Dimensional Accuracy and Surface Property of Titanium Casting Using Gypsum-bonded Alumina Investment

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The aim of the present study was to evaluate the dimensional accuracy and surface property of titanium casting obtained using a gypsum-bonded alumina investment. The experimental gypsum-bonded alumina investment with 20 mass% gypsum content mixed with 2 mass% potassium sulfate was used for five cp titanium castings and three Cu-Zn alloy castings. The accuracy, surface roughness (Ra), and reaction layer thickness of these castings were investigated. The accuracy of the castings obtained from the experimental investment ranged from -0.04 to 0.23%, while surface roughness (Ra) ranged from 7.6 to $10.3 \mu m$. A reaction layer of about $150 \mu m$ thickness under the titanium casting surface was observed. These results suggested that the titanium casting obtained using the experimental investment was acceptable. Although the reaction layer was thin, surface roughness should be improved.

Key words: Gypsum-bonded investment, Titanium, Dental casting

INTRODUCTION

The gypsum-bonded investment has numerous advantages such as easy operation, relatively large setting expansion, and adequate green and fired strengths. Therefore, the gypsun-bonded investment is now being widely used for low- and medium-fusing dental casting alloys¹⁻⁵⁾.

In previous papers^{6,7)}, we developed an experimental gypsum-bonded alumina investment for highfusing alloys, and its characteristics were investigated; an alumina investment with 20 mass% gypsum content offered the fundamental properties for high-fusing alloy casting; the investment with only a small amount of magnesia content mixed with 2 mass% potassium sulfate was considered a suitable composition, having adequate setting behavior, sufficient green strength, and sufficient compensate expansion for casting.

In the present study, we evaluated the accuracy of titanium castings using the aforementioned experimental investment. In addition, we measured the surface roughness and reaction layer thickness of the titanium casting obtained using the aforementioned experimental investment.

MATERIALS AND METHODS

Investments

The experimental investment consisted of 20 mass% of alpha hemi-hydrate gypsum for a commercial gypsum-bonded investment (Cristobalite Micro, GC, Tokyo, Japan) as a binder, and alumina powder (Fuji-random wa-220, Fujiseisaku, Tokyo, Japan) and magnesia powder (MgO; RA-F, Tateho Kagaku, Hyogo, Japan) as refractory material. The mass ratio of alumina to magnesia was 6:1 (*i.e.*, containing 6.1 mass% magnesia).

The investment was mixed with a 2 mass% K_2SO_4 solution at a water/powder ratio of 0.28 by hand for 30 seconds and for an additional 30 seconds with a vacuum mixing machine (Vac-U-Mixer, Whip-Mix, Fort Wayns, IN, USA).

As a control, a commercial investment for titanium (Titavest CB, Morita, Kyoto, Japan) was employed and mixed according to the manufacturer's recommended liquid/powder ratio of 0.135 by hand for 10 seconds and for an additional 60 seconds with the vacuum mixing machine.

Dimensional accuracy

An invar-steel mold with a small inserter was employed to fabricate a trapezoid wax pattern (Fig. 1). The dimensions of the wax pattern were 12.7 mm in upper length, 14.0 mm in lower length, 11.0 mm in height, and 2.0 mm in thickness. Distances between the upper line of the wax pattern and the mold without the inserter at the three locations were measured using a profile projector (Model 6C, Nikon, Tokyo, Japan) and recorded as DW.

Sixteen wax patterns were prepared using the above-mentioned mold and divided into two groups of eight wax patterns each. For the first group, the pattern was invested using the experimental investment in an alumina ring, an inner diameter of 42 mm and a height of 55 mm, with a 2-mm thick 540

ceramic liner (Ceramic liner Cyclarc, Morita, Kyoto, Japan) for casting. For the second group, the pattern was invested using Titavest CB investment with a metal ring, a diameter of 40 mm and a height of 55 mm. Before burnout procedures began, the experimental and Titavest CB investments were allowed to bench set at room temperature $(23\pm2\%)$ for 24 and 1.5 hours, respectively.

The mold heating schedules of the two investments are outlined in Table 1. The mold of the experimental investment was heated in a hightemperature SiC furnace (FG41, Yamato Engineering, Tokyo, Japan) from room temperature to 1400 °C, and then cooled to room temperature. In addition, the Titavest CB mold was heated in a conventional burnout furnace and then cooled to cast at 700 °C. Casting procedures were also performed according to the manufacturer's recommendations. Five cp titanium castings (Dental pure titanium A, 10 g, Morita, Kyoto, Japan) and three Cu-Zn practice alloy castings (K metal; Cu 67%, Zn 20%; Toyo, Tokyo, Japan) of each investment were made using an elec-



Fig. 1 An invar-steel mold with an inserter was used to fabricate a trapezoid wax pattern and for measuring dimensional changes of the wax pattern and casting accuracy at three points indicated by black arrows.

| | Table | 1 | Mold | heating | schedule |
|--|-------|---|------|---------|----------|
|--|-------|---|------|---------|----------|

tric arc melting/argon gas and centrifugal pressure casting machine (Vulcan-T, Shofu, Kyoto, Japan) and a conventional centrifugal casting machine (TS3, Degussa, Germany), respectively. The castings were divested of residual investment through sandblasting with 50- μ m alumina powder, and then ultrasonically cleaned in distilled water for two minutes.

The dimensions of the casting after cleaning were also measured using the same procedure for the wax pattern, and recorded as DC. Dimensional accuracy of the casting (D) was calculated by the following formula based on the dimensions of the invarsteel mold:

$$D = \Delta d \times [(14.0 - 12.7) / (11.0 \times 12.7)] \times 100$$

where Δd was the amount of dimensional discrepancy between DW (mm) and DC (mm).

Surface roughness

An arithmetical mean deviation of the surface (Ra) of each casting was carried out using a surface roughness analyzer (Surfcom 50A, Tokyo semitsu, Tokyo, Japan) with a transverse length of 2.0 mm and a cut-off value of 0.8 mm. Three measurements at three locations (Fig. 2) of each casting surface were made, and the mean value of the three measurements was used for calculations. A set of five titanium and three Cu-Zn alloy casting specimens from each of the two investments was examined.

Micro Vickers hardness

Obtained titanium castings were embedded in an acrylic resin. Each specimen was then sectioned at the center of the trapezoidal casting using a low-speed diamond saw with a water coolant. The sectioned surface was consecutively polished with 220-, 400-, 600-, 800-, 1000-, 1200-, and 1500-grit silicone carbide paper under running water.

The micro Vickers hardness (Hv) of measuring points 50 to $1000 \,\mu$ m in depth from the surface of the sectioned casting was measured using a micro Vickers hardness tester (HMV-2000, Shimadzu, Kyoto, Japan) under a load of 100 gf and a duration of 15

| | 0 | | |
|--------------|------------|-----------------------|--------------------|
| | Phase | Heating rate (°C/min) | Holding time (min) |
| Experimental | Heating | | |
| | RT-1200℃ | 10 | 0 |
| | 1200-1400℃ | 5 | 0 |
| | Cooling | | |
| | 1400-600℃ | 10 | 0 |
| | 600°C-RT | 15 | |
| Titavest CB | Heating | | |
| | RT-900℃ | 15 | 50 |
| | Cooling | | |
| | 900-700℃ | 15 | 20 |

RT: Room temperature



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Fig. 2 Titanium casting and measuring points.

seconds. Five titanium casting specimens from both investments were examined.

Statistical analysis

Two-way and three-way ANOVA were used for statistical evaluation of casting accuracy and roughness, respectively. Investments and alloys were selected as the main factors for casting accuracy, while investments, alloys and measuring locations were selected as the main factors for roughness.

Two-way ANOVA and the Tukey-Kramer multiple range test were used for statistical evaluation of surface hardness. Investments and measuring locations were selected as the main factors.

Statistical calculations were performed using statistical software (JMP 5.1, Cary, NC, USA) (p< 0.05).

RESULTS

Dimensional accuracy

The casting accuracies of titanium and Cu-Zn alloy casting are shown in Fig. 3. A negative value "-" indicates an undersized casting, while a positive value "+" indicates an oversized casting. For the experimental investment, the obtained castings were either slightly undersized or slightly oversized. The average accuracies of titanium and Cu-Zn alloy using the experimental investment were -0.04% and 0.23%, respectively; the average accuracies of titanium and Cu-Zn alloy with Titavest CB were 1.56% and 1.28%, respectively. Two-way ANOVA revealed that the accuracy of the experimental investment was significantly better than that of Titavest CB. However, the cast alloy factor was not significant.

Surface roughness

The surface roughness (Ra) of different metallic



Fig. 3 Casting accuracy of titanium and Cu-Zn alloy.



Fig. 4 Surface roughness of titanium and Cu-Zn alloy at each point.

castings from each investment is illustrated in Fig. 4. Three-way ANOVA suggests that the investment factor was significant. However, the other factors, cast alloys and the measuring locations and their interactions were not significant. Surface roughness of casting using the experimental investment was significantly greater than that of Titavest CB.

Micro Vickers hardness

Obtained micro-Vickers hardness of the casting is summarized in Fig. 5; the Vickers hardness at a depth of 50 to $1000 \,\mu$ m from the specimen surface of the experimental investments ranged from 159.2 to 339.6, while those of Titavest CB ranged from 150.2 to 408.8. The Vickers hardness of castings using the experimental investment was significantly smaller than that of Titavest CB. Moreover, Vickers hardness varied with measuring depth. In addition, the interaction between the investment and measuring



Fig. 5 Vickers hardness of cast titanium in cross-section.

depth on hardness was significant. The Vickers hardness values of castings from the experimental investment were not significantly different when deeper than 150 μ m, while those from Titavest CB were not significantly different when deeper than 200 μ m (p> 0.05).

DISCUSSION

With regard to dental castings, a dental investment with minimal setting expansion and high thermal expansion is desirable, because a large setting expansion might cause distortion of the invested wax pattern. The maximum exothermic setting reaction temperature and setting expansion of gypsum-bonded investments are lower and smaller than those of phosphate-bonded investments. However, gypsumbonded investments are not suitable for high-fusing casting alloys because of their low strength and gypsum decomposition above $700\,^\circ\!\!\mathbb{C}$ in the presence of $carbon^{8-10}$. The characteristics of the experimental investment used in the present study were smaller setting expansion, larger thermal expansion, and higher heat resistance compared with those of the commercial gypsum-bonded investment $^{6,7)}$.

The casting shrinkage was estimated based on the casting accuracy and the total expansion of the investment using the following formula:

Estimated casting shrinkage=Setting expansion +Thermal expansion-Casting accuracy

The setting and thermal expansions of this experimental investment were 0.03 and 2.36%, respectively⁷⁷, while those of the Titavest CB investment were 0.0 and 0.6%, respectively¹¹⁾. Therefore, the estimated casting shrinkages of titanium and Cu-Zn alloy obtained from the experimental investment were 2.43% and 2.16%, respectively. The casting shrinkage of titanium in the present study was slightly greater than the previously reported values of 1.8-2.0%^{12,13}, but that of Cu-Zn alloy was similar to a previously reported value of 2.17%¹⁴. On the other hand, the casting shrinkages of cp titanium

and Cu-Zn alloy obtained from commercial investments were -0.96 and -0.68%, respectively. Regarding the titanium casting using Titavest CB, casting accuracy has been reported to range from -0.2 to $1.8\%^{11,15-17}$. The expansion of Titavest CB indicated that several factors affected expansion, such as batch number¹⁸, waiting period after mixing and before heating¹¹, as well as casting liner type and thickness¹⁷⁾. It has been reported that the investment expansion has a great effect on fit of titanium castings^{12,13,18-20}. Moreover, another report²¹ showed that dimensional accuracy of a titanium casting could not be computed nor explained by the abovementioned dental cast theoretical formula which estimates casting shrinkage based on setting expansion, thermal expansion, and casting accuracy. Results of the present study suggest a theoretical problem for casting shrinkage based on titanium casting accuracy and total expansion of the investment. The trapezoid pattern employed in the present study was relatively simple and large compared with actual dental inlays Although the accuracy of castings and crowns. using the experimental investment was significantly better than that from Titavest CB, it is necessary to investigate the fit of crown- and inlay-type castings to simulate clinical treatment.

The surface roughness of titanium castings using Titavest CB in the present study was similar to a previously reported value of $2.5 \,\mu m^{16}$. The surface roughness values of titanium and Cu-Zn alloy castings using the experimental investment were higher than those of Titavest CB. There were two possibilities for this roughness. First, melting titanium might have reacted with the casting mold. In a previous paper⁷⁾, small amounts of Al, Mg, S, Ca, K were detected by x-ray microprobe analysis on titanium castings and were considered as contaminants. However, no data about the effects of Al, Mg, S, Ca, K on surface roughness of castings are available. Secondly, the particle size of the experimental investment was also a factor. Particle size influences surface smoothness of the casing mold, as well as permeability, setting time, and setting expansion of the dental investment. The surface roughness (Ra) of a Cu-Zn casting using an experimental zirconphosphoric acid investment has been reported to be $0.7-1.3 \,\mu m^{24}$. The Cu-Zn alloy did not react with the experimental investment nor the zircon-phosphoric acid investment. These results suggest that the surface of the investment in the present study might be rough. The particle size of alumina powder as the refractory material, with a median diameter of 84.7 μ m, was larger than that of commercial gypsumbonded investments. The different particle size and type of alumina has a significant effect on thermal behavior and characteristics of experimental investment⁶⁾. Therefore, it would be necessary to modify the particle distribution of the refractory material to

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It is well known that a reaction layer is formed on the external titanium surface because of reaction between melting titanium and silica at high temperatures. This layer increases hardness and decreases mechanical properties of titanium. Recently, several non-silica investments for titanium have been developed. There are many reports on the interfacial reaction of pure titanium and titanium alloy with the commercial investment for titanium. Cast titanium reaction layers obtained from newly introduced investments were reduced approximately from 50 to $200 \,\mu$ m. Formation of a 100- μ m reaction layer between titanium and alumina was reported using the x-ray microprobe analysis²³⁾. The thickness of the reaction layer, which was rich in O, Si, Al, Zr, Mg, and P, ranged from 10 to 60 µm with various commercial investments. No correlation was found between the thickness of the reaction layer and hardness. However, hardness at $500 \,\mu$ m from the surface of castings obtained using alumina and zirconia-based investments was greater than that with spinel-based and magnesia-based investments²⁴⁾. In the present study, the refractory oxides employed were alumina, magnesia, and calcia, which are relatively inert to melting titanium even at high temperatures. The reaction layer of titanium casting using this experimental investment was as thin as those obtained using commercial investments for titanium when similar oxides were contained as refractory materials^{11,24)}. The alumina content of the experimental investment was assumed to be less than that of Titavest CB. Moreover, the mold temperature of the casting using Titavest CB was 700°C, while that of the experimental investment was at room temperature. Meng Y et al.²⁵⁾ reported that the thickness of the hardened surface layer of castings made from a room temperature mold was lower than that made from a high temperature mold. Hence, these factors might result in different reaction layer thicknesses between the two investments.

CONCLUSIONS

The accuracies of the titanium and Cu-Zn alloy castings obtained from the experimental investment ranged from -0.04 to 0.23%. Regarding surface properties, the surface roughness (Ra) of castings ranged from 7.6 to $10.3\,\mu$ m. The thickness of the reaction layer of titanium casting was about $150\,\mu$ m from the surface.

These results suggest that the accuracy of titanium castings using this experimental gypsumbonded investment for high-fusing casting was acceptable. The experimental investment produced a thinner reaction layer than the commercial investment for titanium casting. However, surface roughness should be improved.

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