Influence of grade and surface topography of commercially pure titanium on fatigue properties

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The objective was to investigate the influence of grade and surface topography of commercially pure titanium (Cp-Ti) on fatigue properties evaluated via staircase method. Cp-Ti grades 2 and 4 were roughened by shot blasting and acid etching, and compared with machined specimens. Yield force under static loading for Cp-Ti grades 2 and 4 were 672±51 and 1,088±93 N for machined and 724±99 and 1,118±96 N for roughened group. Yield force under cyclic loading for Cp-Ti grades 2 and 4 decreased 27 and 40% compared to static loading. Cp-Ti grade 4 demonstrated significantly greater decrease in yield force after cyclic loading; however surface topography had no effect.

Keywords: Commercially pure titanium, Dental implant, Fatigue property, Shot blasting, Acid etching

INTRODUCTION

The excellent corrosion resistance and biocompatibility of titanium enable them to be used for biomedical applications such as prosthetic heart valves, heart pacemakers, vascular stents, dental implants, artificial joint components, and orthopedic screws. Commercially pure titanium (Cp-Ti) is commonly used for dental implants; however, fractures have been reported in clinical settings1-3. Goodacre et al. investigated clinical complications in root form implants and implant prostheses performed between 1981 and 2001, and found 142 incidences of implant body fractures out of 12,157 cases5. This 1% rate of incidence was low relative to other complications, but was nevertheless a serious problem, especially since the outcome was equivalent to severe cases of bone resorption caused by peri-implantitis, which the implant and prosthesis must be removed1. Eckert et al. suggested that implant fracture was occurred after prosthetic or abutment screw loosening2. Therefore, a cause of fracture was most likely fatigue of the implant material due to repetitive occlusal masticatory forces2,9.

Cp-Ti are categorized into 4 grades depending on impurity content (e.g., carbon and oxygen) under the International Organization for Standardization (ISO) standards 5832-2. Dental implant and orthopedic plates are made of Cp-Ti grade 2 and 4. The strength increases with grade while purity and elongation decreases6. Although grade 4 Cp-Ti possess larger strength than grade 2, fractured orthopedic plates were made of grade 4 Cp-Ti9. Additionally, plates for mandibular reconstruction surgery have fractured in regions of stress concentration6. This suggests a possibility that dental implants made from grade 4 Cp-Ti which possess the highest static strength may be prone to fracture in regions of high stress concentration.

Dental implant bodies undergo various surface treatments to encourage osseointegration after placement. Two types of surface treatments currently exist: one alters the surface topography and the other alters the surface chemistry6. Treatments that modify titanium topography include plasma spraying and a combination of shot blasting and acid etching. Treatments that alter the surface chemistry include hydroxyapatite coating, ion implantation, and hydrophilic modifications (cold plasma-surface modification, ultraviolet light irradiation, hydrogen peroxide treatment)7. In certain cases, a treatment that alters the surface chemistry can also subsequently change the topography (e.g., ion implantation).

A combination of shot blasting and acid etching is often performed on Cp-Ti implants because it reportedly accelerates osseointegration8-10. In vitro experiments have demonstrated that the combination promotes adhesion, proliferation, and differentiation of osteoblasts11-15. In vivo experiments have demonstrated that it improves bone-implant contact and increases removal torque8-10. Gil et al. reported that the residual compressive stress layer formed after shot blasting improves the fatigue life of Cp-Ti10. Pazos et al. reported that the fracture strength of a shot-blasted group and a combined shot-blasted and acid-etched group exhibited similar behavior with a machined group; however, an acid-etched group had lower fracture strength16. In contrast, Ketabchi et al. reported that acid-etched topography did not affect Cp-Ti fracture strength18. These studies suggest that surface topography affects titanium fatigue, but this topic has not been studied in detail.

Recent findings have shown that static loading may be insufficient to evaluate implant durability in a...
clinical setting\(^3\). As an alternative, fatigue strength has been investigated using cyclic loading\(^{19-21}\). In 2003, ISO developed a standardized protocol for implant fatigue testing\(^{22}\), and there have been several studies that have followed the procedure\(^{23-27}\). Although these papers were evaluated to implant systems, there have been no reports on fatigue of Cp-Ti cylinder, which were treated with the same surface treatment as commercial implants. The evaluation of implant systems using the staircase method, a fatigue testing procedure of ISO standard, has been increasing\(^{27}\). However, the fatigue property of Cp-Ti on its own has not been studied using the same staircase method used for dental implant systems.

The objective of this study was to investigate the effects of grade and surface topography on fatigue property of Cp-Ti. Fatigue property was assessed using the staircase method then analyzed by cross sectional morphology and hardness.

**MATERIALS AND METHODS**

**Sample preparation**
The chemical composition and mechanical properties of grades 2 and 4 Cp-Ti (Tokyo Titanium, Saitama, Japan) are summarized in Table 1\(^4\). Fifty Cp-Ti cylinders (3-mm diameter, 17-mm length) were fabricated for each grade [Fig. 1(a)], and polished with 800-grit silicon carbide wheels using the centerless method (performed by Tokyo Titanium). For each grade, 25 cylinders were kept with machined surfaces (MS). Surfaces of the other 25 were roughened via shot blasting and acid etching (RS). Specifically, they were blasted with 250- to 300-μm alumina particles perpendicular to the surface from a distance of 10 mm, at a pressure of 0.5 MPa, followed by acid etching with an equal mixture of 37% hydrochloric and 96% sulfuric acid at 70°C for 4 min (conducted by San-ai Plant, Tokyo, Japan). The samples were ultrasonically washed in acetone and distilled water before use.

**Three-dimensional surface imaging**
The cylindrical surfaces were imaged and reconstructed in three dimensions with a three-dimensional electron beam surface roughness analyzer (ERA-8900FE, Elionix, Tokyo, Japan), operated at an accelerating voltage of 15 kV. To obtain a multi-characterization of the surface topography, images were acquired in two-dimensional ranges of 240×180 μm\(^2\) (500× magnification) and 24×18 μm\(^2\) (5,000× magnification). Surface roughness (Sa) was evaluated as one of three-dimensional parameters in a measured area of display diagram, which expanded from the linear roughness (so-called as Ra). The Sa was calculated from a followed equation.

\[
Sa = \frac{1}{A} \int \int |Z(x,y)| \, dx \, dy \quad (A: \text{measured area})
\]

**Static and cyclic loading tests**

1. **Apparatus**

Static and cyclic loading tests were performed according to ISO 14801\(^{22}\). A hemispherical cap made of carburized quenching steel with chromium plating was placed over one end of each sample, which was then inserted into an aluminum pipe (70-GPa elastic modulus, 5-mm external diameter, 1-mm thickness) with a slit longitudinally running along its length [Fig. 1(b)]\(^{19}\). The sample in the aluminum pipe was then fixed in a holder at a 30° angle from the vertical axis [Fig. 1(c)]. To simulate the level of

| H    | O    | N    | Fe   | Ti   | Percentage elongation (%) |
|------|------|------|------|------|---------------------------|
| Grade 2 | <0.0125 \(^a\) | <0.25 | <0.03 | <0.30 | Balance | >20 |
| Grade 4 | <0.0125 \(^a\) | <0.40 | <0.05 | <0.50 | Balance | >15 |
|       |       |      |      |      | Proof stress of non-proportional elongation (MPa) |
|       |       |      |      |      |            | >275 |
|       |       |      |      |      |            | >483 |

\(^a\) Except for billets, for which the maximum hydrogen content shall be 0.0100% (mass fraction) and for flat products for which the maximum hydrogen content shall be 0.015% (mass fraction).
bone, the distance between the center of the spherical cap and the sample holder was set at 11 mm.

2. Static loading test
For the static loading test, load-deflection curve at room temperature was measured with a universal testing machine (Autograph AG-I 20 kN, Shimadzu, Kyoto, Japan) for five samples from each group. A perpendicular load was applied at a crosshead speed of 0.5 mm/min to the samples inclined at an angle of 30°. The load was recorded as the yield force, Ys, under the static loading test when a 0.1-mm displacement of the load point was observed; the displacement value was determined as an initial indicator for non-linear structural behavior on load-distance curve in pilot study. The Ys values were statistically analyzed with two-way analysis of variance (ANOVA), followed by the Student’s t-test (α=0.05).

3. Fatigue property evaluation (cyclic loading test)
Cyclic loading test was performed by a servo-hydraulic universal testing machine (EHF-FD05, Shimadzu) that the sample was inclined at an angle of 30° and applied a cyclic load at 10 Hz for 10^6 cycles, according to confirm the standard ISO 14801. The yield force, Yc, was determined via the staircase method as follows. Initially, 70% of Ys was applied to the first sample. If deformation (0.1-mm displacement of the load point) occurred in less than 10^6 cycles (a failure), the cyclic load for the next sample was reduced by one increment, which was 5% of Ys. If deformation did not occur after 10^6 cycles of loading (run-out), the cyclic load was decreased by one increment for the next sample. This procedure was repeated for all 20 samples in each group.

The arithmetic mean of Yc was calculated by first counting the number of run-outs and failures. The run-out or failure with the lowest number of samples was used for the calculation. For the case of the run-out with the lowest number of samples:

\[ Y_c = \frac{\sum_{i=1}^{n} (f_i + 0.5 \cdot d)}{n} \]

For the case of the failure with the lowest number of samples:

\[ Y_c = \frac{\sum_{i=1}^{n} (f_i - 0.5 \cdot d)}{n} \]

where \( f_i \) is the load used for testing, \( n \) is the total number of samples used for the calculation, and \( d \) is the step width derived from 5% of Ys. The standard deviation (\( \sigma \)) was calculated from:

\[ \sigma = \sqrt{\frac{\sum_{i=1}^{n} (f_i - Y_c)^2}{n-1}} \]

**Optical microscopic observation**
After static and cyclic loading tests, samples were embedded in epoxy resin along the direction of the long axis and sectioned lengthwise. The sectioned surfaces were ground and mirror polished with a polishing machine (Ecomet 3, Buehler, Lake Bluff, IL, USA) with waterproof SiC abrasive papers (120, 240, 400, 800, 1200 grit), 3-μm diamond suspension, and 0.02-μm colloidal silica suspension. The surfaces were then etched with a hydrofluoric acid solution (4.5% hydrofluoric acid and 31.3% nitric acid; Chemipolish, Shofu, Kyoto, Japan) for 30 s, and imaged with an optical microscope (BX51, Olympus, Tokyo, Japan).

**Vickers hardness**
After static and cyclic loading, the Vickers hardness of the samples was measured with a micro hardness tester (MVK-E, Akashi, Tokyo, Japan). The load of the indenter was 980 mN for 20 s. One indentation was made on each sample before static and cyclic loading as a control, and five indentations were made after the cyclic loading test. Samples were placed parallel to the longitudinal direction and indentations were made on the end of the tensile side, the middle of the tensile side, the center, the middle of the compressive side, and the end of the compressive side (Fig. 2). Vickers hardness was statistically analyzed with one-way ANOVA, followed by Dunnett’s test to compare data before loading (\( \alpha=0.05 \)).

**RESULTS**

**Three-dimensional SEM imaging**
Figure 3 shows SEM and three-dimensional topographical images and the Sa for each sample. On the MS samples, many features were observed along one direction, while the RS samples had relatively roughness in nanoscale morphologies. The Sa of grade 2 MS, grade 4 MS, grade 2 RS, and grade 4 RS at 500× magnification (240×180 μm²) were 0.4, 0.1, 1.5, and 1.2 μm, respectively. At 5,000× magnification (24×18 μm²), the Sa were 0.07, 0.05, 0.17, and 0.18 μm, respectively.

**Static and cyclic loading tests**
Figure 4 shows the static loading yield force, Ys,
which was 672±51, 1,088±93, 724±99, and 1,118±96 N, respectively, for grade 2 MS, grade 4 MS, grade 2 RS, and grade 4 RS. Two-way ANOVA revealed a significant difference in Ys between grade 2 and grade 4 ($p<0.05$), and no significant difference in Ys between surface topographies ($p=0.317$). Therefore, Ys is affected only by the titanium grade. There was no correlation between grade and surface topography ($p=0.778$).

The results of the cyclic loading staircase method to determine Yc are shown in Fig. 5. Figure 6 summarizes Yc for 20 samples. For grade 2 MS, grade 4 MS, grade 2 RS, and grade 4 RS, the Yc were 472±81, 620±58, 560±68, and 675±50 N, respectively.

**Optical microscopy**

Figure 7 shows optical microscope images of grade 2 and 4 samples that failed during cyclic loading. Cracks were observed on the tensile sides of both samples. Figure 8 shows typical microstructure images of grade 2 RS and grade 4 RS samples after cyclic loading. Twinning is observed in the grade 2 RS sample, but not in the grade 4 RS sample. There was appreciable twinning not only on the tensile and compressive sides, but also at the center of the samples. The number of twin on the tensile and compressive sides was larger than on the center.

**Vickers hardness**

Figure 9 shows the typical Vickers hardness for grade 2 and 4 samples before and after cyclic loading. Hardness at the tensile and compressive sides was significantly different between the two grades, which are also compared with the pre-tested control (Ctrl).
Fig. 5 Mean yield force $Y_c$ under the cyclic loading tests via the staircase method.

Fig. 6 Yield forces $Y_c$ under cyclic loading via the staircase method, based on 20 samples. Data are mean±standard deviation.

Fig. 7 Optical microscope images of samples that failed during cyclic loading. Left of each image is the tensile side of the sample and the right is the compressive side. (a) Grade 2 sample, (b) grade 4 sample.
**DISCUSSION**

Previously, the cyclic loading of vertical and horizontal stresses along the longitudinal direction of a sample was performed as fatigue test\(^9\). In a dental clinical setting, however, occlusal stress is applied not only perpendicularly but also diagonally. These factors make it difficult to establish whether the calculated fracture
strengths can actually be expected for dental implants.

To create a more severe condition here, the diameters of the titanium samples were 3 mm, which is narrower than commercial implants. Cyclic loading was performed in accordance with ISO 14801, which is used to determine implant fracture strength and durability. Thus, Ys and Yc for Cp-Ti were determined according to the standardised procedure by fixing the sample at an angle of 30° from the load direction.

Many fatigue testing procedures have been devised to determine the yield force for cyclic loading, Yc, including the standard method using the S-N curve, constant stress level testing, the response or survival method (the probit method), the step-test method, the plot method, and the extreme value method\(^{29,30}\). All require enormous testing time and effort, and have to conduct repeated loads until many samples were broken. In contrast, the staircase method used here can be used to quickly derive an average fatigue force with a smaller number of tests\(^{29}\). Dental zirconia implants were found to be suitable for use for long periods of time in the oral cavity after determining fracture strengths using the staircase method for various surface topographies\(^{12,19,31}\). Here, the staircase method was used to efficiently and accurately perform cyclic loading tests on a small number of samples to evaluate pure titanium durability\(^{29}\).

Our results showed that Yc for grade 2 was 25% lower than Ys, and was 43% lower than Ys for grade 4. Accordingly, the difference between Ys and Yc is greater for grade 4 than that for grade 2. A possible explanation is that grade 4 Cp-Ti has a lower ductility relative to grade 2\(^{40}\); therefore, the low ductility may lead to crack propagation in less than 10\(^6\) cycles-loading, and then detection of 0.1 mm-deformation on the inclined sample could be accelerated. Generally, less slip occurs within the planar metallic structure during loading, which leads to less plastic deformation. Plastic deformation in metals is caused by slip and twinning. During slip deformation, the crystal orientations do not change during plastic deformation along the slip plane; whereas, in twinning deformation, the crystal orientations change during plastic deformation. In the hexagonal close-packed lattices of titanium, there are fewer slip planes relative to body-centered cubic or face-centered cubic lattices. However, because twinning deformation causes a change in crystal orientation, a new slip plane can occur. Therefore, plastic deformation in grade 2 was a result of slipping and twinning. Hence, grade 4 Cp-Ti had a larger difference between Ys and Yc, relative to that of grade 2.

After cyclic loading, cracks were observed on the tensile side in some grade 2 and 4 samples as shown Fig. 7. Vickers hardness tests, which was subject to clarify the hardening behavior after fatigue test, also showed those both grade 2 and 4 samples were harder than the pre-tested samples (control), indicating that work hardening occurred for the grade 2 and 4 samples during cyclic loading. Therefore, work hardening reduced plastic deformation while increasing brittleness that caused cracks on the tensile side.

As expected, the Sa of the shot-blasted and acid-etched RS group was greater than that of the MS group at both magnifications (Fig. 3). Structures of nano-meter level can form on the surface of implants by immersion in hot solutions of strong acids (hydrochloric acid, sulfuric acid, nitric acid, and hydrofluoric acid)\(^{32}\). Roughness of nano-meter level has known to synergistically promote osteogenic proliferation and differentiation, and bone formation\(^{36}\). In this study, we confirmed that the same modification of the surface topography for dental implants was properly performed on Cp-Ti samples, and fatigue property was evaluated.

The Ys and Yc values in present study indicated that surface topography with shot-blasting and acid etching had no effect on yield force. Our result were in agreement with a previous report\(^{17}\), where surface treatments had no effect on fatigue life of titanium. Guilherme et al. also reported that no correlation exists between surface roughness and fracture strength\(^{53}\); however, surface treatments for ceramics often cause cracks on ceramics, which propagate internally causing mechanical strength to decrease. For yttria-stabilized tetragonal zirconia polycrystals, shot-blasted and acid-etched samples had lower fracture strengths relative to mirror polished samples\(^{19,34}\). However, crack propagation does not occur in metals because of plastic deformation caused by slip and twinning due to the existence of free electrons.

Titanium implant which were embrittled by hydrogen adsorption had led to fracture\(^{35}\). The amount of hydrogen adsorption on the acid etched pure-titanium was larger than those on Ti-6Al-4V. That is, the additive elements to titanium and the contents might influence the amount of hydrogen adsorption\(^{30}\). However, in this study, shot blasting and acid etching for Cp-Ti with different grade had no influence on Ys and Yc. Further investigation on the effect of hydrogen adsorption of dental titanium implant by acid etching on cyclic fatigue of the implant will be needed.

Over long periods, fatigue strength of grade 4 Cp-Ti decreased relative to static strength at a rate higher than that of grade 2. Therefore, in cases where a patient exhibits bruxism, or any other habit causing high occlusal masticatory forces, a grade 4 Cp-Ti implant, based on its standard value of static strength (yield and tensile strength), may have insufficient fatigue strength. In such cases, higher strength materials, such as titanium alloys, are necessary for dental implants\(^{37-39}\).

To understand the structural factors that lead to failure of implants, cylindrical Cp-Ti samples with various grades and surface topographies were used as a first step to evaluate fracture strengths. In future studies, cyclic loading tests using actual dental implants are required to overcome these limitations.

CONCLUSIONS

Static and cyclic loading was performed on cylindrical samples made from grade 2 and 4 Cp-Ti. Sample surfaces were either smooth or rough. Conclusions are
as follows:
1. The yield force under cyclic loading of grade 2 and 4 Cp-Ti was respectively 27 and 40% lower than the yield force under the static loading.
2. Surface modification via shot blasting and acid etching had no effect on the fatigue properties.

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CONFLICTS OF INTEREST

The authors declare no conflicts of interest.

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