



# In vitro evaluation of a removable partial denture framework using multi-directionally forged titanium

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**PURPOSE.** This study evaluated the availability of multi-directionally forged (MDF) titanium (Ti) as a component of removable partial dentures (RPDs). MDF-Ti remarkably improved the mechanical properties of RPDs due to its ultrafine-grained structure. **MATERIALS AND METHODS.** The wear resistance, plaque adhesion, and machinability of MDF-Ti were tested. As controls, commercially pure (CP) titanium was used for wear, plaque adhesion, and machinability tests. For wear resistance, the volume losses of the titanium teeth before and after wear tests were evaluated. Plaque adhesion was evaluated by the assay of *Streptococcus mutans*. In the machinability test, samples were cut and ground by a steel fissure bur and carborundum (SiC) point. An unpaired t-test was employed for the analysis of the significant differences between MDF-Ti and the control in the results for each test. **RESULTS.** Wear resistance and plaque adherence of MDF-Ti similar to those of CP-Ti ( $P > .05$ ) were indicated. MDF-Ti exhibited significantly larger volume loss than CP-Ti in all conditions except 100/30,000 g/rpm in machinability tests ( $P < .05$ ). **CONCLUSION.** Although the wear resistance and plaque adherence of MDF-Ti were comparable to those of controls, MDF-Ti showed better machinability than did CP-Ti. MDF-Ti could be used as a framework material for RPDs. [J Adv Prosthodont 2020;12:369-75]

**KEYWORDS:** Multi-directionally forged titanium (MDF-Ti); Wear resistance; Machinability; Removable partial dentures (RPDs)

## INTRODUCTION

Dental alloys, namely, Co-Cr, Au-Pt, and titanium alloys, have been used for a removable partial denture (RPD) framework due to their mechanical properties and corrosion resistance. Especially, titanium has been widely used for prostheses, including implant superstructures, by its superior elastic modulus and cost effectiveness.<sup>1-3</sup> However, some problems of Ti-6Al-4V, such as toxicity and irritability, have been pointed out due to the release of Al or V.<sup>4-8</sup> Therefore, the attempt has been made to fabricate frameworks of prostheses from commercially pure (CP) titanium.<sup>9-12</sup>

On the contrary, the mechanical properties of metallic materials can be improved without alloying by refining their crystal grains to ultrafine crystals, which is widely recognized as the Hall-Petch relationship.<sup>13-15</sup> Several methods

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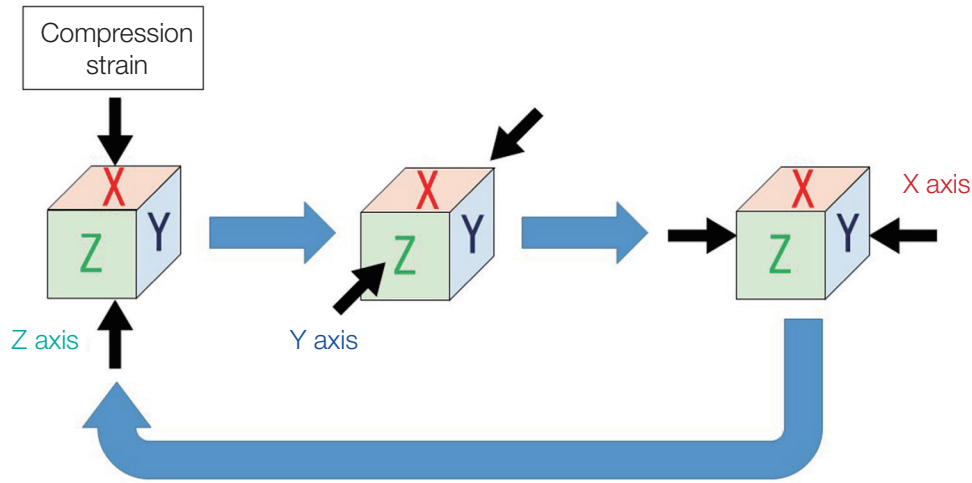
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**Fig. 1.** General concept for the MDF method. Forging axis was changed as illustrated for 90 degrees.

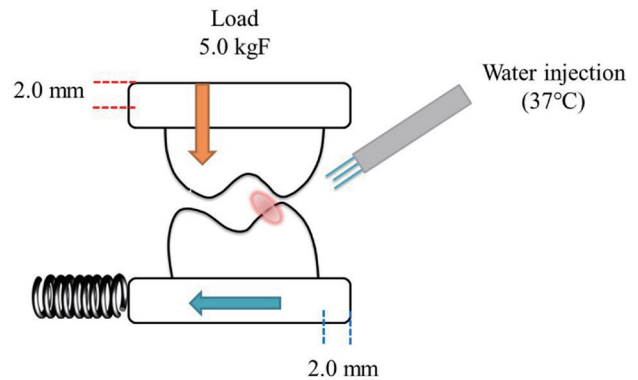
have been reported for reducing the grain size by plastic deformation technique, such as accumulative roll bonding (ARB),<sup>16</sup> high pressure torsion (HPT),<sup>17</sup> equal-channel angular pressing (ECAP),<sup>18</sup> and multi-directional forging (MDF).<sup>19,20</sup> Of these, MDF would be applicable to large samples, and there are few limitations on the shape. Figure 1 shows a schematic diagram of the MDF process. The strain is produced by forging repeated while forging axis is changing as shown in Figure 1. Miura *et al.* reported that application of MDF method to titanium and magnesium alloys produced ultrafine structure and produced higher tensile strength.<sup>20</sup> The size of conventional CP titanium is approximately 30  $\mu\text{m}$ , but grade 2 titanium treated with MDF (MDF-Ti) had an average particle size smaller than 100 nm and a maximum tensile strength exceeding 1 GPa. It is reported that MDF-Ti has higher mechanical strengths such as tensile strength and Vickers hardness than those of conventional CP titanium.<sup>21</sup> Arai *et al.* also found that proliferation of osteoblast-like cell on MDF-Ti was significantly promoted than that on conventional CP-Ti after sulfuric acid treatment to MDF-Ti and CP-Ti.<sup>22</sup> Suzuki *et al.* monitored the corrosion behavior of MDF-Ti by fluoride solution and found that MDF-Ti was less susceptible to corrosion when immersion period in acidulated phosphate fluoride solution was less than 6 hours.<sup>23</sup> Bone formation was also evaluated after the implantation of MDF-Ti into the sockets of rat maxillary molars after the extraction. Suzuki *et al.* reported that sulfuric acid or NaOH heat treatment on the surface of MDF-Ti improved the bone response.<sup>24</sup>

MDF-Ti might be considered as a suitable material for the framework of removable dentures because the strength can be improved without alloying. Thus, the wear resistance, plaque adhesion, and machinability of MDF-Ti were evaluated *in vitro* to fabricate the frameworks of RPDs.

## MATERIALS AND METHODS

For wear test, disk-shaped blocks of Grade 2 MDF-Ti (Ti 99.5%, Kawamoto Heavy Industries Co., Ltd., Hyogo, Japan) and CP titanium (CP-Ti, Kawamoto Heavy Industries Co., Ltd., Hyogo, Japan) were processed into the shape of upper and lower right first molars (Livdent FB30, GC Corp., Tokyo, Japan) using dental CAD/CAM system (GN-1, GC Corp., Tokyo, Japan) (n = 5). The occlusal surface of the titanium teeth was sandblasted with 50  $\mu\text{m}$  alumina particles at a distance of 10 mm for 15 sec.

Figure 2 shows a schematic drawing of the impact sliding wear test using an original wear testing machine (JAPAN MEC, Tokyo, Japan).<sup>25-27</sup> The samples were mounted in the correct position using a special jig so that all teeth had the



**Fig. 2.** Schematic illustration of the impact sliding wear test.

same occlusal contacts. The same titanium was paired for the upper and lower teeth. The vertical and horizontal moving distance were set to 2.0 mm under a load of 5.0 kgf. The test was performed up to 50,000 times at 60 cycles per minute with the injection of 37°C water. The weights of the titanium teeth before and after the wear tests were measured by using electronic balance (AUW120D, Shimadzu Corp, Kyoto, Japan), which is a semi-micro (0.01 mg) model. The volume loss was calculated from the weight loss based on the specific gravity of titanium ( $= 4.5 \text{ g/cm}^3$ ) and was evaluated as wear resistance. The worn surface was observed using a scanning electron microscope (SEM, S4000, Hitachi High-Technologies Corp., Tokyo, Japan). The acceleration voltage was 15 kV and each specimen was coated with gold.

For plaque adhesion test, MDF-Ti and CP Ti (Grade 2) were processed into a disk shape with 15.0 mm diameter and 1.0 mm thickness (Fig. 3A) ( $n = 5$ ). After the surface of titanium disks was polished with silicone points, all disks were autoclaved and coated with filtered saliva from a volunteer for easy adhesion of plaque to surfaces. The surface roughness (Ra) of the CP-Ti and MDF-Ti after polishing was measured with Handysurf E-35A (Tokyo Seimitsu, Tokyo Japan) with a scan length of 4 mm and a cut off value of 0.8 mm. The surface appearances after polishing was observed using a scanning electron microscope (SEM,

S4000, Hitachi High-Technologies Corp., Tokyo, Japan) at an accelerating voltage of 15 kV after ion coating with gold.

One day later, *Streptococcus mutans* bacteria were inoculated into brain-heart infusion broth (BHI; DIFCO, Becton Dickinson Co., Sparks, MD, USA) and sucrose medium (liquid medium) at 37°C with aerobic shaking. After 3 days of culturing, the attached plaque was removed with distilled water. Each disk was stained with a DENT Liquid plaque tester (Lion Dental Products Co., Ltd., Tokyo, Japan). The stained sample was rinsed with water when the colorization of rinsing water was not detected. Finally, stained disks were decolorized with 99% ethanol (500 mL) by rinsing. Plaque adhesion was evaluated by measuring the absorbance (562 nm) of the rinsed ethanol.

For machinability test, MDF-Ti and CP-Ti (Grade 2) plates were cut into rectangular plates with 30.0 mm  $\times$  10.0 mm  $\times$  3.0 mm (Fig. 3B) ( $n = 5$ ). For the machinability test, a steel fissure bur (Dentsply Sirona K.K., Tokyo, Japan) with a diameter of 2.0 mm and a carborundum (SiC) point (Shofu Inc., Kyoto, Japan) with a diameter of 2.0 mm for a micromotor handpiece were used to evaluate cutting efficiency and grindability, respectively. A micrometer handpiece was set into an original setting apparatus as shown in Fig. 4. The force and distance acted by the handpiece was constant during machinability test.

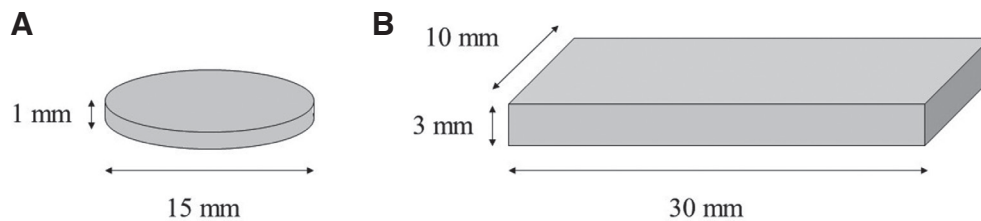


Fig. 3. Schematic illustration of the disks (A) and plates (B) for plaque adherence and machinability tests, respectively.

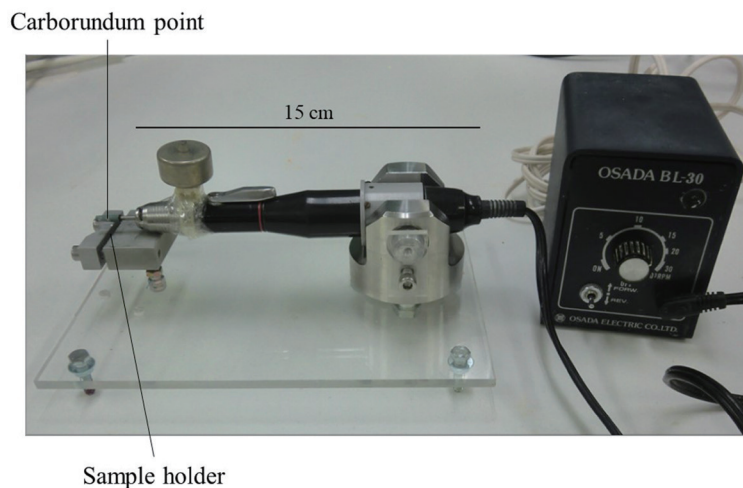


Fig. 4. Picture of the equipment used in the machinability tests.

Machinability tests were performed using four patterns of paired loads and rotational speeds (100/1,500, 100/30,000, 300/15,000, and 300/30,000 g/rpm). After the machinability tests, the volume loss of each plate was measured.

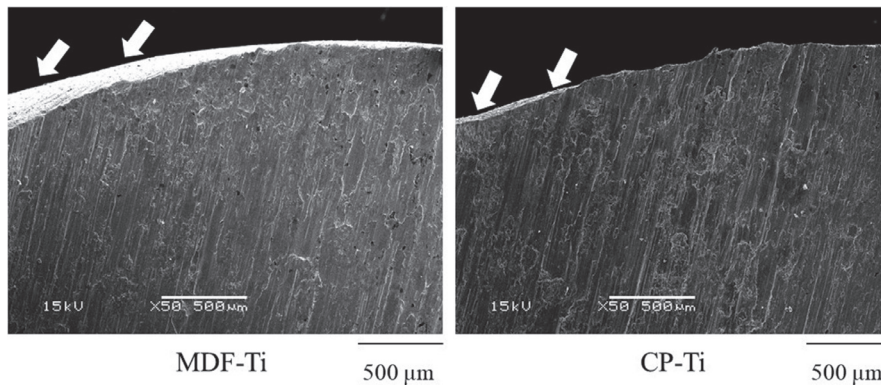
SPSS version 23 (IBM Japan, Tokyo, Japan) was used for the statistical analysis. The results in wear resistance, surface roughness after polishing with silicone points, plaque adhesion, and machinability of MDF-Ti and CP-Ti were analyzed using an unpaired t-test. *P* values of less than .05 were considered significant, and data were depicted as the mean ± standard deviation (SD).

**RESULTS**

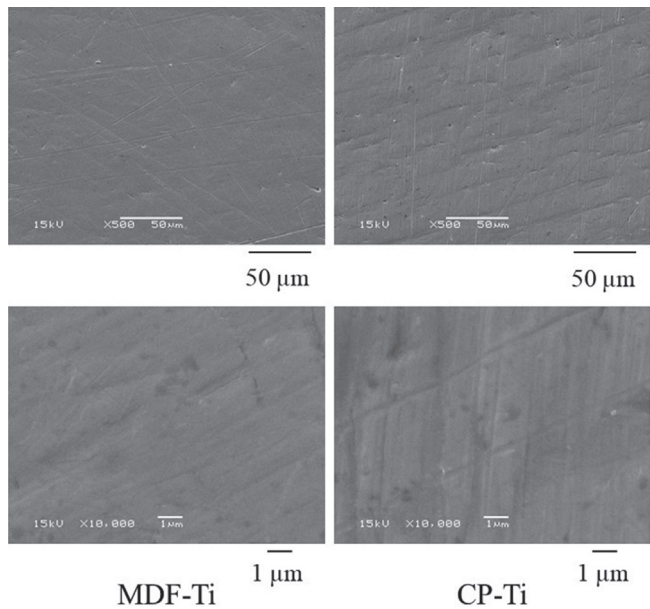
The average volume loss of MDF-Ti was  $2.91 \pm 0.26 \text{ mm}^3$  and that of CP-Ti was  $3.36 \pm 0.72 \text{ mm}^3$ . Volume loss of MDF-Ti was not significantly different from that of CP-Ti

(*P* > .05). Figure 5 shows the SEM observation of the worn surface. Wear marks along the sliding direction were recognized on roughened surfaces of MDF-Ti and CP-Ti (see arrows).

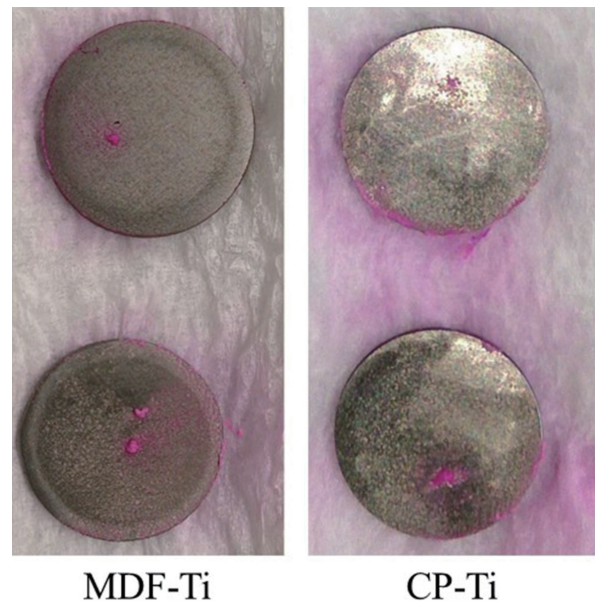
In the plaque adhesion test, Ra values of CP-Ti and MDF-Ti after polishing were  $0.21 \pm 0.01 \text{ }\mu\text{m}$  and  $0.20 \pm 0.02 \text{ }\mu\text{m}$ , respectively. There was no significant difference in Ra value between CP-Ti and MDF-Ti (*P* > .05). Figure 6 shows the surface appearances observed by SEM. There was no distinct difference between CP-Ti and MDF-Ti. Figure 7 shows the disk surface immediately after staining and washing with water. The pink stained part indicated the plaque attachment site. The average absorbances of rinsed ethanol after plaque adhesion test were  $0.31 \pm 0.28$  for MDF-Ti and  $0.32 \pm 0.18$  for CP-Ti. There was no significant difference in plaque adhesion between MDF-Ti and CP-Ti (*P* > .05).



**Fig. 5.** SEM appearances of the worn surfaces of MDF-Ti and CP-Ti.



**Fig. 6.** SEM pictures of the surfaces of MDF-Ti and CP-Ti used in the plaque adhesion test.

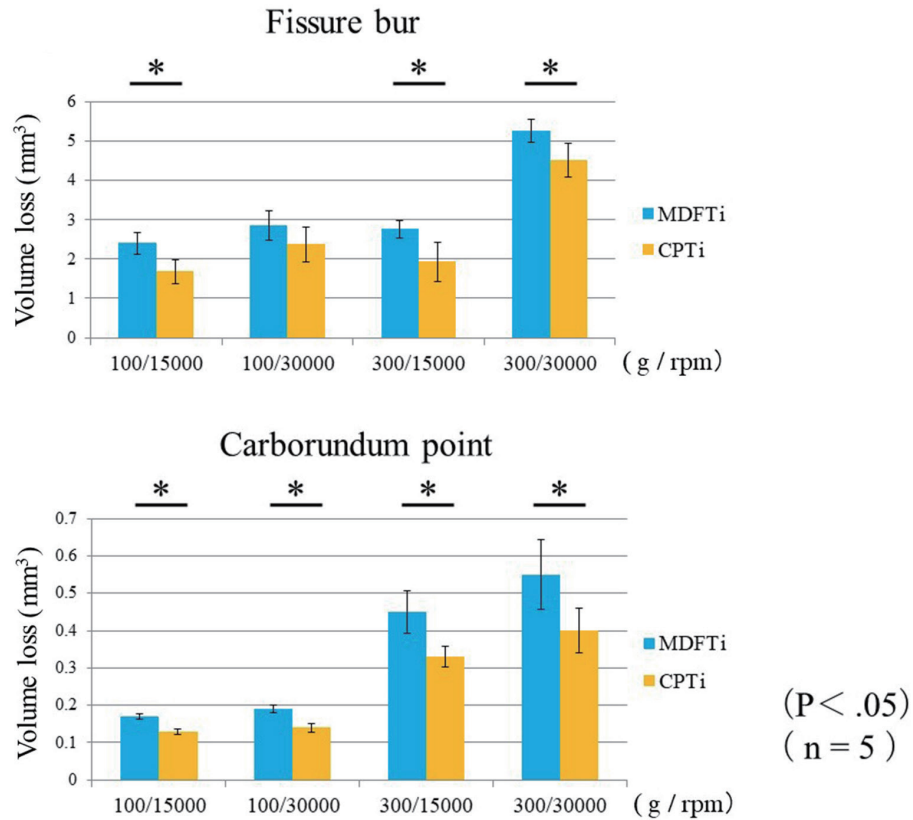


**Fig. 7.** Disk surface immediately after the dyeing and washing of MDF-Ti and CP-Ti.

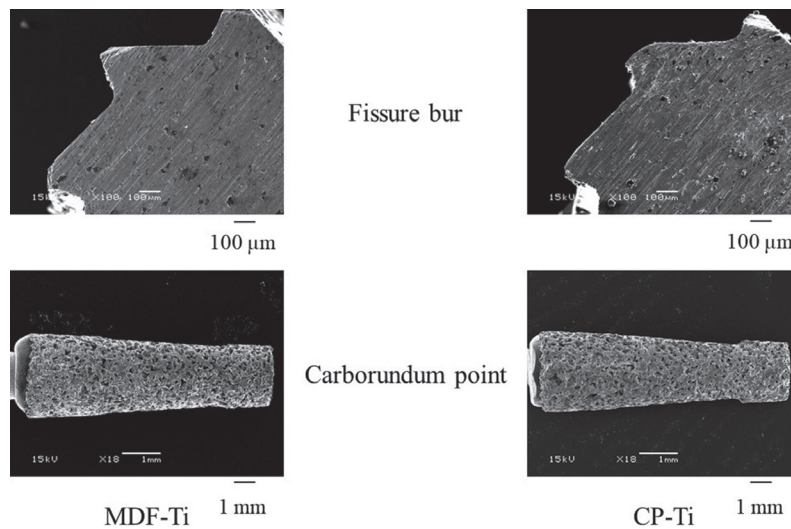


Figure 8 shows the volume loss of the sample after machinability testing. MDF-Ti showed significantly greater volume loss than CP-Ti on all conditions except for 100/30,000 g/rpm using a fissure bur. Figure 9 shows the SEM images of the fissure bur and carborundum points after a

1-minute test under the 100/15,000 g/rpm. The fissure bur and the carborundum point were abraded by the cutting and grinding of both MDF-Ti and CP-Ti. MDF-Ti was recognized as having slightly less damage than CP-Ti in both the fissure bur and the carborundum point.



**Fig. 8.** Volume loss of the sample under each condition of MDF-Ti and CP-Ti after machinability test.



**Fig. 9.** SEM pictures of the fissure bur and SiC point after a 1-minute test under the condition of 100/15,000 g/rpm of MDF-Ti and CP-Ti.

## DISCUSSION

In the present study, we evaluated the basic properties of ultrafine grained Ti for framework of RPDs. Ultrafine grained Ti has not been applied in dental clinics. This is the first trial of ultrafine grained Ti in prosthetic dentistry. Ultrafine grained Ti was produced by MDF method, which is more applicable compared to other plastic deformation technique. It is expected that higher mechanical strengths of MDF-Ti will be beneficial for framework materials of RPDs. So we designed the wear tests, plaque adhesion tests, and machinability tests.

The wear resistance of titanium teeth was worse in cases of same grades of titanium (for example, grade 2 - grade 2, grade 3 - grade 3) for upper and lower teeth.<sup>28</sup> If different grades of titanium teeth-for instance, grade 2 - grade 3, grade 2 - grade 4-were used, wear resistance was increased.<sup>29</sup> In this study, MDF-Ti indicated wear resistance similar to that of CP-Ti. Thus, to keep appropriate occlusal contacts for the long term, MDF-Ti should be used for either the upper or lower tooth and CP-Ti for the other tooth.

In the present experiment, plaque adhesion was evaluated on the surface of CP-Ti and MDF-Ti, which was polished in the same way used in the actual dental clinics. As a result, the adherence of plaque to MDF-Ti was also similar to that of CP-Ti. This means that framework manufactured by MDF-Ti has similar plaque properties as that by CP-Ti. Therefore, it is concluded that MDF-Ti will be applicable as a material for a framework. On the contrary, it is presumed that more roughness surface will produce more plaque adhesion. In the clinical situation, it is important to avoid rougher surface of MDF-Ti framework. Urushibara *et al.* reported that the amount of biofilm from *S. mutans*, or *C. albicans* on the CP-Ti and Au-Pt alloys was nearly equal or higher than that on the resin surface.<sup>30</sup> To clarify the details of plaque adherence to MDF-Ti, the biofilm formation on MDF-Ti and the removal of biofilm from MDF-Ti should be studied further. In addition, the surface roughness will influence the formation of biofilm in oral conditions.<sup>31</sup> Controlling surface roughness after polishing the MDF-Ti is another important issue for clinical usage.

Generally, it is very difficult to cut and grind CP-Ti.<sup>32-35</sup> The machinability of MDF-Ti was greater than that of CP-Ti in this study. This is thought to be due to the refinement of titanium crystal grains. MDF will solve the problem for cutting and grinding for the fabrication and adjustment of RPDs. When framework was prepared by CP-Ti, it took a lot of time for processing, grinding and polishing the material. It is presumed that it will be easier to do processing, grinding and polishing by the usage of MDF-Ti and it is also expected to shorten the chair time for adjusting the RPDs in dental clinics. Since the wear of both cutting and grinding tools was less with MDF, the cost of machining would decrease.

The tendencies of wear resistance, plaque adherence, and the machinability of MDF-Ti for RPDs were clarified in this study, but the details of the mechanism remain

unclear.

The problem of MDF-Ti is that it is difficult to repair the broken MDF-Ti framework. For example, laser welding will produce greater grain size by heat effect. The repair method which maintains the ultrafine grain structure of MDF-Ti should be further studied. Further fundamental mechanical studies such as 3 or 4 point bend test and/or accelerated fatigue tests, and simulated oral environmental experiments should be necessary for the clinical use of MDF-Ti. Clinical investigation would be necessary for the clinical use of MDF-Ti.

## CONCLUSION

In the present study, the following conclusions could be obtained:

1. The wear resistance of the MDF-Ti tooth was similar to that of the CP Ti tooth in the case where the same titanium was used for the upper and lower teeth.
2. The plaque adhesion to the MDF-Ti surface was observed to be similar to that of the CP-Ti surface.
3. The machinability of MDF-Ti was significantly better than that of the CP-Ti using a fissure bur and SiC point.

The above results suggest that MDF-Ti can be used as the material for RPD framework.

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