Grindability of alpha-case formed on cast titanium

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The hardened alpha-case (α -case) layer inevitably forms on the surface of titanium castings when prepared by investment casting. Because the hardness of the α -case is incomparable to that of the interior structure, the perception exists that the α -case is difficult to remove during cutting, grinding and polishing. Grindability (ease of grinding) of cast cpTi and cast Ti-6Al-4V was evaluated by grinding cast specimens incrementally using a SiC abrasive wheel. The present study revealed that the presence of the brittle α -case with lower fracture toughness is beneficial in grinding titanium. The α -case on the ductile cpTi can be ground much easier than its bulk interior structure. In less ductile Ti-6Al-4V, the grinding rate is much higher than that of cpTi, and the α -case and its interior structure are at similar levels since the fracture toughness of its α -case and the bulk material is not large enough.

Keywords: Titanium, Grindability, α -case

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INTRODUCTION

Dental prostheses are generally fabricated using the lost-wax investment casting method. After casting, there are structures attached to the cast metal such as metal-filled runners, sprues, rims and fins, etc. These attachments must be removed by grinding or cutting in order to complete dental prostheses. The efficiency of the manufacturing process is directly related to the cost of the appliance; thus, how easily and quickly castings can be finished is important from the viewpoint of cost effectiveness¹. Unlike conventional dental casting alloys, the grindability and machinability of titanium are generally considered to be poor due to some of the inherent properties of this metal².

Another notable difference from castings made by conventional dental casting alloys is that cast titanium prepared by an investment casting method generally contains a surface layer of α titanium grains, commonly referred to as an alpha-case (α -case). These grains in the α -case are enriched with oxygen and, depending on the chemical composition of the investment materials, include some metallic elements³⁻¹⁰. The α -case is formed as a result of the reaction of liquid titanium with the atmosphere (mainly a minute amount of oxygen) in the melting and casting chamber and, in addition, the interaction with the mold materials². Even when refractories having thermodynamic stability similar to those of the titanium oxide family are used (such as Al₂O₃ and ZrO₂), a strong reducing power of titanium decomposes such common oxides^{11,12}. Since the temperature of molten titanium is very high (Melting Point: 1670°C), the oxygen readily liberated from the oxides diffuses into the surface of the casting to form the α -case. The thickness of the α -case depends on experimental factors such as cooling rate, casting geometry, amount of oxygen at the mold/casting interface, extent of the reaction with the mold material, etc. Alpha-case thickness in the range of 50–500 μ m

has been reported for titanium castings¹³. Such α -case hardened with interstitials (oxygen) and substitutional elements (Al and Zr, etc.) has drawbacks including unnecessary hardness and brittleness and also a reduced ductility and fatigue resistance¹⁴. Because the hardness of the α -case is not comparable to that of the interior structure, researchers perceive that the existence of the α -case hinders the polishing and grinding of titanium dental prostheses made by casting^{15,16}.

Studies of α -case formation in dental titanium castings were reviewed extensively elsewhere^{11,17,18}. These include metallurgical investigations of the α -case microstructures³⁻⁸, theoretical studies regarding the α -case thickness and casting geometry^{18,19}, and the effect of the α -case in tensile testing²⁰, fatigue resistance and tensile ductility².

In dental practice, cast dental prostheses are completed by mechanical procedures of cutting, grinding and polishing after initial sandblasting and chemical milling. Thus, several research groups have investigated the machinability/grindability [the relative ease of removing metal²¹] of cast dental titanium^{16,22-25}. One of our research efforts has been concentrated on finding titanium alloys with improved grindability for the benefit of effective processing of titanium castings^{18,26-31}. The majority of our studies, just like investigations at other institutions, were performed for specimens whose α -case layer was totally removed. To the best of our knowledge, only four investigations evaluated the grindability of titanium alloy specimens with the α -case intact and, at the same time, with the α -case removed^{15,28,29,32}. In these studies, grindability was evaluated as the volume removed from cast specimens after grinding them for a set time period (60 s) using a SiC abrasive wheel at two circumferential speeds (500 and 1250 m/60 s) by applying a constant force of 100 g (0.98 N). Three studies by Koike *et al.* on Ti-Cr-Cu alloys²⁸, Ti-Fe-O-N alloys³³, and Ti-Al-Cu

alloys³²⁾ revealed that the majority of the specimens with the α -case layer were ground significantly greater than specimens from which the α -case was removed, particularly when the higher rotational speed of the wheel was used. These results suggested that grinding the α -case probably is not troublesome, contrary to earlier reports^{15,16)}. Based on our considerable experience in the evaluation of the grindability of titanium alloys, we theorized that the grindability of titanium alloys was enhanced³⁰⁾ when titanium alloys have microstructures that increase brittleness, and reduce tensile ductility, fracture toughness, and the resistance to crack initiation and propagation. The α -case usually has a Vicker's microhardness number ranging from 500 to 600 as compared to that of more ductile interior structures ranging from 200 to 300³³⁾. Thus, it is not surprising that the rate of removal of the α -case is faster than the rate of grinding of the interior structure of specimens with a greater ductility. Even though some of our previous studies have used specimens with the α -case intact, we have not employed an incremented grinding procedure to investigate how the grinding of the α -case per se was different from that in the interior structure, since we evaluated the amount of metals ground after one set of experimental grinding time using continuous grinding without stopping the experiment in the transition region between the α -case and the interior structure. To the best of our knowledge, no studies have investigated the grindability of the α -case itself.

Thus, in the present study, the objective was to estimate separately the ease of grinding of the α -case layer and the interior structures of cast titanium using commercially pure titanium (cpTi) and Ti-6Al-4V as specimens by incrementally grinding the α -case covered specimens as a function of time.

MATERIALS AND METHODS

Metals tested

Commercially pure titanium (cpTi: ASTM Grade 2, Titanium Industries, TX, USA) and Ti-6Al-4V (Ti64: ASTM Grade 5, Titanium Industries) were used. Metal rods of both titanium alloys were machined into discs of 30 g with 10.4 mm thickness and 29.0 mm diameter.

Specimen preparation

Plate wax patterns measuring 3.0 mm × 8.0 mm × 30.0 mm were used for the preparation of specimens by casting. For each casting the two patterns were invested in one mold-ring with a magnesia-based material (Selevest CV, Selec Co. Ltd., Osaka, Japan). The molds were burned out according to the conditions specified by the manufacturer. After holding the mold at 350°C for one hour, the mold was placed in a dental titanium casting machine (Ticast Super R, Selec Co. Ltd., Osaka, Japan). With the manufacturer's suggested operating conditions for the casting machine, the cpTi or Ti-6Al-4V disc was melted in an argon atmosphere and then cast into the mold. A total of two castings

Fig. 1 A schematic drawing of a typical microhardness profile near the surface of cast titanium, indicating the present method of estimating the α -case thickness.

Fig. 2 Schematic diagram of the grindability test machine.

were made for each metal, preparing four cast specimens for each metal.

Evaluation of internal porosity

After the castings were made, their internal porosity was examined with a conventional dental X-ray unit (SIEMENS 5938303, SIEMENS Corp., Munich, Germany) using the following conditions: target distance, 420 mm; tube voltage, 70 kVp; tube current, 7 mA; and exposure time, 1 s. Earlier, the minimum pore diameter detectable with this condition was found to be 0.05 mm³⁴⁾. Specimens with noticeable pores of this diameter range that existed near the grinding areas were excluded from further testing.

Measurements of Vickers microhardness (HV)

The Vickers hardness near the cast surface was determined starting at 25 μ m and continuing to 500 μ m below the surface using a microhardness tester (FM-7, Future Tech, Tokyo, Japan) with a 0.98 N load

and a 15 s dwell time. The microhardness of four randomly chosen locations at each depth was determined on two cast specimens of each alloy.

Determination of the thickness of the α -case

The thicknesses of the α -case for cast specimens were determined from the microhardness profiles obtained as the distance from the cast surface where the decreasing microhardness coincides with that of the bulk interior structure (see A in Figure 1).

Grindability test

A detailed description of testing grindability is found in a previously published article¹⁷. Briefly, a silicon carbide (SiC) wheel (703-120, Brasseler USA, GA, USA; 13 mm diameter, 1.5 mm thick) attached to an electrical dental handpiece (Upower Model 501, Brasseler) was placed against the specimen so that the circumferential surface of the wheel contacts one of the side walls of the rectangular specimen at 90°. A schematic diagram of the apparatus used for grinding specimens is shown in Figure 2. One of the 30.0 mm $\times$ 3.0 mm surfaces of the specimens was ground at the circumferential speed of 20.8 m/s (1250 m/60 s) with an applied force of 0.98 N for the time period of 5, 10, 15, 30, 60, 70 or 80 s. Grinding areas in each grinding were chosen carefully so that each grinding area was at least 1.0 mm apart and did not overlap each other. The volume of metal removed (mm^3) was estimated from the weight loss of the specimen determined previously using the density of each metal²⁹. Two areas were ground for each grinding time in each specimen. Four cast specimens were examined for each metal. The chips formed during the metal grinding were collected and examined using a scanning electron microscope (SEM: JSM-6300, JEOL, Tokyo, Japan).

Microstructure near the ground surfaces

A scanning electron microscope (SEM) was used to take micrographs near ground areas on the cross-sectioned specimen surfaces after appropriate chemical etching³⁵. By observing grooved areas after each grinding time-period, 5 through 80 s, the ground depth was measured

Fig. 3 Hardness profile for cast cpTi and Ti-6Al-4V.

as the distance between the original cast surface and the bottom area in the groove. For each groove, the distances were obtained from three randomly chosen bottom areas. Two cast specimens were examined for each metal. In order to detect metal debris adhered on the grinding surface of the SiC wheels, backscattered electron images of SEM for some selected areas were obtained. These images were compared with secondary electron images for each corresponding area to clearly identify adhered metal pieces.

Data Analysis

The amounts ground as a function of time are statistically analyzed by one-way ANOVA with the Tukey's test ($\alpha=0.05$).

RESULTS

Typical microhardness profiles from the cast surfaces for cpTi and Ti-6Al-4V are shown in Figure 3. The profiles for cpTi and Ti-6Al-4V exhibit hardness values similar to each other near the cast surfaces. Then, the

Fig. 4 Metals removed as a function of grinding time for cast cpTi and Ti-6Al-4V.

Fig. 5 Depth ground from the original cast surface as a function of grinding time. For cast cpTi and Ti-6Al-4V.

hardness values decrease with increasing distance towards the interior of the casting and eventually reach a constant minimum value which is the bulk, interior hardness. The α -case thickness, where the hardness value reaches the minimum value in the interior structure (Figure 1), was found to be 220 μm for cpTi and 114 μm for Ti-6Al-4V. Figure 4 is the plot of the

means of the amount of metals removed (volume loss, mm^3) by grinding for cpTi and Ti-6Al-4V as a function of grinding times. These two metals exhibit specific grinding characteristics that should be noted. At each grinding time, the volume losses in Ti-6Al-4V were significantly higher. The grinding rate (the slope in Figure 4) in Ti-6Al-4V, compared to that of cpTi

Fig. 6 SEM micrographs near ground areas taken from cross-sectioned cpTi and Ti-6Al-4V specimens.

Fig. 7 SEM micrographs of the metal chips from grinding after 5 s and 80 s of cpTi and Ti-6Al-4V.

Fig. 8 Backscattered electron images (a) and Secondary electron images (b) of the surfaces of the SiC wheels used in grinding cpTi and Ti-6Al-4V for 80 s.

increased at a constant rate up to 60 s or so. In the case of cpTi, the grinding rate increased with time at the same rate up to 15 s or so, but the rate appeared to decrease with time. The grinding rates in the initial grinding, up to 15 s for both metals, were similar to each other. Figure 5 is the plot of the depth removed by grinding as a function of grinding times for both metals. The estimated α -case thicknesses shown in Figure 1 are indicated with parallel lines in Figure 5 for each metal. These results indicate that the α -case layer in cpTi and Ti-6Al-4V was removed approximately the first 15 s and 5 s of grinding, respectively. Figure 6 is a series of typical SEM micrographs of the cross-sectioned specimens of cpTi and Ti-6Al-4V after grinding for 5 s, 10 s and 15 s. Figure 7 shows typical SEM micrographs of the metal chips generated in the grinding of cpTi and Ti-6Al-4V of the 5 s or 80 s grinding. Metal chips of the 5 s of grinding of cpTi seem to be finer than chips of the 80 s of grinding. Metal chips from Ti-6Al-4V for both the 5 s and 80 s grinding were similar.

Figure 8 compares the typical backscattered electron images and secondary electron images of the grinding surfaces of the SiC wheel after used in grinding of the 80 s for the cpTi and Ti-6Al-4V specimens, respectively. In the backscattered electron images [cpTi (a) and Ti-6Al-4V (a) in Figure 8], metal chips of both cpTi and Ti-6Al-4V, which were generated during grinding, are seen to have adhered on the surfaces of the wheel. Larger numbers of metal chips

formed by the plastic-shear deformation are attached on the wheel surfaces used for grinding cpTi. Although not shown here, the amount of metal chips attached on the wheels used in less grinding times was found to be much less.

DISCUSSION

According to the grinding theory developed by Chan *et al.*³⁰⁾ the grinding rate, G_R , for K-controlled fracture of Ti alloys is given by

$$G_R = \frac{\alpha F s}{\pi t_w} \left(\frac{L}{K_{IC}} \right)^2 \quad (1)$$

where t_w is the thickness of the grinding wheel, K_{IC} is the fracture toughness (critical stress intensity factor for fracture), and αF is a constant with a value of 100 for Ti castings. For the grinding experiments performed in this study, $s = 20.8$ m/s (1250 m/min), $L = 0.98$ N, and $t_w = 1.5$ mm. Using these inputs, the grinding rates for cpTi and Ti-6Al-4V were computed using the bulk material properties for K_{IC} . The predicted grinding rates are presented in Table 1. The grindability or grinding rate of Ti-6Al-4V is about 3.98 mm³/60 s compared to 2.71 mm³/60 s for cpTi. Once the grinding rate is known, the volume of material, V_G , removed by grinding can be computed readily as

$$V_G = G_R t \quad (2)$$

Table 1 Fracture toughness and grindability of cpTi and Ti-6Al-4V. Fracture toughness values are from Chan *et al.*³⁰⁾

Material	K_{IC} MPa (m) ^{1/2}	G_R (K-controlled) m ³ /min
cpTi	97	2.707
Ti-6Al-4V	80	3.982

where t is the time of grinding. Using Eqs. (1) and (2), the volumes removed by grinding were computed for cpTi and Ti-6Al-4V (dotted lines) and the results were compared against the experimental data in Figure 4. The theory predicted that the amount of volume removed by grinding increased linearly with grinding time from both metals because of the assumption of a constant grinding rate or grindability for each alloy. In comparison, the amount ground from both metals consistently increased with the grinding time up to 60 s, followed by an increase with a lower grinding rate for both metals. After the 15 s grinding, the amount ground in Ti-6Al-4V is significantly higher than that in cpTi at each corresponding time ($p < 0.05$). Note that during the grinding time up to 15 s, the amount ground from both metals is similar. Overall, the predicted volume loss for cpTi is in agreement with the experimental data to within experimental scatter, while the theory appears to underpredict the volume loss observed in Ti-6Al-4V. This discrepancy may be attributed to a lower fracture toughness value that was used to compute the volume loss for Ti-6Al-4V. Interestingly, the grinding rate up to 15 s for both cpTi and Ti-6Al-4V is similar, suggesting that the fracture toughness and ductility of the α -cases of these titanium alloys probably have similar values. Reduced grinding rates observed after 60 s for both metals are changed by the changes in the grinding surface of the SiC wheels with probably the combined effect of flaking off SiC particles, losing sharpness of their surfaces and clogging up with ground chips into open spaces on the wheel surfaces. Some attached metal debris of cpTi and Ti-6Al-4V is shown in Figure 8. Usually, more attached metal chips are found on the wheels used for grinding cpTi, because of its higher ductility and lower fracture toughness of cpTi.

In theory, the depth of grinding, d_G , can be computed from the volume loss according to

$$d_G = \frac{V_G}{A} \quad (3)$$

where A is the area of the specimen surface, which was taken to be the product of the grinding width (3.0 mm) and the thickness (1.5 mm) of the SiC grinding wheel. The predicted results for the depth of grinding (dotted lines) are compared against the experimental data in Figure 5, which shows a plot of the means of the depth

measured from the original cast surface at each grinding time for both cpTi and Ti-6Al-4V. The depth of grinding is predicted to increase linearly with time, which was observed in Ti-6Al-4V. The depth of grinding is predicted to increase at a slower rate in cpTi than Ti-6Al-4V, which was also observed. Within the α -case, the observed depths of grinding for cpTi and Ti-6Al-4V are identical and are higher than those predicted by the theory.

The formation of the oxygen contaminated, hardened surface layer, the α -case, is inevitable in the regular lost-wax investment casting of titanium^{2,19)}. There have been some investigations regarding the effect of the α -case on various characteristics of cast titanium. Its effect is controversial in properties such as corrosion resistance, bond strength of porcelain to the titanium substrates, etc.

From the standpoint of mechanical properties, it may be true that titanium castings perform better with the removal of the α -case surface structure. As for grindability, no careful investigations have been performed indicating the effectiveness of the α -case. The present study, which tested α alloy cpTi and $\alpha+\beta$ alloy Ti-6Al-4V, suggested the grinding rate of the α -case in both types of titanium was very similar. In our theoretical study of grindability for titanium alloys³⁰⁾, we found that their grindability increased with decreasing fracture toughness (K_{IC}) and tensile ductility. What we found in the present study was that the grindability in the α -case layer was higher than the bulk material. This observation can be understood on the basis that the hardened surface layer is enriched with oxygen, which is known to strengthen but embrittle cpTi. The increase in oxygen content in the α -case is expected to reduce the fracture toughness and tensile ductility, thereby improving the grindability of the α -case, as suggested by Eq. (1). Once the α -case is removed, grindability decreases as the subsequent material removal behavior is controlled by fracture resistance and tensile ductility of the bulk material. In addition, the grindability of the α -case in different Ti alloys appears to be essentially identical regardless of the microstructure of the bulk material (α Ti or $\alpha+\beta$ Ti), since the α -case in both titanium alloys is rich in oxygen and its properties are dictated by the oxygen content. As we found previously³⁶⁾, the grindability of the interior structure of cast cpTi is much worse due to its higher tensile ductility and fracture toughness (K_{IC}). In cpTi, the grinding rate decreased with grinding inside the α -case structure as the K_{IC} increased in the bulk material. By virtue of an embrittled layer with a lower K_{IC} , the presence of the α -case is beneficial in grinding cast titanium dental prostheses since the lower fracture toughness allows the α -case to be fractured easily and removed by the grinding wheel.

CONCLUSIONS

The ease of grinding of the α -case on cpTi and Ti-6Al-4V was very similar, probably due to the similar

microstructure of the α -case regardless of the metal. Perhaps the α -case composition formed on both cpTi and Ti-6Al-4V has a similarly lower tensile ductility and fracture toughness (K_{IC}). The grinding of the interior of cpTi, as compared to that of Ti-6Al-4V, was more difficult because of its higher ductility. In contrast to the perception of difficulty in cutting, grinding and polishing the α -case layer when compared to processing the interior metal structures, the presence of the α -case, which is an embrittled layer with a lower fracture toughness, is beneficial in grinding cast titanium because the lower fracture toughness allows the α -case to be fractured and removed easily by a grinding wheel. As a consequence, the α -case on a ductile titanium-like cpTi can be ground much easier than its bulk interior structure. In a less ductile alloy such as Ti-6Al-4V, the grinding rate of the α -case and its interior structure is at a similar level when the difference in fracture toughness values between the α -case and the bulk material is not large.

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