 0.3$ mm^3) were either coated with RU, or not and invested into the phosphate-bonded investments: Rematitan Plus (RP), Rema Exakt (RE), Castorit Super C (CA), and RU (control group). The castings were made in an Ar-arc vacuum-pressure machine. The castability area (mm^2) was measured by an image-analysis system ($n = 10$). For internal porosity, the casting ($12 \times 12 \times 2$ mm^3) was studied by the X-ray method, and the projected porous area percentage was measured by an image-analysis system ($n = 10$). The apparent porosity of the investment ($n = 10$) was measured in accordance with the ASTM C373-88 standard.
Results: Analysis of variance (One-way ANOVA) of castability was significant, and the Tukey test indicated that RU had the highest mean but the investing technique with coating increased the castability for all phosphate-bonded investments. The analysis of the internal porosity of the cast by the nonparametric test demonstrated that the RP, RE, and CA with coating and RP without coating did not differ from the control group (RU), while the CA and RE casts without coating were more porous. The one-way ANOVA of apparent porosity of the investment was significant, and the Tukey test showed that the means of RU (36.10%) and CA (37.22%) were higher than those of RP (25.91%) and RE (26.02%).
Conclusion: Pattern coating with spinel-based material prior to phosphate-bonded investments can influence the castability and the internal porosity of CP Ti.

Titanium and its alloys are promising materials in the dental field because they have good corrosion-resistance, a low specific gravity, excellent biocompatibility,[1,2] low cost, and mechanical properties nearly equal to those of dental gold alloys;[3-7] however, Ti's low density associated with a high-melting point (1720°C) and the high chemical reactivity of Ti with elements in the investment[1,2,8,9] require complex casting machines[6] and special investment.[10] Some of the problems frequently observed are incomplete casting and internal porosity,[11] which are dependent on external factors such as vacuum or pressure applied on the mold, gas permeability of the investment, increased mold temperature, and superheat of the melt.[11-13] In addition, the presence of several elements in the composition of the investments leads to a chemical reaction with the molten Ti.[14] This reaction forms a superficial contamination zone (α-case) responsible for the increase in hardness and brittleness of the Ti.[15,16]

To prevent Ti contamination, a stable high-temperature resistance oxide can serve as a barrier for reducing reactivity.[15] Special mold materials containing MgO, Al_2O_3, ZrO_2, and CaO as refractories have been used for Ti casting and have produced a reduced α-case thickness;[16,17] however, these investments do not have suitable thermal expansion,[18] air permeability, or mechanical properties, and

Table 1 Materials and manufacturers

Commercial brand	Chemical composition*	Manufacturer	Batch number
Rematitan Ultra (RU)	MgO: 60% to 80%; Al_2O_3:10% to 20%; ZrO_2:3% to 8%; MgO binder	Dentaurum, Ispringen, Germany	010440
Rematitan Plus (RP)	MgO:10% to 30%; $NH_4H_2PO_4$:5% to 10%; SiO_2:55% to 75%; Al_2O_3:10% to 25%	Dentaurum, Ispringen, Germany	070192
Castorite Super C (CA)	MgO:6% to 19%; $NH_4H_2PO_4$:10% to 20%; SiO_2:60% to 80%	Dentaurum, Ispringen, Germany	080232
Rema Exakt (RE)	MgO:10% to 30%; $NH_4H_2PO_4$:10% to 30%; SiO_2:60% to 80%; FeO_2:0% to 1%	Dentaurum, Ispringen, Germany	070566

their high cost significantly increases the total cost of the framework.[2,4,5,16]

The investments commonly used for high temperature alloys are silica based, either as crystalline quartz or cristobalite for imparting refractoriness.[19] Despite these investments presenting a thermal expansion compatible with that of Ti, Si has a high chemical affinity for Ti, forming Ti-Si compounds,[5] modifying the physical properties of Ti, and degrading its mechanical properties.[5,19] Moreover, at high casting temperatures the chemical reactivity increases.[20]

In a previous investigation, Ferreira et al[21] studied the setting and thermal expansions of three phosphate-bonded investments: Rematitan Plus (RP) (Dentaurum, Ispringen, Germany), Rema Exakt (RE) (Dentaurum), and Castorit Super C (CA) (Dentaurum). The expansion of RP (control group) at the temperature recommended by its manufacturer (430°C) was 0.86%; RE had similar expansion at 236°C and CA at 69°C. The low casting temperature indicated by Ferreira et al[21] could be interesting with regard to decreasing the chemical reaction between the molten Ti and the investment materials. Nevertheless, the vast difference between the mold and melting temperatures may make it difficult to fill the mold. Furthermore, Takahashi et al[17] affirmed that the composition of phosphate-bonded investments may increase the reactivity between the investment and the molten Ti. In their study, the investments with higher cristobalite content had lower castability and higher surface reaction than those with quartz as the main refractory. Ferreira et al,[21] based on the analysis of the thermal expansion curve, supposed that the investments studied had different cristobalite and quartz content, and this could lead to various levels of Ti contamination.

Some studies have investigated the success of techniques using wax coating with more stable oxides, such as ZrO_2, Y_2O_3, and zirconite before investing into phosphate-bonded investments to prevent the surface attack;[4,5,13,19] however, the castability and porosity of Ti with spinel-based material (MgO/Al_2O_3) used as coating has not yet been studied. This technique could associate the chemical stability of the spinel-based material with the high expansion and low cost of the phosphate-bonded investment.

The purpose of this study was to evaluate the effect of pattern coating with spinel-based investment (Rematitan Ultra [RU], Dentaurum) on the castability and the internal porosity of CP Ti invested into three phosphate-bonded investments, RP, RE, and CA, at the casting temperatures determined by Ferreira et al.[21] In addition, the apparent porosity of these investments was measured.

Materials and methods

The materials used in the present study are presented in Table 1.

Castability of titanium

Square acetate plate-type patterns measuring 15 × 15 × 0.3 mm³ were sprued and coated with spinel-based investment (RU), or not coated and invested into one of three commercial phosphate-bonded investments: RP—Rematitan Plus (especially for titanium), RE—Rema Exakt, and CA—Castorit Super C. The spinel-based investment RU—Rematitan Ultra was the control group. Table 2 presents the experimental groups (n = 10).

To prepare the coated groups, the spinel-based investment (RU) was handled using the usual liquid ratio and applied on the pattern with a brush to cover the model with a coating layer. The coat was 0.2- to 0.4-mm thick, but its layer thickness could not be standardized. The investments were mixed according to manufacturer's instructions and fired in an electric furnace EDG 7000 (EDG Equipments, Sao Carlos, Brazil) at a rate of 5°C/min, in accordance with the heating program presented in Table 3.

In the pilot study, the casts made with the RE and CA investments at higher temperatures, similar to that used in the control group (430°C), were eliminated from the study because they did not produce acceptable castings (Fig 1). The casting temperatures for RE (236°C) and CA (69°C) selected for this study

Table 2 Groups of castability and porosity of titanium tests

Investment	Investing technique	Group
Rematitan Ultra	Uncoated	RU
Rematitan Plus	Uncoated	RP
Rematitan Plus	Coated	RPC
Castorit Super C	Uncoated	CA
Castorit Super C	Coated	CAC
Rema Exakt	Uncoated	RE
Rema Exakt	Coated	REC

Table 3 Program of heating for investments

Material	Program	Phase 1	Phase 2	Phase 3	Phase 4	Casting
Rematitan Plus (RP)	Temperature time	150°C 90 minutes	250°C 90 minutes	1000°C 60 minutes	430°C 30 minutes	430°C
Rematitan Ultra (RU)	Temperature time	250°C 90 minutes	885°C 30 minutes	430°C 30 minutes		430°C
Castorit Super C (CA)	Temperature time	250°C 60 minutes	950°C 30 minutes	69°C 30 minutes		69°C
Rema Exakt(RE)	Temperature time	250°C 60 minutes	1050°C 60 minutes	236°C 30 minutes		236°C

were those at which the investments have expansion similar to that of RP, according of Ferreira et al.[21]

The molds were slowly cooled inside the furnace to the different final temperatures (Table 3), and the cast procedure was carried out in the Discovered Plasma Ar-arc vacuum-pressure casting machine (EDG Equipments), which consists of two chambers: (1) an upper melting chamber, which houses a copper crucible and a tungsten electrode and (2) a lower casting chamber where the invested mold is placed. For each casting, 15 g CP Ti grade 2 (RMI Company, Niles, OH) was used.

All castings were removed from the molds and cleaned in an ultrasonic cleaner. A diamond-cutting disc separated the specimen from the sprue. The specimens were photographed with a digital camera (Cyber-Shot P71, Sony Electronics Inc., Oradell, NJ), and the area (mm^2) of the specimens was measured with the Leica Qwin image-analysis system (Leica Microsystems Imaging Solutions Ltd., Cambridge, UK).

Data were analyzed by one-way ANOVA, and the mean values were compared by the Tukey HSD test ($p < 0.05$). Student's *t*-test was performed for each phosphate-bonded material with or without coating technique.

Internal porosity of titanium

Square resin patterns (Pattern Resin LS, GC America Inc, Alsip, IL) measuring $12 \times 12 \times 2$ mm^3 were made, and the casting technique used was in accordance with the description of the castability test. The porosity was determined radiographically with digital imaging software Vix Win Pro 1.5D (Gendex Dental Systems, Des Plaines, IL) using a dental radiographic unit (Denoptix Imaging Plate 57×76 mm^2, Gendex Dental Systems). All settings were constant (65 kV, 7 mA, 0.160 second) using the digital X-ray unit Model 765 DC (Gendex Dental Systems). The specimen was positioned perpendicularly to the imaging plate at a distance of 40 cm ($n = 10$). The porosity percentage of the projected porous area for each specimen was quantitatively evaluated using a standard image-analysis technique[22] with UTHSCSA Image Tool 3.0 (San Antonio, TX). Kruskal-Wallis nonparametric statistical analysis was performed, and the internal porosity of Ti of the groups was compared by the Student-Newman-Keuls test ($p < 0.05$).

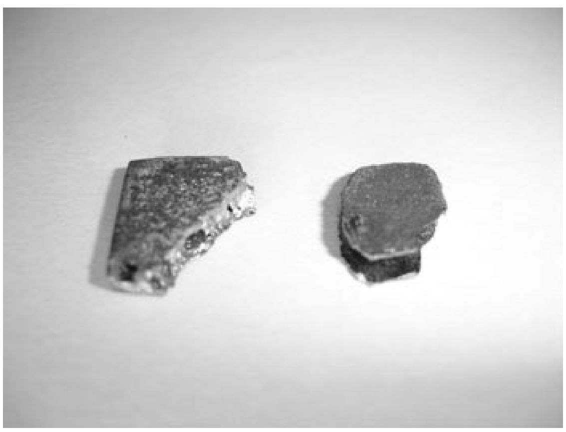

Figure 1 Titanium casts obtained from CA and RE investments heated at high temperatures.

Apparent porosity of the investments

Specimens measuring 20 mm in diameter and 3-mm high were made using a silicone mold. The investments RU, RP, CA, and RE ($n = 10$) were mixed and fired according to the manufacturers' instructions and the heating program (Table 3). Heating was carried out in an electric furnace (EDG 7000, EDG Equipments) at the rate of 5°C/min, and the specimens were slowly cooled to ambient temperature inside the furnace.

The apparent porosity (P) was calculated using Archimedes' Principle, a procedure defined by ASTM C373-88,[23] according the following formula:

$$P = ([M - D])/([M - S]) \times 100$$

where P is the apparent porosity, D is the dry mass, M is the saturated mass, and S is mass suspended in water.

After heating, the dry mass (D) of specimens was measured in a precision balance Sartorius Model 2623 (Sartorius Group, Gottingen, Germany) with accuracy of 0.0001 g. The specimens were stored in distilled water for 3 days. After impregnation, the saturated mass (M) and mass suspended in water (S) were measured using the same scale. Data were analyzed by one-way ANOVA, and the mean values compared by the post hoc Tukey HSD test ($p < 0.05$).

Results

Castability of titanium

Mean castability values and standard deviations for each group are presented in Figure 2. The one-way ANOVA (Table 4) was significant ($p < 0.001$), and the Tukey post-hoc test showed there were significant differences between RU and the other investments. The RP, RPC, CA, CAC, and RE groups demonstrated significantly lower castability, and REC showed an intermediary mean among RU and the other investments. When

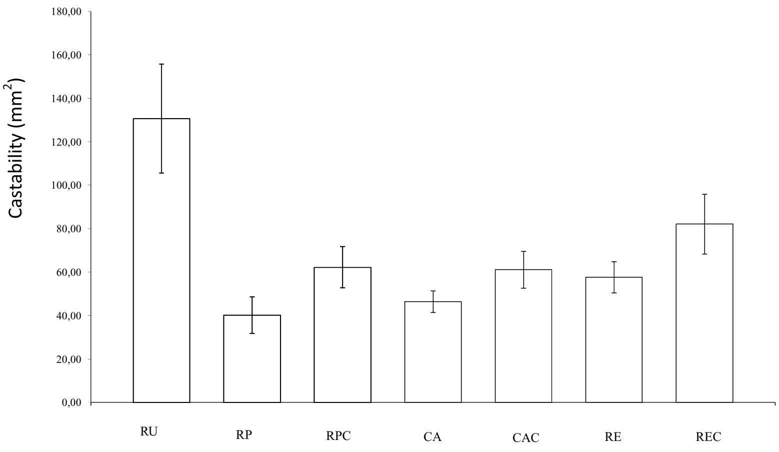

Figure 2 Mean and standard deviation (SD) of castability (mm^2).

the investing technique was investigated, a significantly higher castability mean was observed for the coating technique for all phosphate-bonded investments. To confirm these observations, Student's *t*-test was performed for each phosphate-bonded material with or without coating technique. These comparisons for each investment were significant for RP and RPC ($p = 0.005$), RE and REC ($p = 0.039$), and CA and CAC ($p = 0.005$).

Internal porosity of titanium

The means of internal porosity (%) are presented in Figure 3. Because the data did not show homogeneity and normal distribution, the nonparametric test was carried out. The Kruskal-Wallis test was statistically significant ($p < 0.001$), and the Student-Newman-Keuls multiple comparison test showed that RU (control group) was not statistically different from the following phosphate-bonded investment groups: RP ($p = 0.931$), RPC ($p = 0.510$), CAC ($p = 0.065$), and REC ($p = 0.188$), but CA ($p = 0.011$) and RE ($p = 0.046$) did differ from RU. The comparison between each investment with or without coating technique was not significant for RP (RP *versus* RPC; $p = 0.567$) and RE (RE *versus* REC; $p = 0.051$), but it was significant for CA (CA *versus* CAC; $p < 0.001$).

Apparent porosity of investments

The mean and standard deviation are presented in Figure 4. One-way ANOVA was significant (Table 5), and the Tukey test indicated that the mean apparent porosity of RU was similar to that of CA ($p = 0.092$) and higher than those of the RE ($p < 0.021$) and RP ($p < 0.001$) investments. CA had higher apparent porosity than RP ($p < 0.001$) and RE ($p < 0.001$). RP was not statistically different from RE ($p = 0.174$).

Table 4 One-way ANOVA for the castability test

	SS	df	MS	F	*p*
Between groups	55,522.168	6	9253.695	202.583	<0.001
Within groups	2877.743	63	45.678		
Total	58,399.911	69			

Discussion

The results of castability (Fig 2, Table 4) indicate that the spinel-based investment RU is more suitable than the phosphate-bonded investments. Some studies[10,13,15,20] have shown that the castability of CP titanium into a spinel-based investment is better than it is with a phosphate-bonded investment.

The chemical stability of the investment against molten Ti has great influence on the castability.[2,9] When molten Ti comes into contact with the mold surface, it reduces the oxides of the investment material and liberates elements such as Si, O, P, and Fe to form various compounds with Ti or remains in solid solution after solidification.[5,16,19] The phosphate-bonded investment is crystalline quartz-, cristobalite-, and P_2O_5-based, which are more easily reduced by Ti than are MgO and Al_2O_3.[19] Moreover, when there is a greater difference between the Ti melting point and the mold temperature, the molten Ti solidifies very quickly. Hence, there is not enough time for the mold margins to be effectively filled.[2,5,16] In contrast, the reaction between the mold surface and molten Ti is minimized at a lower temperature,[24] because the heat transference between the metal and mold is accelerated, minimizing the time for diffusion of the elements coming from the investment.[5]

Various authors[4–6,13,15,19,25] have studied the use of coatings on the casting pattern before investment in phosphate-bonded investments, such as a diffusion barrier to prevent reactions between the liquid metal and investment material. To control the extent of Ti/mold interface reaction and the diffusion of reaction products into the Ti surface layer, stable oxides, such as ZrO_2, Y_2O_3, MgO, and zirconite, that cannot be reduced by Ti have been posited as material coating.[19] On the other hand, the stable refractory oxides need a binder system, which may compromise the relative inertness of the refractory oxides.[19]

These studies proposed coatings made of slurries based on relatively inert oxides, but their preparation demands special procedures, which are a barrier to their regular use. The main purpose of this study was to test making the coating with a commercial investment that is easily found on the dental market.

No studies that used spinel-based investment as material coating were found, but it is known that MgO-based investments are less reactive with molten Ti. Guilin et al[3] studied the

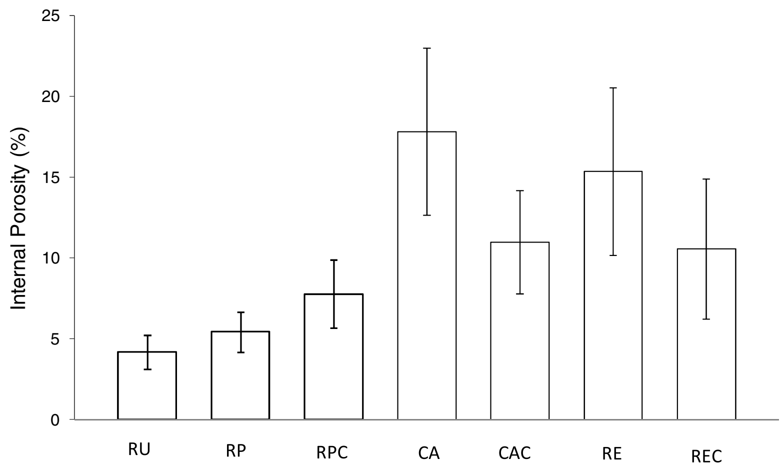

Figure 3 Mean and standard deviation (SD) of internal porosity of titanium (%).

effects of SiO_2-, Al_2O_3-, and MgO-based investments on the α-case layer and demonstrated that MgO cannot react with molten Ti. The composition of the spinel-based investment is mainly MgO and Al_2O_3 (approximately 80% and 20%, respectively). Since MgO cannot be reduced by Ti, the reaction may occur with Al_2O_3, but less intensely than with SiO_2- or Al_2O_3-based investments.

In this study, although better results were obtained with the spinel-based investment (RU), the comparison between each phosphate-bonded investment with coated or uncoated patterns showed that the coating technique increased the Ti castability. The results suggest that the spinel-based coating is able to decrease the effects of the contaminant elements from the phosphate-bonded investments on the Ti surface.

The uncontrolled layer thickness could be a concern. The coating thickness required to obtain an efficient effect on Ti protection is unknown. In this study, it was not possible to standardize the coating layer, and we were unable to find references that explained ways to control layer thickness. Wang et al[19] painted a 0.2-mm-thick layer over the patterns with a brush, but they did not explain how the thickness was controlled. Papadopoulos et al[5] and Luo et al[4] applied different slurries, but they did not comment on how the coatings were applied or how thick they were. Koike et al[25] dipped the sprued wax pattern into a methyl cellulose-based slurry with Y_2O_3 or ZrO_2 three times, but the final coating thickness was not mentioned.

The internal porosities of Ti casting in RU presented significant difference between CA and RE, but the RP groups (RP and RPC), CAC, and REC were not different from the control group. CP Ti melts at 1670°C, but the casting temperatures in CA and RE were 69°C and 236°C (Table 3). The vast difference between the mold and melting temperatures might accelerate the cooling and solidification of liquid metal before filling the mold.[5,13,15] This reduces the chances for gas to escape, and eventually, traps the gas bubbles within the metal casting;[22] however, the reaction layer may have influenced the mold filling of groups CA and RE because the groups with coating technique (RPC, CAC, REC) had internal porosity similar to that of the control group (RU).

In this study, the apparent porosity of the investments did not have a direct influence on the castability and internal porosity of Ti (Fig 4), since CA had a high apparent porosity value but poor castability and a higher internal porosity of Ti than the control group (RU). Other factors, such as refractory composition and casting temperature may influence these properties more than the apparent porosity of the investments does.[13,17,26]

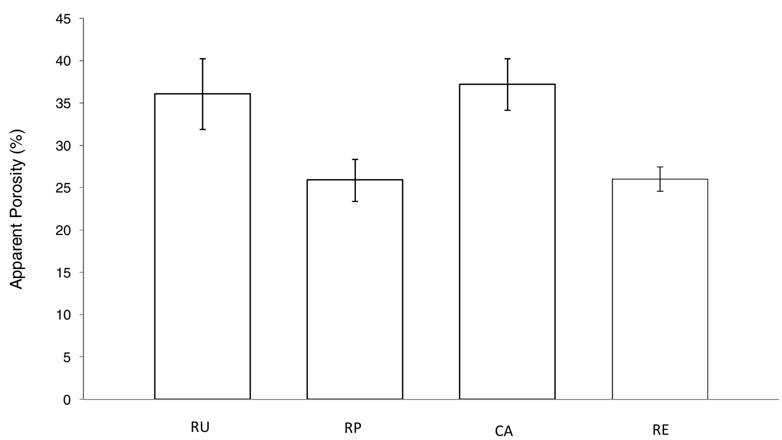

Figure 4 Mean and standard deviation (SD) of apparent porosity of the investments (%).

Table 5 One-way ANOVA to apparent porosity of investment

	SS	df	MS	F	p
Between groups	1159.116	3	386.3719	398.73	<0.001
Within groups	34.88	36	0.9689		
Total	1193.996	39			

The castability test and the internal porosity measurement show that the coating technique may improve the quality of Ti castings made with phosphate-bonded investments. It is a possible alternative to the high cost and lengthy preparation of special MgO/Al_2O_3-based investments. Finally, this research had limitations with regard to the influence of different temperatures and pattern shape on the castability and internal porosity; therefore, further studies must be conducted to evaluate the effects of these factors on other properties of CP Ti castings and fit of crowns.

Conclusion

Within the limitations of this study, the following conclusions were drawn:

(1) The casts made with the spinel-based investment (RU) had better castability than the phosphate-bonded investments. The castability was significantly influenced by the coating technique prior to phosphate-bonded investment.
(2) The internal porosity of CP titanium was affected by the use of the coating technique in castings made into RE and CA but had no effect on RP.
(3) The apparent porosity of the investment was greater in CA and RU than RP and RE, but it did not directly influence the castability and internal porosity of Ti under the conditions of this study.

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