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Evaluation of different bonded investments for dental titanium casting

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The properties of several different investments were investigated including phosphate bonded, magnesia bonded, and alumina cement investments. Measurements included the setting expansion, thermal expansion, and compressive strength of investments, as well as the tensile strength, elongation, Vickers hardness (VHN) and surface roughness of titanium castings. For phosphate bonded investment, the setting expansion after being mixed with its own mixing solution was 2.10%, which was larger than the other investments; the thermal expansion was −0.25% at 200 °C, the compressive strength 14 and 5 MPa after heating. For titanium cast in phosphate bonded investment, the hardness on its top surface was 655 Hv, the tensile strength was 379 MPa, the elongation was 19.4%, and the surface roughness was 2.29 μm. Athough the thermal expansion of phosphate bonded investment is small, the setting expansion is large enough to compensate for the shrinkage of titanium castings. As its thermal expansion at $T \geq 600$ °C was constant and its heating-cooling cycle was almost reversible, these two properties can reduce the thermal shock and thus avoid cracking of the investment.

1. Introduction

Investment materials for titanium castings have been widely studied in recent years. Titanium will react with the refractory material at high temperature due to its high melting point. The mechanism of reactivity and castibility for titanium castings have been studied [1]. And it appears that the diffusion of elements from the investment into the interior of the titanium castings can change their mechanical properties [2]. Though the conventional phosphate bonded investment containing silica exhibits a compatible thermal expansion with titanium, it causes the formation of a reaction layer on the surface of castings [3]. Therefore, other refractory oxide materials have been developed and utilized [4–6]. For example, magnesium oxide (MgO), calcium oxide (CaO), and aluminum oxide (Al_2O_3) have all been used as refractory materials for titanium casting [7]. The phase transformations observed for the main ingredients of these investments is similar to quartz and cristobalite, but they show less thermal expansion, and are thus unable to compensate for the casting shrinkage of the molten metal. In other words, although the refractory silica shows large thermal expansions, it readily reacts with titanium. On the other hand, if the refractory material does not react with titanium, it shows less thermal expansion.

Whilst the phosphate-bonded investment, Gilvest Ti, is used extensively for titanium casting, its properties of Gilvest Ti are little reported. The aim of this research was to characterize the properties of Gilvest Ti; to compare them with other commercial investment materials and to compare the properties of titanium castings produced in these investments.

2. Experimental

Phosphate-bonded investment, Gilvest Ti (Giulini Chemie, Germany) was used in this study, and compared with Alumina investment, *T*-invest (G-C, Japan) and magnesia investments Selevest D and Selevest DM (Nissin, Japan). The names of the four manufacturers, the main refractories in each, the mixing ratios, and the batch numbers of the investment materials used are shown in Table I.

A 30 g titanium ingot (commercially pure [cp Ti], grade 1) was used for each casting. According to the manufacturer's instructions, castings were made using a titanium casting machine (Ticast Super R, Kobelco,

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TABLE I Investment materials

Investments	Manufacturer	Main refractories	Mixing ratio	Batch no.
Gilvest Ti	Selec	SiO_2	L/P: 0.18	813025988
T-invest	G-C	Al_2O_3	W/P: 0.14	220191
Selevest D	Nissin	MgO	W/P: 0.18	XLS
Selevest DM	Nissin	MgO	W/P: 0.17	YHA

L: liquid; P: powder; W: water.

Japan). All wax patterns were invested with the investment materials listed in Table I.

The thermal and mechanical properties of each investment were measured, including setting expansion, thermal expansion, and compressive strength. The setting expansion of the investments was measured using an expansion measurement machine (Kiyowa Seiki, Japan). Having coated the contact surfaces of the machine with PTFE tape, the investment powders was mixed with water or liquid under vacuum. After 40 s, the mixed paste was poured directly into the measuring device to obtain the setting expansion of the investment.

The thermal expansion was measured by a thermomechanical analyser (Rigaku, thermalflex series 8100). Heating and measurements were made according to the manufacturer's instructions.

Compressive strengths before and after heating were measured using a universal testing machine (Instron 5567), at a cross head speed of 1 mm/min. The cylindrical specimens used for this had a diameter of 20 mm, and a height of 40 mm.

Mechanical properties of Ti castings made in each investment were measured. These included the Vickers hardness, tensile strength, elongation and surface roughness. Vickers hardness was measured from the surface layer to the a depth of 450 μm into the casting at intervals 50 μm, with loads of 0.98 N and load times of 15 s (Akashi micro, model-MVK, Type C). Tensile strengths and elongations were measured using the universal testing machine, with a cross-head speed of 1 mm/min. The specimens for these tests were 18 mm in length and 2 mm in diameter. Surface roughness was analyzed using a surface texture measuring instrument (Surfcom 1400A, Tokyo Seimitsu), with measurement conditions according to JIS-94. This required a measured length of 13 mm, and a cut-off wavelength of 0.1 mm.

3. Results

Fig. 1 shows the setting expansions of Gilvest Ti (phosphate bonded), T-invest (alumina cement), Selevest D and Selevest DM (magnesia bonded). The setting expansions of Gilvest Ti are 0.4%, 0.8%, 2.1% when using concentrations of 50, 75, and 100% of its special liquid respectively, as shown in Fig. 1(a)–(c). Gilvest Ti shows extremely large setting expansion with the mixing liquid. The setting expansion of Gilvest Ti is increased with increasing the concentration of mixing liquid.

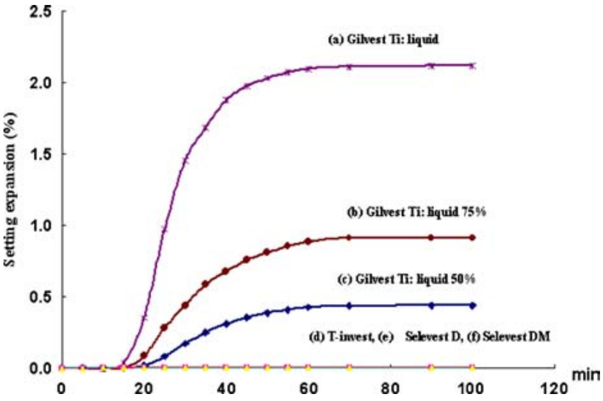

Figure 1 Setting expansions of investments: (a) Gilvest Ti (phosphate bonded) with its own mixing liquid at a concentration of 100%, (b) Gilvest Ti (phosphate bonded) with its own mixing liquid at a concentration of 75%, (c) Gilvest Ti (phosphate bonded) with its own mixing liquid at a concentration of 50%, (d), (e) and (f) T-invest (alumina cement), Selevest D, and DM (magnesia bonded), respectively.

Fig. 2 shows the thermal expansion of the investments. The thermal expansion of Gilvest Ti with a concentration of 50% of its liquid is 0.63% at 1000 °C, T-invest was 1.25% at 1050 °C, Selevest D was 0.42% and Selevest DM was 0.97% at 850 °C as shown in Fig. 2(a)–(d), respectively. In fact, the titanium is usually cast in a low temperature mold. After heating, the casting mold is cooled to 200 °C for casting (from manufacturer's instructions). From Fig. 2(a) and (b), the thermal expansion of Gilvest Ti is −0.25% at 200 °C, and T-invest is 0.6% at 200 °C. It was found that setting expansion is larger than thermal expansion for Gilvest Ti.

Fig. 3 shows the VHN (Vickers hardness) of cast titanium. The hardness is 655 Hv for Gilvest Ti near the surface layer of the casting. After that, the VHN's 444, 315, 206 Hv and 10 at depths of 50, 100, 150, and 200 μm, respectively from surface layer to the interior of casting. The hardness is decreased from the surface to the interior for titanium castings and the hard reaction surface layer is about 200 μm for the casting s produced with Gilvest Ti and Selevest D investments, but 150 μm for T-invest. This means that a hard reaction layer forms on the surface of all castings, but the interdiffusion of Ti with alumina (T-invest) is less than with silica (Gilvest Ti) or magnesia (Selevest).

Table III shows the tensile strength and elongation of castings. The tensile strength of Selevest D of 422 MPa is higher than the other two. The result is possibly due to its lower surface hardness as shown in Fig. 3. It is well known that the higher the hardness the more brittle the metal is and the easier the crack propagation.

4. Discussion

The setting expansion can be controlled by adjusting the concentration of the mixing liquid. The setting expansion of T-invest (alumina cement), Selevest D and Selevest DM (magnesia bonded) are approximately zero as shown in Fig. 1(d)–(f). This result is consistent with the data that the setting expansion of T-invest is 0.01% [8]. The setting expansion of Gilvest Ti (phosphate bonded) is always greater than the other investments

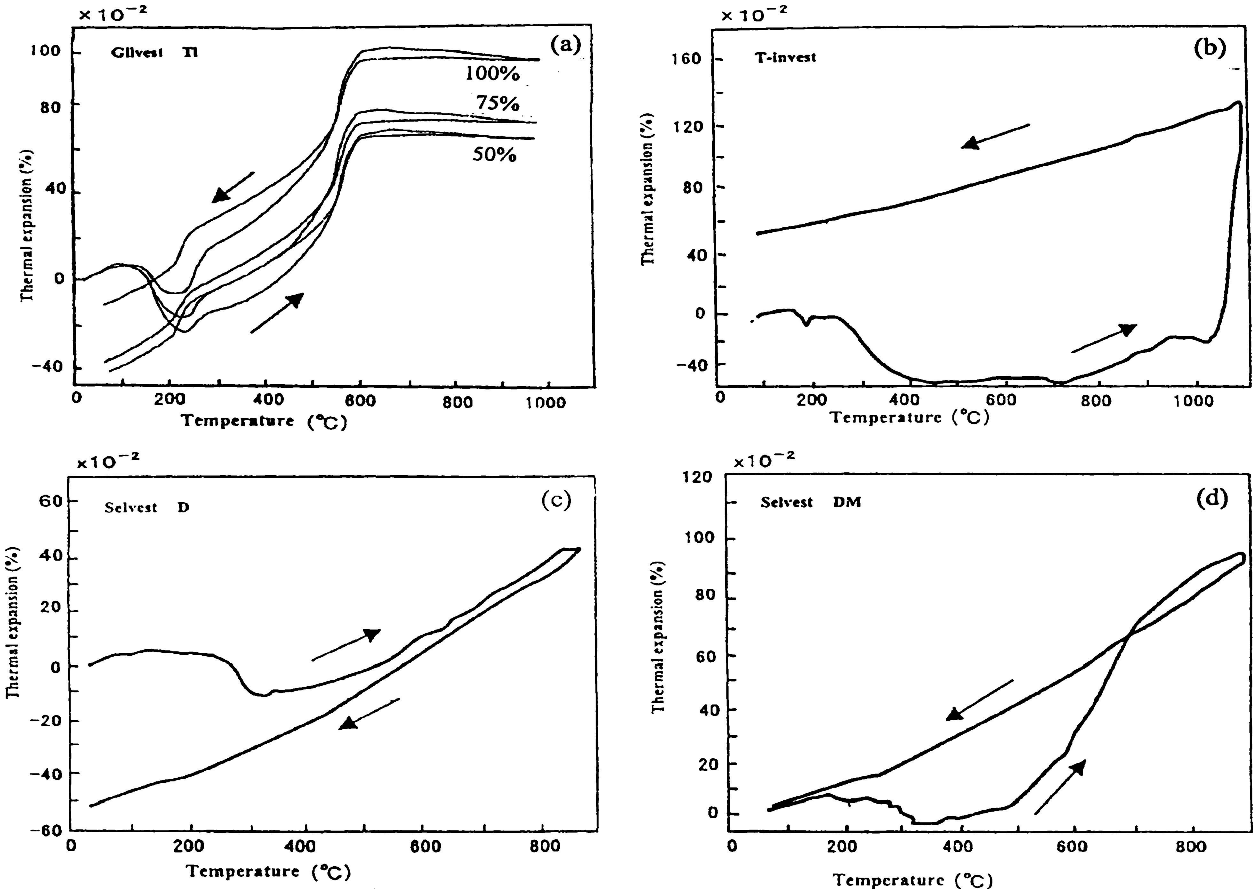

Figure 2 Thermal expansion of investments (a) Gilvest Ti (phosphate bonded) mixed with its own mixing liquid at a concentration of 50%, 75% and 100% (L/P = 0.18), (b) T-invest (alumina cement) mixed with water (*W/P* = 0.14), (c) Selevest D (magnesia bonded) mixed with water (*W/P* = 0.18), and (d) Selevest DM (magnesia bonded) mixed with water (*W/P* = 0.17).

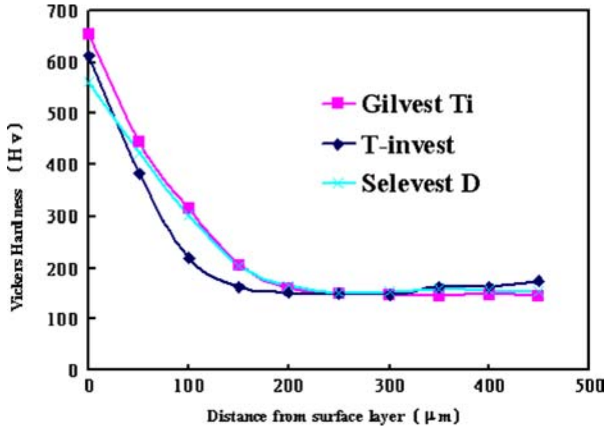

Figure 3 Vickers hardness of cast titanium in cross section. Hardness was measured at depths of 50, 100, 150, and 200 μm from surface layer to the interior of castings.

and can compensate for the cooling shrinkage of titanium castings.

The improvement of dimensional fit between a titanium casting and an abutment was achieved by utilizing the oxidizing expansion of zirconium metal powder mixed in the Selevest dental magnesia investment [9]. Molds, using a spinel investment were developed to cast pure titanium. The spinel content of T-invest in the mold and the remaining expansion were also measured [10]. However, the expansion of spinel is less than that of silica-based investment. The spinel content in such investment is very small. The additives that distribute themselves non-uniformly in the mold will cause poor reproduction. Though investments for titanium casting can compensate for shrinkage, the fit of castings is not guaranteed [11, 12]. In contrast, the thermal expansion of Gilvest Ti is small, but the setting expansion is large, enough to compensate for the shrinkage of titanium castings. Besides, its thermal expansion at $T \geq 600\,^{\circ}\mathrm{C}$ was constant and its heating-cooling cycle was almost reversible as shown in Fig. 2(a). These two results can also reduce the thermal shock and prevent investment cracking.

The compressive strengths of the investments are listed in Table II. The compressive strengths of most of the investments is decreased after heating except for Selevest D. 24 h after mixing, the compressive strength of Gilvest Ti 14 MPa is the highest, After heating, the

TABLE II Compressive strengths (MPa) of various investment materials

Investments	After 24 h	After heating
Gilvest Ti	14.05 ± 2.10^a	4.88 ± 0.68^a
T-invest	7.62 ± 1.14^b	5.02 ± 2.66^a
Selevest D.	9.00 ± 1.04^b	9.90 ± 1.60^b
Selevest DM	6.08 ± 0.83^b	5.52 ± 1.21^a

Values are means SD, $n = 3$. Values in the same column with different superscript letters (a, b) are significantly different at $p < 0.05$ by pair test.

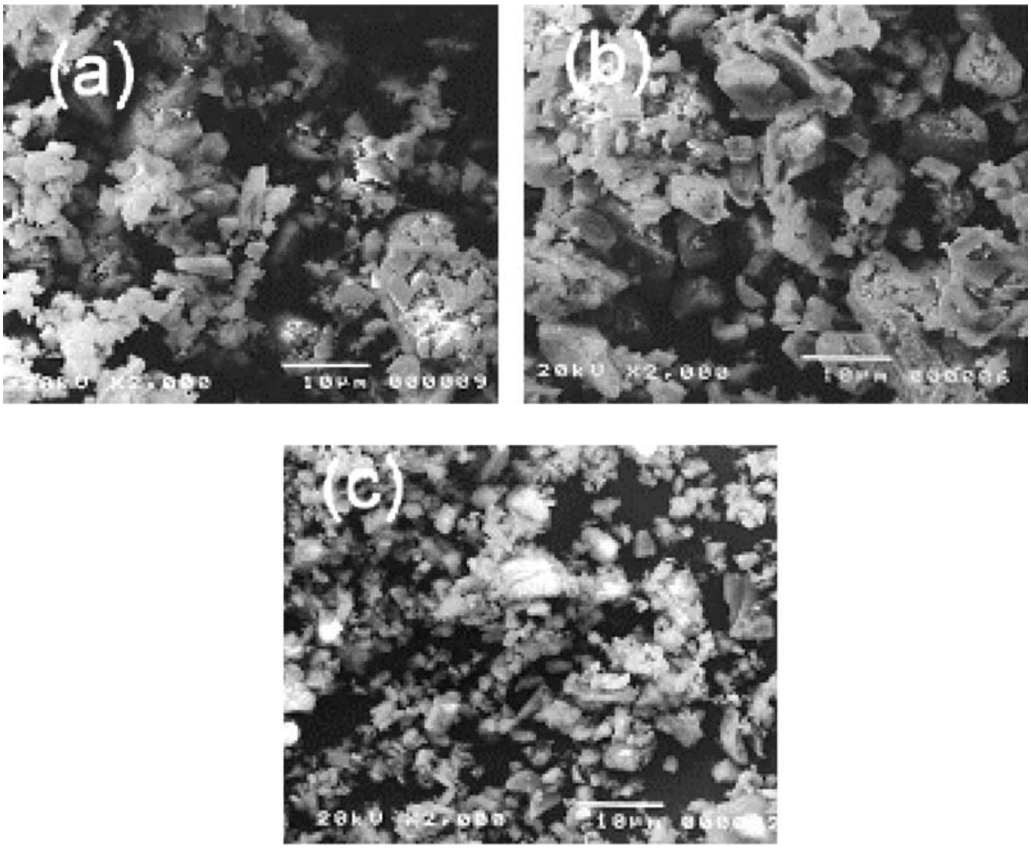

Figure 4 SEM observations of investment surfaces, (a) Gilvest Ti (phosphate bonded), (b) T-invest (alumina cement), and (c) Selevest D (magnesia bonded).

compressive strength of Selevest D, 9.9 MPa, is the highest, whereas the other 3 investments have strengths around 5 MPa.

The compressive strength of the investment should be strong enough to endure the casting pressure of 3 MPa. This is the compressive strength of phosphate-bonded investment (International organization for standardization (ISO) 9694 Dental phosphate-bonded casting investment). The compressive strength of Gilvest Ti falls after heating. However, it still satisfies the regulator requirement of the ISO standard.

Hard reactive zones on the surface of titanium castings are due to molten titanium interdiffusion with the investment. This can decrease physical properties of titanium castings. Furthermore, polishing efficiency will also fall. The thickness of the reaction layer is about 150–200 μm, which is similar to the commercial investments. It is expected that the formation of a reaction layer can be controlled during the development of a new investment.

The surface roughness of a casting made in Gilvest Ti was 2.3 μm (Ra), T-invest was 3.4 μm (Ra), and Selevest D was 1.3 μm (Ra) which was the lowest in the study, as listed in Table IV. The smoother surface will also inhibit crack initiation and delay crack propagation, and this could explain the improvement in tensile strength, as listed in Table III. The lower roughness of Selevest D is due to its small particle size, as shown in Fig. 4(c). On the other hand, the particle size and roughness of Gilvest Ti and T-invest are larger as shown in Fig. 4(a) and (b), respectively.

TABLE III Tensile strength (MPa) and elongation (%) of cast titanium

Used investments	Tensile strength	Elongation
Gilvest Ti	379.4 ± 15.9^{a}	19.4 ± 2.3^{a}
T-invest	371.2 ± 9.5^{a}	20.8 ± 2.8^{a}
Selevest D	422.2 ± 27.4^{b}	19.8 ± 1.7^{a}

Values are means SD, $n = 3$. Values in the same column with different superscript letters (a, b) are significantly different at $p < 0.05$ by pair test.

TABLE IV Surface roughness of cast titanium

Used investments	Ra (μm)	Rz (μm)
Gilvest Ti	2.29 ± 0.05^{a}	8.21 ± 0.68^{a}
T-invest	3.43 ± 0.72^{b}	24.31 ± 2.30^{b}
Selevest D.	1.34 ± 0.09^{a}	6.17 ± 0.38^{a}

Values are means SD, $n = 3$. Values in the same column with different superscript letters (a, b) are significantly different at $p < 0.05$ by pair test.
Ra: Average roughness.
Rz: Maximum roughness.

5. Conclusion

The physical and mechanical properties of the new phosphate bonded investment (Gilvest Ti) and titanium cast against it can be summarized as: The thermal expansion of Gilvest Ti is small, but the setting expansion is large enough to compensate for the shrinkage titanium castings. Also; its thermal expansion at $T \geq$

600 °C was constant and its heating-cooling cycle was almost reversible. These two results can reduce thermal shock and avoid the investment cracking. The surfaces of castings form an α-case reaction layer with phosphate bonded investment. This behavior is similar to commercial alumina and magnesia bonded investments such T-invest and Selevest. The tensile strength of cast titanium was related to surface hardness and roughness. The least hard and rough surface (Selevest D) revealed the highest tensile strength.

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