Mechanical Strength and Microstructure of Laser-welded Ti-6Al-7Nb Alloy Castings

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Received August 4, 2005/Accepted September 12, 2005

INTRODUCTION

In the recent decade, commercially pure titanium (CP Ti) and titanium alloys have expanded in their range of dental applications — dental implants1-30, crowns, and fixed/removable partial dentures4-18 — because of their superior biocompatibility. However, disadvantages of CP Ti have also been pointed out, such as lack of mechanical strength for some dental applications19 and poor wear resistance20-22. There is also a concern about the cytotoxicity of vanadium in Ti-6Al-4V alloy23,24. With high mechanical properties and biocompatibility, Ti-6Al-7Nb alloy was consequently introduced to be an alternative to CP Ti or Ti-6Al-4V alloy25-27 as a dental casting alloy.

Soldering is a common method that joins dental alloy prostheses for clinical use although the corrosion resistance of the soldered materials is a concern28-31. However, soldering is not suitable for joining CP Ti or titanium alloys because of the decrease in corrosion resistance and biocompatibility due to the contact of different types of metal32. Other methods for joining CP Ti or titanium alloys have been introduced, such as plasma welding33, tungsten inert gas (TIG) welding34-36, and infrared brazing37-39. However, the disadvantage of large heat-affected zone created by plasma welding38, TIG, and infrared brazing39 was also reported. In addition, these methods require filler metals that could potentially reduce corrosion resistance40,41. Against these disadvantages, laser welding has become a preferred method to join metals in dentistry, especially for CP Ti and titanium alloys.

Since the use of laser welding in dentistry was reported by Gordon and Smith in 197032, many studies on laser-welded titanium have been reported33-44,50-51. Some studies showed reduced strength of laser-welded specimens36,37, while others reported no differences in the strength after laser welding42,43,45,46,47. As most of the studies focused mainly on CP Ti, laser welding on titanium alloys, especially Ti-6Al-7Nb alloy, has not yet been sufficiently investigated. In this study, the mechanical properties of laser-welded Ti-6Al-7Nb alloy castings at different welding conditions were investigated in relation to their microstructure for clinical application in prosthodontics.

MATERIALS AND METHODS

Specimen preparation
ISO 6871 dumbbell-shaped specimens, 18 mm in gauge length and 3 mm in diameter at the parallel part, were cast with Ti-6Al-7Nb alloy (T-Aloy Tough, GC, Japan), Grade 2 CP Ti (TITAN INGOT JS 2, Selec, Japan), and Co-Cr alloy (Cobaltan Clasp, Shofu, Japan). Six specimens of each casting metal were prepared for each laser welding condition group. In addition, five specimens of each metal were cast and used as control specimens. Ti-6Al-7Nb alloy and CP Ti specimens were cast with an argon gas centrifugal casting machine (Ticast Super R, Selec, Japan) and a magnesia-based investment (Selevest15 CB, Selec, Japan). Co-Cr alloy specimens were cast with a
centrifugal casting machine (Denko Auto Sensor MD-201, Denko, Japan) and a phosphate-bonded investment (Wiroplus® N, Bego, Germany).

After casting, the specimens were bench-cooled to room temperature and sandblasted with 50 μm alumina powder. Following which, the specimens were inspected with nondestructive X-ray instrument (DCX-100, Asahi Roentgen, Japan) to detect any noticeable internal defects. All specimens except the control groups were cut perpendicularly at the center of the parallel part with 0.5-mm thick cutting wheel under water coolant. The specimens were then cleaned with acetone before laser welding.

Laser welding and macroscopic observation
Laser welding was performed with an Nd:YAG laser welding machine (Alpha Laser ALP 50S, Yasui, Japan) under the flow of argon gas. Based on the results of our pilot study, two different laser voltages, 220 V and 260 V, were employed. These two laser voltages were within the voltage range recommended by the manufacturer of this laser welding machine. Pulse duration and spot diameter were set at 8 ms and 1 mm, respectively.

To examine the penetration depth into alloy castings after laser-welded at the above two laser voltages, 3-mm thick plate specimens were cast with the same three casting metals and cut in half following the previously described method. Four specimens of each metal were used for each laser voltage. Only one spot of laser welding was applied on one side at the interface of two halves of the plate specimens so that they could be mechanically separated. The penetration depths of laser welding were measured on the separated surfaces by a profile micrometer (VF-7500, Keyence, Japan).

A group of six dumbbell-shaped specimens of each metal was laser-welded at 220 V or 260 V of laser voltage. Two halves of each specimen were positioned touching each other on a grooved copper block, and four spots of laser welding were first applied on each side of the specimen. Then, continuous weld seam was completed around the circumference without the copper block. Each welded spot was overlapped approximately 50-60% by the next welded spot. The surface characteristics of laser-welded specimens were observed with an optical microscope (SZX 12, Olympus, Japan) before tensile testing was carried out.

Tensile test and fractographic examination
Five specimens of each experimental and control groups were tested with a multi-axial hydraulic testing machine (MTS 858 Mini Bionix®, Minnesota, USA). Tensile loading force was applied with a cross-head speed of 1.6 × 10^-5 m s^-1. Mechanical strength and elongation were calculated with the cross-sectional area and gauge length of the specimens. ANOVA and Tukey-Kramer post-hoc test were performed to analyze the data with significance level at 95%. Fractographs were obtained from the fractured specimens with a scanning electron microscope (SEM) (JSM-6400, JEOL, Japan).

Metallographic examination
After laser welding, one of six specimens in each experimental group was used for metallographic examination. They were embedded in resin (Technovit® 400, Heraeus, Germany) and ground parallel to the long axis of the specimens until the central part of each laser-welded joint was exposed. The surfaces were polished and finished with 0.05 μm alumina. The surfaces of laser-welded Ti-6Al-7Nb alloy and CP Ti specimens were etched with a reagent of 32 mL H2O, 15 mL 60% HNO3 and 3 mL 46% HF, while the surfaces of laser-welded Co-Cr alloy specimens were etched with a reagent of 20 mL 35% HCl and 1 mL 30% H2O2. The microstructures were observed using an optical microscope (BX60M, Olympus, Japan).

RESULTS
The penetration depth values are shown in Table 1. Regardless of casting metal, the depth values were more than 1.5 mm for 260 V and less than 1.5 mm for 220 V of laser voltage. It could be assumed that the joints of the 3-mm diameter specimens were completely laser-welded in the 260 V condition, while only the peripheral depth of the joints was welded in the 220 V condition.

**Mechanical strength of laser-welded joint**
The mechanical strength and elongation of the laser-welded alloy castings and control specimens are shown in Fig. 1. It was found that the mechanical strength of 260 V laser-welded specimens was signifi-

<table>
<thead>
<tr>
<th>Laser voltage (V)</th>
<th>Penetration depth</th>
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<tr>
<td></td>
<td>Ti-6Al-7Nb</td>
</tr>
<tr>
<td>220</td>
<td>1.0 (0.15)</td>
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<tr>
<td>260</td>
<td>2.2 (0.17)</td>
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( ): standard deviation
cantly higher than that of 220 V laser-welded specimens for both Ti-6Al-7Nb and Co-Cr alloys (p<0.05), except for CP Ti. Between the control and 260 V groups, Ti-6Al-7Nb alloy and CP Ti specimens showed no significant differences in both mechanical strength and elongation. On the other hand, the mechanical strength and elongation of the laser-welded Co-Cr alloy groups were considerably lower than the control group (p<0.05). The elongation of laser-welded Co-Cr alloy specimens was not significantly different between 220 V and 260 V conditions.

Comparison among the alloys revealed that laser-welded Ti-6Al-7Nb alloy specimens had the highest mechanical strength, followed by laser-welded CP Ti and Co-Cr alloy specimens (p<0.05), while laser-welded CP Ti specimens showed the highest elongation.

Fractured part of laser-welded specimen after tensile test
Although all 220 V laser-welded Ti-6Al-7Nb alloy specimens were fractured within the welded joints, all 260 V laser-welded Ti-6Al-7Nb alloy specimens were fractured outside the welded joints. One specimen in the 220 V laser-welded CP Ti group was fractured within the welded joint, while the rest in the two voltage groups of CP Ti specimens were fractured outside the welded joints. All laser-welded specimens of Co-Cr alloy, i.e., including both 220 V and 260 V groups, were fractured within the welded joints.

Macroscopic and fractographic observations
Surface characteristics of the laser-welded specimens at low magnification are shown in Fig. 2. The welded areas of Ti-6Al-7Nb alloy and CP Ti specimens showed a smooth border (Figs. 2-1 and 2-2). In contrast, Co-Cr alloy specimens showed irregular bubble-like appearance at the periphery of the welded area with cracks, regardless of laser voltage (Fig. 2-3).

Figs. 3 to 5 show the SEM micrographs of the fracture surfaces for each experimental condition as well as those of the control specimens. Laser-welded Ti-6Al-7Nb alloy and CP Ti specimens of the 220 V condition, which fractured within the welded joints, showed the same fracture characteristics of equiaxed dimples formed by microvoid coalescence (Figs. 3-1A and 4-1A). As for the 260 V laser-welded Ti-6Al-7Nb
alloy and CP Ti specimens, which fractured outside the welded joints, they exhibited fracture characteristics of cup-like dimples (Figs. 3-2A and 4-2A) similar to the control specimens (Figs. 3-3A and 4-3A). However, all laser-welded Co-Cr alloy specimens showed the same fracture characteristics of the so-called cleavage fracture, identified by sharply defined planes and angles (Fig. 5), unlike the control specimen which showed ductile fracture (Fig. 5-3A). More porosity was observed in Co-Cr alloy specimens than in Ti-6Al-7Nb alloy or CP Ti specimens.

Microstructure
Metallographic pictures obtained from the intact laser-welded specimens are shown in Fig. 6. In contrast to the laser-welded CP Ti and Co-Cr alloy specimens, three basic zones of laser-welded joint identified as fusion, heat-affected, and unaffected zones could be observed in the laser-welded Ti-6Al-7Nb alloy specimens. The acicular α structure at prior-β grain boundaries was observed in the unaffected zone whereas the fusion zone exhibited fine α structure in a matrix of primary β grains (Fig. 6-3 for Ti-6Al-7Nb alloy). For the laser-welded CP Ti specimens, the fusion zone exhibited acicular α-prime (martensite) structure (Fig. 6-3 for CP Ti), while the unaffected zone showed a coarse α grain structure. Distinct cracks existed in the fusion zone of both 220 V and 260 V laser-welded Co-Cr alloy specimens, corresponding to the finding in macroscopic observation. The metallographic picture showed very fine precipitates in the matrix of the fusion zone (Fig. 6-3 for Co-Cr alloy). No clear heat-affected zones could be identified for both laser-welded CP Ti and Co-Cr alloy specimens.

DISCUSSION
The mechanical properties of a laser-welded joint are influenced not only by laser parameters including
pulse energy, pulse duration, and spot diameter, but also by the properties of alloys and the welding method\(^9\)\(^). The penetration depth of laser-welded alloys varies among the alloys due to different laser beam absorption rates, thermal conductivity values, and melting temperatures. It is known that a greater laser beam absorption rate and lower thermal conductivity will lead to greater penetration depth into the metal. Hence, the high rate of laser beam absorption and low thermal conductivity of titanium (40%, 0.17 W cm\(^{-1}\) K\(^{-1}\), respectively) are favorable factors for joining CP Ti or titanium alloys by laser welding\(^9\).

With respect to laser parameters, it was reported that an increase in spot diameter decreased the penetration depth, and it became very low if the spot diameter was larger than 1.2 mm, even though pulse duration was increased\(^11,13\)\(^). Shorter pulse duration was reported to cause less penetration depth, but long pulse duration with low voltage made the laser beam slide over the surface of the metal without achieving deep welding\(^30\). Consequently, the spot diameter and pulse duration in this study were determined as 1 mm and 8 ms, respectively, in consideration of these factors. To compare two different penetration depths of laser-welded alloy castings (peripheral and full depths of laser-welded joints), two laser voltage conditions, 220 V and 260 V, were selected.

Although some authors reported on reduced mechanical strength of laser-welded CP Ti or titanium alloys\(^26,27\), our study showed that the strength of laser-welded Ti-6Al-7Nb alloy and CP Ti joints could be as strong as the control specimens under the present conditions, which was also found in other studies\(^26,27,30,36,45,50\). In this investigation, all 260 V laser-welded Ti-6Al-7Nb alloy specimens fractured outside the welded joints, which revealed that the laser-welded joints were at least as strong as the unwelded parts of the same alloy castings. Therefore, the mechanical strength of 260 V laser-welded Ti-6Al-7Nb alloy castings was as high as that of the control specimens.

The 220 V laser-welded Ti-6Al-7Nb alloy specimens, which fractured within the welded joint, showed fracture characteristics of dimples formed by microvoid coalescence (Fig. 3-1A), whereas the 260 V laser-welded Ti-6Al-7Nb alloy specimen, which fractured outside the welded joint, showed cup-like dimples, typical of ductile fracture characteristics (Fig. 3-2A). By metallographic observation, the heat-affected zone was conspicuous in the laser-welded Ti-6Al-7Nb alloy specimens and very fine a structure in the fusion zone could be induced by the rapid solidification of molten alloy.

Regardless of laser voltage, there were no significant differences in mechanical strength between the laser-welded CP Ti specimens and the control specimens. Although the mechanical strength of the 220 V specimens was as high as that of the 260 V or control specimens, the elongation was lower than that of the other two. Laser-welded CP Ti specimens showed the same fracture characteristics as the laser-welded Ti-6Al-7Nb alloy specimens (Fig. 4). The different fracture characteristics and reduced cross-sectional area of the peripherally laser-welded specimens seemed to result in lower elongation. The metallographs showed acicular a-prime structure in the fusion zone, resulting from martensitic transformation during rapid solidification, and coarse a grain structure in the unaffected zone due to slow cooling process as commonly found in as-cast CP Ti\(^30\). No clear heat-affected zones could be identified, an observation similar to previous reports\(^26,30\).

Due to the high affinity of CP Ti and titanium alloys for oxygen and other elements, the influence of impurities is inevitable while operating the laser welding machine in dental laboratories. Therefore, the microstructural changes observed in this study were brought about not only by the rapid
solidification of the molten metals but also due to the presence of impurities, leading to increased hardness and decreased ductility in the welded metals.\(^\text{52,57,30-36}\)

Unlike Ti-6Al-7Nb alloy and CP Ti specimens, all laser-welded Co-Cr alloy specimens were fractured within the welded joints with low mechanical strength and elongation even though full depth of joint was laser-welded, which coincided with a previous report.\(^\text{57}\) From the macroscopic observation of the laser-welded surface, cracks in the welded area were found only in Co-Cr alloy specimens regardless of laser voltage (Fig. 2-3). This could be one of the reasons for the early failure of the laser-welded Co-Cr alloy specimens. These cracks were reportedly attributed to residual stress induced by the rapid cooling of molten Co-Cr alloy.\(^\text{3,6}\) Bubble-like surfaces were also observed at the periphery of the laser-welded area of Co-Cr alloy specimens. According to a previous report, a steep rise in temperature occurred and reached the vaporization point at the surface by laser irradiation. Due to lower boiling point and heat of vaporization of Co and Cr than those of Ti, metal vapor is thought to be produced more easily in welded Co-Cr alloy. In addition, the metal vapor appeared to be trapped during the molten

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**Fig. 6** Metallographic pictures of three laser-welded alloy castings of each metal at (1) 220 V and (2) 260 V. The arrow indicates the original gap between two halves of specimen without being welded. (a) fusion zone; (3) higher magnification view of fusion zone; (b) unaffected zone; (c) porosity; and (d) heat-affected zone. Cracks are present in the Co-Cr alloy specimens (triangle mark).
metal solidification due to the higher viscosity of Co-Cr alloy than CP Ti and Ti-6Al-7Nb alloy.

It has been stated that porosity in welded alloys reduced the strength of laser-welded joints. Therefore, not only cracks but also porosity could cause the early fracture of laser-welded Co-Cr alloy specimens. Furthermore, it has been reported that increased porosity caused by higher laser power could impair the mechanical strength of laser-welded joints, which corresponded to the porosity found in the 260 V laser-welded Co-Cr alloy specimens. From the fractographs, the cleavage pattern indicated brittle fracture characteristics for all laser-welded Co-Cr alloy specimens, while fine precipitates in the fusion zone could be induced by rapid solidification.

In this study, the mechanical strength of laser-welded Ti-6Al-7Nb alloy and CP Ti specimens was comparable to that of the control specimens. However, reduced ductility of the welded joints could be expected. Although there was a small difference in mechanical strength between the two groups of laser-welded Ti-6Al-7Nb alloy specimens, the fully laser-welded depth should be considered, because the reduction in elongation was much larger than that in mechanical strength. Nevertheless, increase in penetration depth by increasing irradiation energy does not always improve the mechanical strength. On the contrary, more detrimental effects such as cracks, concavity, porosity, or distortion could be induced by higher laser energy, thereby affecting unfavorably the mechanical strength of laser-welded joints.

For clinical application, it is very important to select the suitable laser parameters according to the types of metal used and the thickness of the work piece in order to achieve reliable mechanical strength in laser-welded dental alloy prostheses.

CONCLUSIONS

Within the limitations of the current study, the following conclusions were drawn:

1. Ti-6Al-7Nb alloy and CP Ti castings with fully laser-welded depth (260 V condition) showed high mechanical strength and elongation comparable to the control specimens.

2. Ti-6Al-7Nb alloy and CP Ti castings with peripherally laser-welded depth (220 V condition) showed significantly lower elongation than the control specimens.

3. The mechanical strength and elongation of laser-welded Co-Cr alloy castings were significantly low.

4. Fractographs of laser-welded Ti-6Al-7Nb alloy and CP Ti castings showed ductile characteristics, while those of laser-welded Co-Cr alloy castings exhibited brittle characteristics.

5. Cracks induced by laser welding were found in laser-welded Co-Cr alloy castings, but they were not noticeable in laser-welded Ti-6Al-7Nb alloy and CP Ti castings.

6. Microstructural changes had a great influence on fracture characteristics, which were closely related to the mechanical properties of laser-welded dental castings.

ACKNOWLEDGEMENTS

The authors would like to thank Mr. Hisashi Matsubara, Dental Hospital, Tokyo Medical and Dental University, Tokyo, Japan, for his technical assistance.

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