Application of multi-directional forged titanium for prosthetic crown fabrication by CAD/CAM

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Titanium are often used as dental materials, pure titanium present low strength and titanium alloy is reported poor biocompatibility, respectively. To overcome the problem, we fabricated high-strength multi-directional forged (MDF) titanium with improved mechanical properties without changing the chemical composition and evaluated its applicability in prosthetic crowns. Cutting tests: the average absolute value of the difference before and after cutting was calculated as the uncut amount. Surface evaluations: MDF titanium, pure titanium, and the Ti-6Al-4V alloy were the surface properties (the surface roughness, the contact angles, glossiness) of the samples were evaluated. The fitness test used digital data. These demonstrated that the good workability of high-strength MDF titanium. The surface-roughness and contact-angle properties of MDF titanium and pure titanium were similar. The fitness test showed no significant differences between MDF titanium and pure titanium crowns. These results suggest that MDF titanium is promising for fabricating prosthetic crowns in dental applications.

Keywords: MDF titanium, CAD/CAM, Fitness test, Glossiness, Digital data

INTRODUCTION

Conventionally, metal materials used for dental treatment are typically manufactured by the lost wax method1,2. In this method, various factors such as the setting expansion of the plaster model and casting shrinkage of the metal affect dimensional accuracy. Therefore, the suitability of prosthetic devices often depends on the skill of the operator, which makes it difficult to obtain standardized products. Nevertheless, with advances in computer aided design/computer aided manufacturing (CAD/CAM) technology, it is now possible to manufacture prosthetic devices with standard specifications3.

In Japan, CAD/CAM is used in various dental treatment scenarios, such as for manufacturing dental crowns under the National Health Insurance scheme. Among the metal materials often processed by CAD/CAM for dental applications4, titanium-based materials which exhibit good stability, such as pure titanium and titanium alloys, are often used in implants, dentures, and prosthodontic treatments. It has been reported that the fit accuracy of the CAD/CAM approach for processing such materials is good5.

The strength of pure titanium can be enhanced by alloying it with elements such as Ni, Co, Cr, V, and Al. However, alloying Ti with Ni, Co, and Cr might result in allergic reactions in patients6, while V poses the risk of carcinogenicity and Al may induce Alzheimer’s disease7,8. Although pure titanium exhibits better biocompatibility than titanium alloys, it presents several disadvantages such as easy oxidation, difficulty in forming, and low strength when compared to the latter9. Under these circumstances, in recent years, the development of processing methods that can alter the properties of metals by generating ultrafine grains with a crystal size of less than 1 μm is attracting much attention from both clinicians and materials scientists10. This is primarily because grain refinement can increase the strength of metals without the need for additional alloying elements11,12.

Miura et al. developed high-strength multi-directional forging (MDF) pure titanium (hereafter referred to as MDF titanium) without changing the chemical composition of pure titanium13-15. In the MDF method, the forging direction is changed by 90° for each forging pass and the crystal structure is ultrafine-grained by applying a large machining strain; meanwhile, the sample shape remains unchanged. MDF titanium exhibited a maximum tensile stress, yield stress, and elastic modulus 2.5, 4, and 0.5 times of the corresponding values of grade 2 pure titanium (hereafter referred to as pure titanium), respectively. These characteristics attest to the enhanced properties (high strength and elasticity) of MDF titanium16. In fact, the strength of MDF titanium was found to be higher than that of titanium alloys.

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(Ti-6Al-4V) used for dental implants (ultimate tensile strength: 995.3 MPa, Vickers hardness: 293.5 Hv); in addition, it exhibited excellent corrosion resistance and cell adhesion. Therefore, it can be potentially applied in implants and crown restorations.16-18

However, to apply MDF titanium clinically to crown restoration and fixed partial prosthesis, the machinability and its dimensional accuracy must be equal to or better than that of pure titanium already used in CAD/CAM systems, there is not much information available on this subject.

Therefore, in this study, we investigated the machinability, surface properties, and fitness of MDF titanium manufactured by CAD/CAM to evaluate its clinical application in crown restorations.

MATERIALS AND METHODS

Physical property testing

1. Cutting tests

MDF titanium (Kawamoto Heavy Industries, Yokosuka, Japan), Grade 2 pure titanium (JIS, Japan Industrial Specification H 4600, 99.9 mass% Ti, GN-1 titanium block GC, Tokyo, Japan), and titanium alloy (Ti-6Al-4V: titanium block GC for dental CAD/CAM GN-I) blocks (all samples \( n = 10 \); width of 12.0×, height of 16.0 mm) were used as the experimental samples.

On a cutting drill (WXL-LN-EBD R1×18DR1.0 ball end mill; neck length 18 mm, OSG, Aichi, Japan), each block sample was rotated at a frequency of 10,000 min\(^{-1}\) and feed rate of 1,000 mm/min with a step down of 0.1 mm to achieve a width of 11 mm and height of 7.0 mm. Cutting was performed without changing the drill during the processing operation (Fig. 1).

After cutting, a micrometer (Coolant Proof Micrometer SPM2-25MJ, Mitutoyo, Kanagawa, Japan) was used to measure the width of the sample after cutting at three places (at the ends and center), and the average absolute value of the difference before and after cutting was calculated as the uncut amount.

2. Surface-property testing

The surface roughness, contact angle, and glossiness of the prepared samples were measured to evaluate their surface properties. MDF titanium (\( n = 10 \)), Grade 2 pure titanium (\( n = 10 \)), and the Ti-6Al-4V alloy (\( n = 10 \)) with all disk width of 20.0 mm and height of 1.0 mm were initially ground with #400, #600, and #1000 waterproof sandpaper. Subsequently, the surface properties of the samples were evaluated. The surface roughness (Center line average roughness: Ra) was measured using a surface-roughness-measuring instrument (Handysurf E-40A, Tokyo Seimitsu, Tokyo, Japan). The contact angles of the samples were measured using an automatic contact-angle meter (DCA-VZ, Kyowa Interface Science, Saitama, Japan) with 1.5 \( \mu \)L drops of water; the contact angle was measured 1 s after the droplet came into contact with the sample surface. Meanwhile, the sample glossiness was measured at an angle of 60° using a gloss meter (GM-268Plus, Konica Minolta, Tokyo, Japan). In all, 10 each material samples were analyzed for each property and each sample was tested thrice to reduce the experimental error (total of 30 times per material).

Fitness testing

As the master model, an abutment tooth with a taper of 6°, height of 8 mm, and diameter of 10.0 mm with an all-around heavy chamfer was designed by CAD and manufactured by CAM with a PEEK (Poly Ether Ether Ketone) material.

Based on the designed master model data (hereafter referred to as master data), crowns with a cement space of 30–50 \( \mu \)m were manufactured by CAM (GM-1000, R0.5, R1.0, R1.5, GC) with Grade 2 pure titanium and MDF titanium (both material samples: \( n = 10 \)). The fitness of the manufactured crowns with respect to the abutment tooth was confirmed by pressure welding using finger pressure.

A geometric block (LEGO, Billund, Denmark) was set...
at a distance of 3 mm from the abutment tooth and used as the reference point for superimposition during fitness testing. For each crown, a light-body silicone impression material (ImprintTM 4 Light, 3M, Dusseldorf, Germany) and a putty silicone impression material (Fusion II putty type, GC) were used to make impressions. To ensure that the crown margin was scanned, a digital scan (TRIOS E3, 3Shape, TRIOS, Copenhagen, Denmark) of the inner surface of the crown was generated in the impression material and abutment was performed.

Two acquired data (Standard Triangulated Language: STL) were integrated using a data-measurement software, Geomagic (3D systems, South California, CA, USA), and based on the geometrical form of the blocks, 3D data were generated (Fig. 2). From the prepared abutment tooth data, the dimensions of the inner surface of the crown were measured at two locations—the margin and 2 mm from the margin; using these results, the fitness was evaluated.

### Results

#### Physical property testing

1. Cutting tests

The results of the cutting test showed that the uncut amount increased in the order of pure titanium (0.08±0.01 mm) followed by MDF titanium (0.10±0.01 mm) and titanium alloy (0.14±0.01 mm) (Table 1).

#### Surface-property testing

After polishing sample surfaces with waterproof #400,

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Table 1  Cutting-test analysis

|                | MDF titanium | Pure titanium | Titanium alloy |
|----------------|--------------|---------------|---------------|
|                | 0.1±0.01     | 0.08±0.01     | 0.14±0.01     |
|                | a            | b             | c             |

Mean values (n=10) and standard deviations in parentheses. All samples showed significant differences. Values denoted with same letters are not significantly different (p<0.05).

Table 2  Summary of results

|                | MDF titanium | Pure titanium | Titanium alloy |
|----------------|--------------|---------------|---------------|
|                | #400         | #600          | #1000         |
| surface roughness | 0.36±0.04   | 0.33±0.05     | 0.25±0.03     |
| Contact angle   | 69.25±5.81   | 70.22±5.57    | 72.00±10.90   |
| Glossiness      | 76.82±8.36   | 96.32±11.51   | 108.67±11.81  |

Mean values (n=10) and standard deviations in parentheses.

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Statistical analysis

Significant differences were determined using the software (SPSS Statistics 22.0, IBM, Armonk, NY, USA). The data were analyzed for normal distributions by Shapiro-Wilk test and for variance equality by Levene test. One-way analysis of variance and Tukey’s tests were performed on the cutting and surface-property results. Conformity testing was performed by the Mann-Whitney U test (p<0.05).

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Table 3  Comparison of the fitness accuracy of MDF titanium and pure titanium crowns

| Sample number | MDF titanium | Pure titanium | MDF titanium | Pure titanium |
|---------------|--------------|---------------|--------------|---------------|
| 1             | 23.9         | 6.9           | 17.7         | 25.9          |
| 2             | 40.3         | 3.5           | 9.7          | 20.3          |
| 3             | 10.8         | 8.8           | 17.4         | 22.3          |
| 4             | 17.9         | 37.5          | 23.6         | 18.4          |
| 5             | 12.2         | 23.0          | 23.9         | 13.7          |
| 6             | 15.0         | 6.8           | 21.4         | 19.7          |
| 7             | 12.0         | 12.1          | 17.2         | 29.1          |
| 8             | 4.2          | 0.6           | 17.8         | 27.2          |
| 9             | 36.3         | 27.8          | 17.7         | 27.8          |
| 10            | 8.0          | 8.3           | 18.0         | 17.8          |

18.1±11.3 a
13.5±11.3 a
18.4±3.8 a
22.2±4.8 a

All samples were not significantly different. Values denoted with same letters are not significantly different ($p<0.05$).

#600, and #1000 sandpaper, the titanium alloy samples showed a significantly lower surface roughness and smaller contact angle than pure titanium and MDF titanium. However, there was no significant difference between pure titanium and MDF titanium (Table 2).

In the case of gloss measurements, significant differences could be observed between MDF titanium and pure titanium samples, and pure titanium and titanium alloy samples polished with #400 and 600 sandpaper. In both sets, the glossiness of pure titanium was lower. When the samples were polished with #1000 sandpaper, significant differences could be observed between all the groups; in this case, the glossiness of the titanium alloy samples was particularly high (Table 2).

Fitness testing

The fit of the MDF titanium crown was evaluated to be 18.1±11.3 μm at the margin and 18.4±3.8 μm at a distance of 2 mm from the margin; in the case of pure titanium, the corresponding values were 13.5±11.3 and 22.2±4.8 μm, respectively. This indicates that there were no significant differences between the fitness of MDF titanium and pure titanium crowns (Table 3).

DISCUSSION

In recent years, titanium-based materials are being extensively used for dental treatment. However, the lost wax method, which is often used to manufacture components based on titanium, causes casting shrinkage, defects, and the formation of an $\alpha$-case surface layer. Therefore, CAD/CAM, a new manufacturing method that can be standardized industrially and ensures reproducibility, is being adopted to enhance the original properties of titanium-based components.

It has been shown that MDF titanium exhibits an ultimate tensile stress of 995.3 MPa, elastic modulus of 51.0 GPa, and Vickers hardness of 293.5 Hv (greater hardness than pure titanium: 468.4 MPa, 100.3 GPa and 176.8 Hv). In addition, it has been suggested that when used in implants, MDF titanium exhibits a high affinity for osteoblasts, thus promoting cell adhesion and proliferation.

In the current study, we observed that in addition to its high strength, MDF titanium exhibits good cutting workability; furthermore, its surface properties and fit accuracy are similar to those of pure titanium.

CAD/CAM is considered the main method for manufacturing crowns based on titanium. However, titanium is difficult to cut and very often, the drill life is short, which makes it difficult to maintain product quality. This is because during machining, the cutting temperature increases, which results in deformation of the titanium material and tool wear.

The results of the present study illustrate that MDF titanium has cutting properties between pure titanium and Ti-6Al-4V alloys, even though it is stronger than the Ti-6Al-4V alloy. This implies that the damage incurred by the cutting drill is mitigated and the economics of machining are improved, which is an immense advantage in clinical applications.

It was also observed that the surface roughness and contact angle of polished MDF titanium were similar to those of pure titanium; in addition, it exhibited high glossiness, which is desirable in dental materials. The surface properties of dental materials are usually evaluated in terms of their surface roughness and the intensity of specular reflection; in general, the
lower the roughness, the higher the gloss\textsuperscript{23}. However, one particular study suggests that this correlation is incorrect because glossiness changes with respect to surface roughness, depending on the surface-processing or surface-treatment method\textsuperscript{24}. The glossiness is Ra less than 0.2 μm and the diffuse reflection light due to the uneven slope of the surface decreases, and it increases sharply as most of the light is specular\textsuperscript{25}. In addition, it is thought that metallic luster is realized when Ra is 53 nm or less\textsuperscript{23}.

In the present investigation, we found that MDF titanium exhibits a surface roughness and contact angle similar to those of pure titanium; meanwhile, its glossiness was slightly lesser than that of the titanium alloy. According to previous reports, MDF titanium consists of ultrafine particles (average grain size<200 nm) with a uniform structure\textsuperscript{16}. This implies that the surface roughness after polishing can be attributed to minimal reattachment with cutting chips and easier polishing ability than that of pure titanium. Meanwhile, the surface roughness of the titanium alloy was lesser than that of pure titanium, but the effect of polishing was minimal unlike in the case of MDF titanium\textsuperscript{9}.

The results of contact-angle analysis indicate a trend similar to that reported earlier —the contact angle of titanium-alloy samples were slightly smaller than that of pure titanium\textsuperscript{26}.

With respect to glossiness, it was easier to obtain a smooth surface with MDF titanium than with pure titanium; the microstructure of the former contained uniform fine particles less than 200 nm in size. It may be considered that the glossiness increased because the low-frequency component of the polished surface increased and the intensity of reflected specular light increased\textsuperscript{27}. However, it should also be noted that the glossiness of the alloy surface, whose surface roughness, was smaller than that of MDF titanium, was higher.

We also analyzed the fit accuracy of MDF titanium and pure titanium crowns. Pure titanium processed by CAD/CAM is being used clinically and the accuracy of these components has been reported to be equivalent to that of cast components\textsuperscript{28,29}. However, it is difficult to evaluate the fit accurately using conventional methods as it is difficult to stabilize measurement points; the conventional analog method measures distances microscopically using silicone impressions\textsuperscript{30-40}. Furthermore, it is not objective and poor in reproducibility.

To overcome this limitation, in this study, we employed a new method to compare digital data using a data-measurement software (3D systems). Using this method, all the data can be converted into digital units and evaluated in three dimensions\textsuperscript{41}. When the data points of the master model and crown inner surfaces are superimposed, a reference point is always required. At this time, if the surface of the scan data is superimposed on the whole, the result is erroneous. However, in the new digital method, it is possible to easily set a reference point by simultaneously applying a geometric block and scanning. This makes it possible to accurately superimpose the two sets of data\textsuperscript{42}. Using this method, the accuracy of the crown was up to 36.3 μm in the margin space. The clinical tolerance of crowns made by CAD/CAM has been reported to be 120–150 μm space\textsuperscript{30,32,36,40}; because our results are within this range, it may be inferred that CAD/CAM MDF titanium crowns are suitable for clinical use. In future, other aspects such as their abrasion resistance and plaque adhesion should be studied to evaluate their suitability for clinical applications.

**CONCLUSION**

In summary, the results of this study demonstrate that CAD/CAM MDF titanium exhibits a strength similar to that of titanium alloys but easier cutting workability. Furthermore, its surface roughness and contact angle are similar to those of pure titanium; in addition, it exhibits a higher gloss. With respect to fit accuracy, crowns made of MDF titanium exhibited a fit equivalent to that of pure titanium crowns and this value was well within the clinical tolerance limits. Together, these results suggest that CAD/CAM MDF titanium may be potentially used for dental applications.

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