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The possibility of recasting of pure titanium

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Abstract *Background/purpose:* Pure titanium (Ti) has many advantages, such as high corrosion resistance and excellent biocompatibility. The mechanical properties of pure Ti are like those of type IV gold alloys. Furthermore, gold alloys can be successfully recast in dental clinics. The aim of this study was to investigate the possibility of recasting pure Ti.
Materials and methods: Magnesium oxide (MgO)-based investment that contained a 5 wt. % zirconium dioxide (ZrO_2) additive was used. An argon-casting machine (Castmatic-S, Iwatani) was used to recast pure Ti. The first generation and second generation pure Ti (50 wt. % new Ti + 50 wt. % surplus Ti) were used. Five specimens were fabricated and tested. The data were evaluated using two-sample t-test analysis ($P < 0.05$).
Results: The experimental results showed that recasting the Ti did not decrease the marginal accuracy, average surface roughness, Vickers hardness value of the superficial surface, and the thickness of the reaction layer.
Conclusion: This study clearly showed Ti could be recast when a 5 wt. % ZrO_2 additive MgO-based investment was used. This modified investment has the potential for use in clinical applications.
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Introduction

For centuries, alloys with a high content of noble metals including gold, palladium, and platinum have been used to fabricate fixed prostheses because of the resulting high quality, performance, and ability to meet safety standards. However, the escalating cost of gold has contributed to the widespread use of base metal alloys, such as Co–Cr, Ni–Cr, Ti, and Ti-alloy. Among these materials, pure titanium (Ti) has adequate mechanical properties, high corrosion resistance, and excellent biocompatibility.[1] Therefore, pure Ti has been extensively used in medical and dental applications, such as implants.

In implant prostheses, late failure has been linked to infections, incorrect occlusal loadings, and material incompatibilities. Popular dental implant components are conventionally made of pure Ti only. However, the suprastructures can be made of several alloys, such as Au–Pd, Ni–Cr, Co–Cr, and Pd–Ag. The use of a combination of different types of metals or alloys in the oral cavity can lead to galvanic corrosion and can subsequently induce implant failure. Therefore, the use of a single restorative and biocompatible material, pure Ti, is recommended to avoid galvanic corrosion.[2] Precise dental restoration requires a casting system to replicate complex shapes with high fidelity. In addition, the marginal accuracy of dental prostheses is usually better with a traditional casting technique than with computer-aided design and computer-aided manufacturing fabrication.[3] However, pure Ti can easily react with oxygen at high casting temperatures or a reaction could occur during the casting process. Ti ingots may accumulate dental plaque, which leads to poor marginal accuracy in the restoration and results in secondary caries. Therefore, the role of pure Ti in dental prostheses has increased in importance, and several studies have shown that the accuracy and quality of pure Ti castings have been improved with the use of new casting machines and mold materials.[4–11]

Commercially available silica investments can easily be reacted with Ti to produce a very thick reaction layer for a Ti casting. Therefore, alumina (Al_2O_3), magnesia (MgO), calcia (CaO), zirconia(ZrO_2), and yttria (Y_2O_3) have recently and frequently been used as an oxide inhibitor in Ti casting.[4–11] One application of these compounds is a magnesium oxide (MgO)-based investment, which can significantly reduce the interfacial reactivity and is easy to manipulate.[1,4] However, the disadvantage of MgO-based investments is the low thermal expansion rate that might not correspond to the thermal properties of Ti ingots, making it difficult to obtain a high-precision Ti casting due to the marginal discrepancy. Hung et al[7] added 5.0 wt. % zirconia to a MgO-based investment; this modification was able to increase the thermal expansion and solve the problem of marginal discrepancy. In addition, the interfacial reactivity of the castings made from the zirconia/MgO-based investment was less than that of the MgO-based investment even at high casting temperatures.

In dental laboratories, surplus alloys from the initial casting are commonly reused for economic reasons and to avoid the exploitation of natural resources. Several investigations have shown the effect of recasting within different dental alloys. For example, recasting a low-Au alloy obviously affected the basic properties, such as the yield strength, elemental distribution, hardness, and percentage elongation.[12] Furthermore, the zone of Ag_2O oxidation could be increased by recasting a Pd-Ag porcelain alloy; this recasting led to increased hardness and corrodibility.[13,14] In contrast, recasting Ni-Cr alloy did not lead to remarkable degenerative changes in the physical properties, microstructure, and clinical characteristics.[15] Recasting different alloys through investments that had different components led to varied properties. Therefore, the effect of the recasting pure Ti using a 5.0 wt. % zirconia/MgO-based investment must still be investigated. Consequently, whether Ti recastings can be produced with high quality and high precision is unclear. This investigation evaluates a Ti recasting that uses a new investment mold and characterizes the effect of the Ti recasting on the marginal precision.

Materials and methods

Preparation of investment

The MgO-based investment Selevest CB (Selec Co., Osaka, Japan) was used as the control group. The investment was further modified by the addition of 5.0 wt. % zirconia (ZrO_2, Hayashi Pure Chemical Industries, Japan), and the modified investment was used as the experimental group. The powders for the modified investment were blended in a mechanical vacuum mixer (Whip Mix, Louisville, Kentucky, USA) before mixing with water. The recommended liquid/powder (L/P) ratio for both groups was 20 mL/100 g.

Preparation of castings

The mesial-occlusal-distal (MOD) metal die cast molding was used to assess the casting inlay accuracy. This method is schematically shown in Figure 1. In brief, a wax pattern (8.0 mm in diameter, 7.0 mm in height, and 2.0 mm in thickness) was placed on the metal die. The distance separating the metal die shoulder from the margin of the wax pattern was measured at four fixed points for each specimen, set as dx, and calculated. Five specimens were fabricated and tested for each group. The specimens were invested in casting rings (36 mm in diameter and 46 mm in height) with a layer of ring liner (35 mm in diameter and 2 mm in thickness, J. Morita Co., Kyoto, Japan) and a sprue (3 mm in diameter and 25 mm in length). The invested molds were burned out in a programmed electric furnace at a rate of 6°C/min to 850°C and were then kept at this temperature for 1 hour. After 1 hour of heat soaking, the invested molds were bench cooled to 800°C.

An automatic argon-arc vacuum-pressure casting machine (Castmatic-S, Iwatani Co., Osaka, Japan) and commercial pure Ti ingots (JIS Grade 2, Ohara Co., Osaka, Japan; about 7 g, >99.5%) were used in this research. In the first generation, all new Ti ingots were placed on the copper crucible, and a tungsten electrode was placed 5 mm above the ingot center. The argon pressure was 1.8 kgf/cm^2 with 250 A of current. After casting, the molds were bench
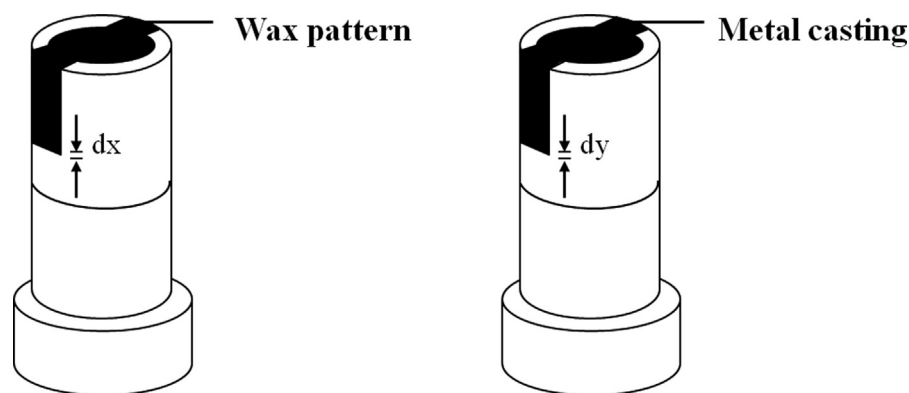
Figure 1 The dimensional accuracy of a cast crown is expressed by the discrepancy (dy − dx) measured between its wax pattern and the casting on the same die.

cooled to room temperature. The castings were removed from the molds and cleaned in water using an ultrasonic cleaner. The sprues and buttons that were sectioned from each casting in the first generation were sandblasted with 50 μm aluminum oxide and ultrasonically cleaned with distilled water for 10 minutes; these sections were considered the surplus. In the second generation, 50 wt. % new Ti was combined with 50 wt. % surplus as the ingot. Then, the recasting method proceeded as described above.

Marginal discrepancy measurement of castings

The irregularities and nodules of the castings were examined using Occlude (Pascal Co., USA) to eliminate defects before verifying the dimensions. The distance separating the margin of the castings and the metal die was set as dy and was measured at the same four points. The inlay was under a load from a 5-kgf mass when the castings were seated. The marginal discrepancy was calculated as dy − dx for each point, and the mean value was determined from the four measurements for each crown. Two-sample t-tests were used to analyze the influence of the generation on the marginal accuracy of the casting.

Surface roughness measurement of castings

The surface roughness was measured on the occlusal surface of the MOD casting. Four repetitive measurements of each casting were performed. The average roughness (Ra) was measured with a surface tester (Surface SJ-301, Mitutoyo Co., Aurora, Illinois, USA), with a sampling length (λc) of 0.8 mm at 0.5 mm/sec.

Microhardness measurement of castings

Sprues of the MOD castings were cut 3 mm from the inlay attachment point. The exposed cross-section was mounted in epoxy resin and subsequently polished with a 600-mesh emery paper with 0.05-μm alumina particles on a cloth-polishing wheel. The Vickers hardness values (VHN) of these specimens was measured using the cross-section of the outer surface at 40-μm intervals using a Vickers microhardness indenter at a load of 200 gf (MTX-50, Matsuzawa Seiki Co., Akita, Japan).

X-ray diffraction evaluation of castings

The chemical reactions of the occlusal outer surfaces of the MOD Ti castings were evaluated using X-ray diffraction (XRD) analysis (X-ray diffractometer D5000; Siemens, Munich, Germany) with CuKα radiation at 30 kV and 20 mA and a scan range of 2θ (20°−80°). Phase identification was performed using the Joint Committee on Powder Diffraction Standards file.

Results

Marginal discrepancy

Unsuccessful castings include any of the following: incomplete castings, rounded short margins, and undesirable porosity. The success rates for complete casting were 100% in both generations. The mean marginal discrepancy values of the MOD inlays are shown in Figure 2. The mean marginal discrepancy value was 55.48 ± 12.12 μm in the first generation and 48.33 ± 7.82 μm in the second generation. Although the mean discrepancy value in the first generation was larger, there was no statistically significant difference between the two groups (two-sample t-test, P > 0.05).

Surface roughness

The measurement of Ra is shown in Figure 3. The Ra values in the first and second generations were 3.60 ± 0.13 μm and 3.62 ± 0.29 μm, respectively. Although the recasting group showed a rougher casting surface, the recasting did not significantly influence the Ra (two-sample t-test, P > 0.05)
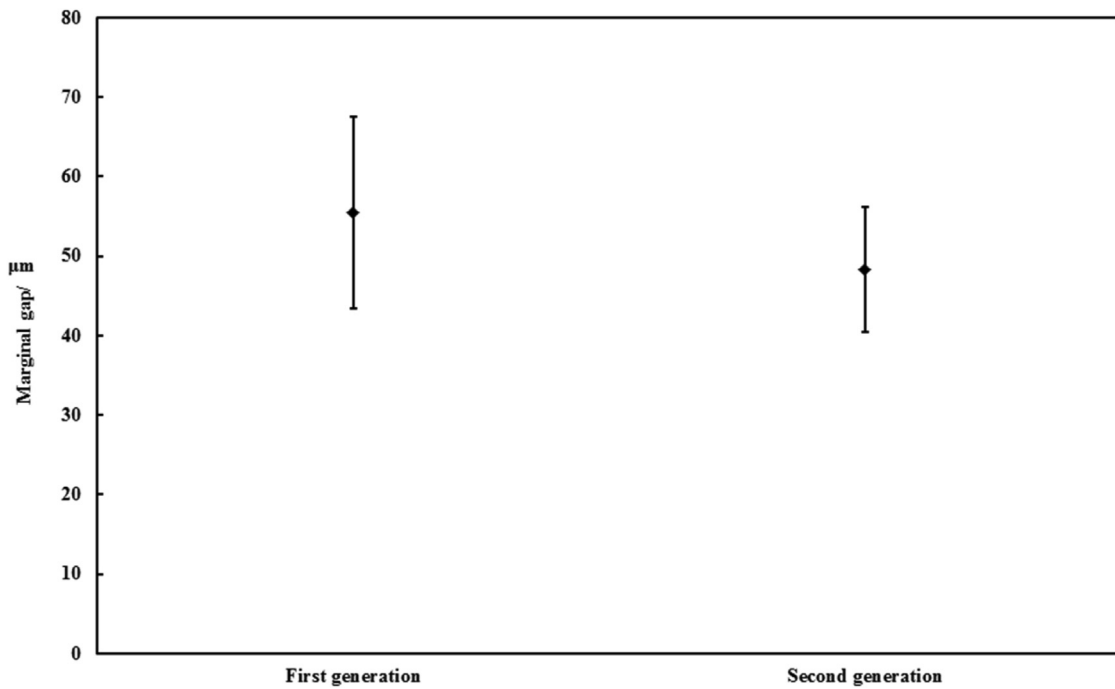
Figure 2 Dimensional accuracy of titanium castings for zirconia/MgO-based investment in both generations. Each column gives the mean and standard deviation.
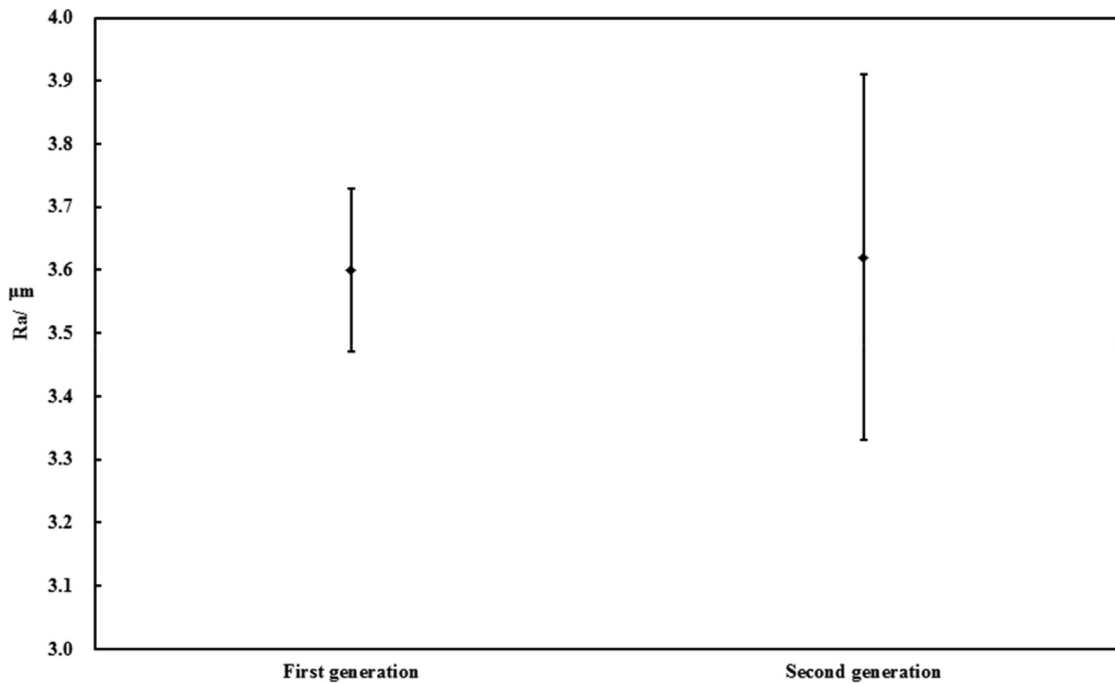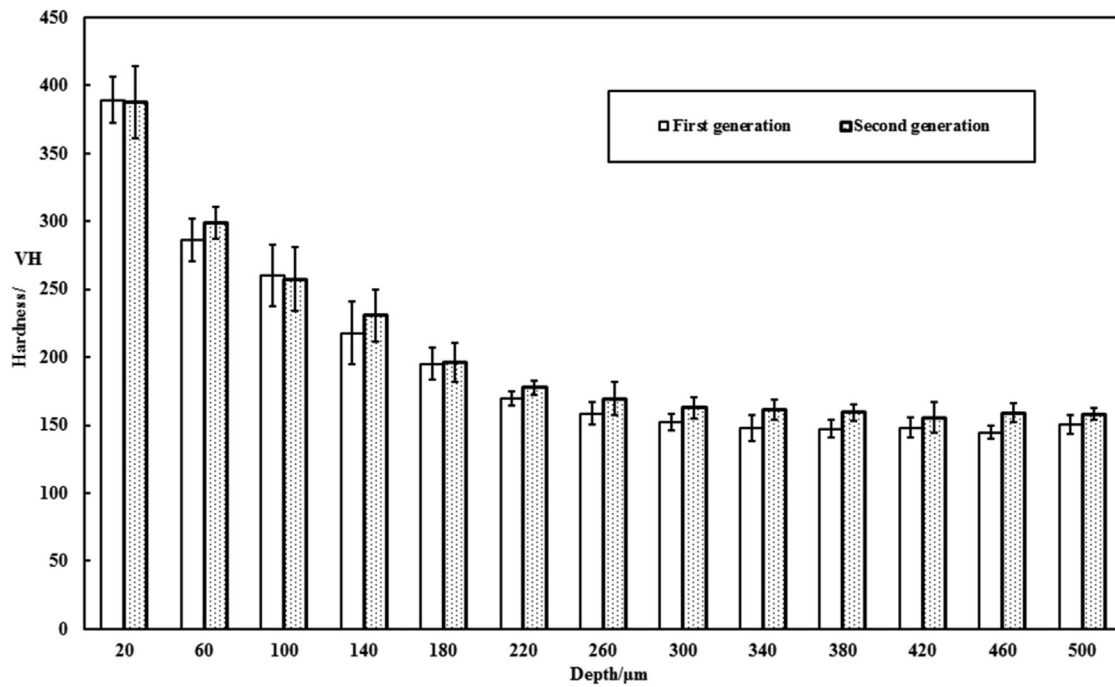

Figure 3 Average surface roughness of titanium castings for zirconia/MgO-based investment in both generations. Each column gives the mean and standard deviation.

Microhardness

The changes in the VHN of the Ti castings from the outer to the inner surface are shown in Figure 4. A gross observation revealed that the superficial surface of the casting at 20 μm had the largest VHN in both the generations. The VHN decreased from the outer surface to the inner surface. For distances ranging from 180 μm to 500 μm from the surface, the VHN did not obviously change. The VHN was slightly lower at each depth in the first generation than at the corresponding depth in the second generation. However, the differences between the two generations were only

Figure 4   Microhardness of titanium castings at every 40 μm for zirconia/MgO-based investment in both generations from the outer to the inner surface. Each column gives the mean and standard deviation.

significant at 220 μm, 300 μm, 340 μm, 380 μm, and 460 μm (two-sample t-test, $P < 0.05$).

XRD analysis

The XRD analysis is shown in Figure 5. Only α-Ti (2θ: 35.4, 38.2, 40.4, 53.2, 70.2), TiO_2 (2θ: 26.1, 44.6, 62.5, 64.1), and TiO (2θ: 36.8, 42.9, 77.1) were detected. The relative

intensities of α-Ti and TiO_2 increased in the first generation. Furthermore, the relative intensities of the TiO phases were more obvious in the second generation.

Discussion

The surface treatments cannot uniformly remove the oxidation layer, and inconsistencies in the oxidation layer
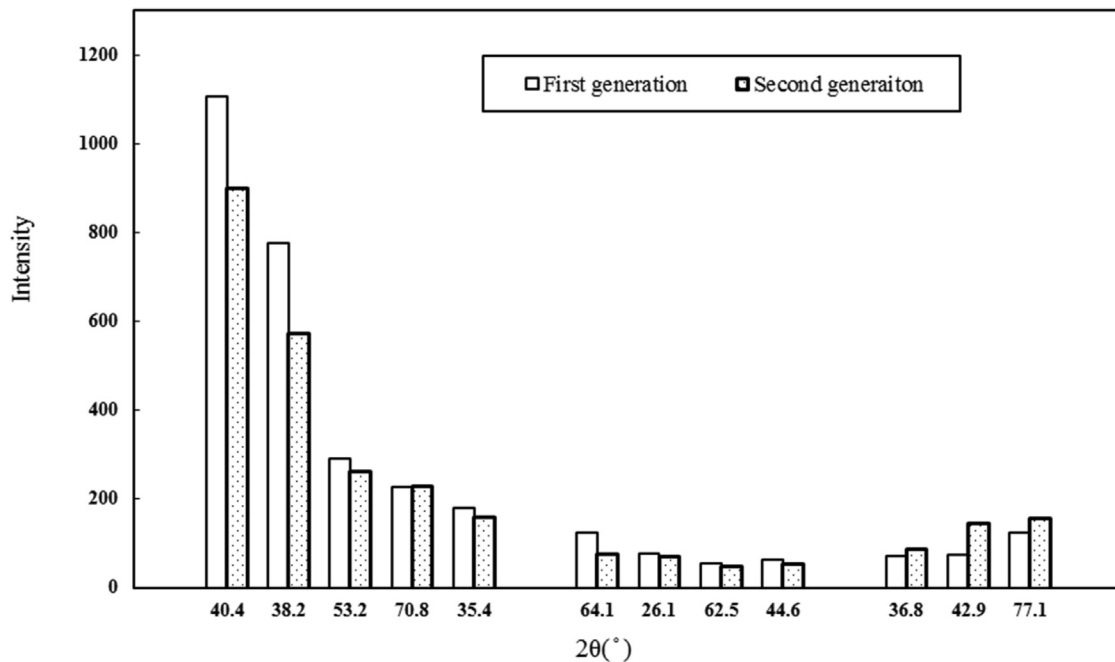
Figure 5   X-ray diffraction patterns of titanium castings for zirconia/MgO-based investment in both generations.

may be caused by the accumulation of oxides during the recasting of alloys. One strategy used to reduce the effect of oxidation on recasting is to use fresh alloys in joints during recasting.[13—15]

Dental prostheses usually require high accuracy in the marginal fitness to avoid secondary caries, and such high accuracy in the marginal fitness usually prevents the late failures caused by infection. In this study, the marginal gap was smaller in the second generation than in the first generation. The average marginal gaps were 55.48 μm in the first generation and declined to 48.33 μm in the second generation. This phenomenon might result from the more homogeneous distribution in the molten metal that is generated during the recasting process.

Christensen[16] suggested that the marginal accuracy of the crown should not be greater than 50 μm. In this study, the mean value of the marginal accuracy of the Ti casting is slightly greater than 50 μm in the first generation; however, over 60% of the Ti casting specimens could still fall within this criterion. Most of the castings in the second generation met this requirement. The marginal accuracy of the recastings was more stable, and the recastings can be used clinically with more confidence. In addition, the surface roughness of a dental casting is also an important factor that can potentially affect the marginal fit and the time required for finishing and polishing.[17,18] The surface roughness of a dental casting is usually affected by factors such as the type of alloy, mold material, casting temperature, and casting machine.[19—23] Chan et al[24] investigated Ti castings that were made using a silica-based investment and a centrifugal casting machine. The researchers found that the surface roughness of the Ti casting reached 12.52 μm to 15.76 μm. In this study, a zirconia/MgO-based investment combined with an argon-arc vacuum-pressure casting machine could decrease the surface roughness of the Ti casting. A mean roughness of 3.47 μm was measured and differed from the value for a casting using an Au alloy.[25] Although the casting in the second generation had a rougher surface than that in the first generation, the Ra was not significantly different between the two generations (two-sample t-test, P > 0.05). Thus, recasting pure Ti did not worsen the surface texture.

The reaction layer often influences the strength of the bonding between pure Ti and porcelain. Miyakawa et al[5] called this reaction layer an "α-case", which results from the reaction between the molten Ti and the investment. A high-surface reaction results in a thick layer of α-case and a high VHN. In the present study, the VHN at the superficial surface of the casting was less than 400 in both generations. Kikuchi et al[26] evaluated the VHN of a Ti casting using MgO-based investment mold at different temperatures, and they found that the VHN increased with casting temperature. The microhardness value of the Ti casting reached 531 VHN at 600°C and only 473 VHN at the ultra-low temperature of −196°C. In the present study, the hardness was less than 400 VHN even in the recasting generation at a 750°C casting temperature. At depths that were less than 180 μm, the VHN was stable at approximately 140—180 μm in the generations. Although the hardness values were slightly smaller at each depth in the first generation for depths in the 20—180 μm range, there were no significant differences between the two generations (two-sample t-test, P > 0.05).

However, at the depths of 220 μm, 300 μm, 340 μm, 380 μm, and 460 μm, the first generation had significantly smaller VHN than those in the second generation (two-sample t-test, P < 0.05). Recasting did not affect the VHN at the superficial surface but influenced the inner surface. Before the recasting of the Ti, the surplus was sandblasted to remove the reaction layer at the superficial layer; thus, the VHN at the interface should only be influenced by the mold material. However, the interstitial elements that are dissolved at the interface might be not removed. These residual elements further influence the VHN at the interface during the recasting procedure.

The XRD analysis showed that α-Ti was the main component of the Ti casting even in the recasting group. Pure Ti in an α-Ti lattice has mechanical properties that are like those of type II and IV dental Au alloys. MgO, Li_2TiO_3, and/or $Li_2Ti_3O_7$ were formed through reactions with the metal and the constituents in the MgO-based investment when heating to temperatures greater than 600°C.[27] The only detected oxides were TiO_2 and TiO in both casting generations in this study because zirconia was able to stabilize the oxygen and prevent the production of metal oxides. The results of this study showed the Ti could be recast when a zirconia/MgO-based investment was used. The marginal accuracy, surface roughness, interfacial surface reaction, and elemental composition of the resulting Ti recasting were comparable with those of the first-generation casting group.

Conflicts of interest

The authors have no conflicts of interest relevant to this article.

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