Retraction: Effects of welding pulse energy and fluoride ion on the cracking susceptibility and fatigue behavior of Nd:YAG laser-welded cast titanium joints

Her-Hsiung HUANG¹, Mau-Chin LIN², Chien-Chan LIN³, Sheng-Chieh LIN⁴, Chii-Chih HSU⁵, Fang-Lung CHEN⁵, Shyh-Yuan LEE¹ and Chun-Cheng HUNG⁵

¹ Faculty of Dentistry, School of Dentistry, National Yang-Ming University, Taipei 112, Taiwan
² Department of Dental Laboratory Technology, Central Taiwan University of Science and Technology, Taichung 406, Taiwan
³ Institute of Oral Materials Science, Chung Shan Medical University, Taichung 402, Taiwan
⁴ Department of Dental Laboratory Technology, Shu-Zen College of Medicine and Management, Kaohsiung 821, Taiwan
⁵ Graduate Institute of Dental Sciences, Kaohsiung Medical University, Kaohsiung 807, Taiwan

doi:10.4012/dmj.25.632-e  JOI JST.JSTAGE/dmj/25.632

This article has been retracted by the Editorial Board of Dental Materials Journal due to violation of our publishing policies and procedures as of December 1, 2012.
Effects of Welding Pulse Energy and Fluoride Ion on the Cracking Susceptibility and Fatigue Behavior of Nd:YAG Laser-welded Cast Titanium Joints

Her-Hsiung HUANG¹, Mau-Chin LIN², Chien-Chan LIN³, Sheng-Chieh LIN⁴, Chi-Chih HSU⁵, Fang-Lung CHEN⁶, Shyh-Yuan LEE⁷ and Chun-Cheng HUNG⁸

¹Faculty of Dentistry, School of Dentistry, National Yang-Ming University, Taipei 112, Taiwan
²Department of Dental Laboratory Technology, Central Taiwan University of Science and Technology, Taichung 406, Taiwan
³Institute of Oral Materials Science, Chung Shan Medical University, Taichung 402, Taiwan
⁴Department of Dental Laboratory Technology, Shu-Zen College of Medicine and Management, Kaohsiung 821, Taiwan
⁵Graduate Institute of Dental Sciences, Kaohsiung Medical University, Kaohsiung 807, Taiwan

Corresponding author, Chun-Cheng Hung E-mail: biomaterial@msn.com

Received March 6, 2006/Accepted August 18, 2006

In this study, the cracking susceptibility and fatigue behavior of Nd:YAG laser-welded cast Ti joints (welding pulse energy: 11, 15, and 18 J) in fluoride-containing (0 and 0.5% NaF) artificial saliva were evaluated using constant elongation rate test (CERT) and fatigue test (FT), respectively. Both CERT and FT were also carried out in open air as controls. Results showed that increasing the welding energy increased the elongation and fatigue life, but decreased the tensile strength, of cast Ti joints in open-air environment. With a welding energy of 11 J, the fluoride ions in the artificial saliva increased the cracking susceptibility and decreased the fatigue life of Ti joints. When the welding energy exceeded 15 J, the presence of fluoride ions still increased the cracking susceptibility, but did not reduce the fatigue life of Ti joints. Rupture of Ti joints — if it occurred — occurred only at the welded metal (versus the non-welded part).

Key words: Welding pulse energy, Fluoride, Mechanical property

INTRODUCTION

Commercial pure titanium (Ti) is widely used for dental applications because of its excellent biocompatibility and mechanical properties. Clinical experience has revealed that the accuracy of final Ti prostheses might change because of some unavoidable errors during the fabrication process. Therefore, clinicians have often tried to make up for these errors by cutting and re-connecting (welding) Ti dental prostheses. Among the various welding techniques, laser welding in an argon gas environment is frequently used to re-connect Ti prostheses. Several reports have focused on the microstructure¹⁻² or mechanical properties³⁻⁷ of laser-welded Ti joints in the open-air environment. However, from the dental application point of view, the cracking susceptibility and fatigue behavior of laser-welded Ti prostheses in the oral environments should be of greater importance — but there is very limited related information in the literature.

In the oral environment, fluoride-containing commercial mouthwashes, toothpastes, and prophylactic gels are widely used to prevent dental caries or reduce dental sensitivity. However, the detrimental effects of fluoride ions on the corrosion resistance of static Ti or Ti alloys have been extensively reported⁸⁻¹⁰. Fluoride ions are found to be very aggressive toward the protective passive film (normally composed of TiO₂) formed on the surfaces of Ti and Ti alloys. On the other hand, as the outermost surface of laser-welded Ti joints still contains the protective oxide film, fluoride-enhanced corrosion of welded Ti joints may also occur. Based on metallurgical concept, deterioration in the surface film could influence the cracking and/or fatigue behavior of stressed Ti or Ti alloys in a fluoride-containing environment. Nonetheless, such information is also limited in the literature.

The aim of this study, therefore, was to determine the influence of Nd:YAG laser welding pulse energy on the cracking susceptibility and fatigue life of cast Ti joints in different environments — open air and artificial saliva with and without fluoride addition.

MATERIALS AND METHODS

Materials preparation

A Ti casting machine (Vulcan-T, Shofu, Kyoto, Japan) was used to produce Grade 2 commercially pure Ti (Titan, Shofu, Kyoto, Japan) dumbbell rods with a 3-mm central diameter and 18-mm gauge length (as per American Dental Association (ADA) Specification No.14). A MgO-based investment (Titavest CB, J. Morita, Osaka, Japan) was used with a mixture of 16 mL liquid and 100 g powder according to the manufacturer’s instructions, and maintained a temperature of 500°C before casting.

Each cast Ti dumbbell rod was cut into two even...
pieces by a fine cutting machine in a direction perpendicular to the longitudinal direction. Two pieces of Ti rods of uniform length were put together on the platform in an Nd:YAG laser welding machine (LaserStar, Bego, Bremen, Germany). The laser beam was aimed perpendicular to the metal rod under an argon gas environment. A 0.8-mm diameter laser beam with a 10-ms pulse duration was chosen in this study. Three welding voltages of 310, 340, and 360 V were used for welding, which corresponded to the pulse energies of about 11, 15, and 18 J, respectively, according to the manufacturer’s instructions. Two diametrically opposed points were tack-welded to ensure initial fixation and to compensate for the stress concentration that arises with joint welding. The remaining welds overlapped the previous welds by approximately 50%.

Fig. 1 shows a schematic diagram of the Nd:YAG laser-welded cast Ti joint. Before the mechanical tests that followed, specimens were ultrasonically cleaned in an alcohol bath and then rinsed with distilled water.

After completing the laser welding procedure, radiographs of the joints were obtained with an ultra-speed Kodak occlusal dental film (DF-50, Eastman Kodak Co., Rochester, New York, USA) to verify the presence of any eventual voids (resolution: few μm). A dental X-ray machine (Oralix AC, Dentsply Italia, Gendex Division, Milano, Italy) was set at 65 kV (peak), 7.5 mA, and with 0.9-second exposure time. Fig. 2 shows the radiographs of the laser-welded cast Ti dumbbell rods with different welding pulse energies. Note that no interval voids were observed.

Fig. 1 Schematic diagram of an Nd:YAG laser-welded cast Ti joint.

Fig. 2 Radiographs of Nd:YAG laser-welded cast Ti joints produced at different welding pulse energies: (a) 11 J; (b) 15 J; (c) 18 J.

Cracking susceptibility and fatigue behavior analyses
To evaluate the cracking susceptibility of the cast Ti joints at various welding pulse energies, a custom-made tensile test machine was used to carry out the constant elongation rate test (CERT) with a slow elongation rate of 10⁻³ mm/s in three different environments: open air and NaF-containing (0 and 0.5%) artificial saliva at 37°C and pH 5.6. The artificial saliva contained NaCl (400 mg/L), KCl (400 mg/L), CaCl₂·2H₂O (795 mg/L), NaH₂PO₄·H₂O (690 mg/L), KSCN (300 mg/L), Na₅·9H₂O (5 mg/L), and urea (1000 mg/L). Furthermore, CERT was also performed for the non-welded Ti dumbbell rod in the open air as a control. Five specimens were used for every CERT condition.

For fatigue behavior analysis, a custom-made fatigue test machine was used to carry out the fatigue test (FT) with a frequency of 3 Hz to evaluate the fatigue behavior of the cast Ti joints welded under different welding pulse energies. To simulate the clinical biting situation, a sine waveform with a maximum load of 40 kg and a minimum load of 0 kg was used for FT. In addition, FT environments were the same as those used for CERT. Five specimens were used for every FT condition.

It should be noted that CERT was carried out until the test specimens fractured. In other words, regardless of the welding pulse energy applied, all the test specimens ruptured after CERT. On the other hand, FT was stopped when the number of fatigue cycles reached 10⁶ even if the test specimens still did not fracture. After CERT and FT, the fracture surfaces of the tested cast Ti joints were observed using a scanning electron microscope (SEM) (S-3000N, Hitachi, Tokyo, Japan) with energy dispersive spectroscopy (EDS) (EX-200, Horiba, Kyoto, Japan). Compounds on the fracture surfaces were also identified using an X-ray photoelectron spectrometer (XPS) (ESCALAB 210, VG Scientific Ltd., East Grinstead, UK).

Statistical analysis
After CERT and FT in all test environments, the cracking susceptibility (i.e., tensile strength and elongation percentage) and fatigue life (i.e., number of cycles prior to fracture) of laser-welded cast Ti joints were statistically analyzed using two-way analysis of variance (ANOVA) to analyze the factors of welding pulse energy and test environment. Tukey’s test
\( \alpha = 0.05 \) was chosen as the following multiple comparison technique when necessary.

**RESULTS**

Fig. 3 shows the overall fracture morphologies of laser-welded cast Ti joints produced at different welding pulse energies after CERT in air. After sequential grinding, polishing and etching processes, optical micrographs of the weld zone indicated by arrows in Figs. 3(a)–(c) are manifested in Figs. 4(a)–(c) respectively. As shown in Fig. 3, all test Ti joints fractured at the weld zone location after CERT regardless of the welding pulse energy. The welded area on fractured cross-section of Ti joints was calculated using an Image-Pro® Plus image analysis software (version 4.5.1) (Media Cybernetics Inc., Silver Spring, MD, USA) and listed as follows: 2.4 ± 0.04 mm² for 11 J, 3.3 ± 0.22 mm² for 15 J, and 4.3 ± 0.07 mm² for 18 J. It was obvious that increasing the welding pulse energy led to an increase in the welded area (but still lower than the non-welded cross-sectional area of 7.1 mm²). Optical micrographs of the weld zone (Fig. 4) indicated that the weld zone had a uniform lamellar/acicular structure that became coarser with increase in the welding pulse energy.

Table 1 shows the tensile strengths and elongation percentages of cast Ti joints with various
Nd:YAG laser welding pulse energies after CERT in different environments. Note that fracture in all of the test Ti joints occurred only at the weld zone after CERT. Therefore, tensile strength was defined as the ratio of the maximum load (during CERT) to the welded area on fractured cross-section of Ti joints. Regardless of the test environment, increasing the welding pulse energy of cast Ti joints led to an increase in elongation, but to a decrease in tensile strength. For the same welding pulse energy, the artificial saliva (especially the fluoride-containing environment) increased the cracking susceptibility, i.e., decreased the tensile strength and elongation of Ti joints, as compared to the open-air environment.

Fig. 5 shows the fracture morphologies of cast Ti joints (welded at 15 and 18 J) after CERT in artifi-

Table 1 Mechanical properties, including tensile strength and elongation percentage, of cast Ti joints with various Nd:YAG laser welding pulse energies after the constant elongation rate test in different environments (note: standard deviations are given in parentheses)

<table>
<thead>
<tr>
<th>Pulse energy (J)</th>
<th>Environment</th>
<th>Air</th>
<th>Artificial saliva</th>
<th>Artificial saliva+ 0.5% NaF</th>
</tr>
</thead>
<tbody>
<tr>
<td>0*</td>
<td></td>
<td>440 (12)</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>11</td>
<td></td>
<td>581 (14)</td>
<td>572 (19)</td>
<td>538 (13)</td>
</tr>
<tr>
<td>15</td>
<td></td>
<td>563 (23)</td>
<td>552 (11)</td>
<td>447 (35)</td>
</tr>
<tr>
<td>18</td>
<td></td>
<td>543 (21)</td>
<td>515 (15)</td>
<td>387 (9)</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Pulse energy (J)</th>
<th>Environment</th>
<th>Air</th>
<th>Artificial saliva</th>
<th>Artificial saliva+ 0.5% NaF</th>
</tr>
</thead>
<tbody>
<tr>
<td>0*</td>
<td></td>
<td>27.51 (1.63)</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>11</td>
<td></td>
<td>3.80 (0.30)</td>
<td>2.90 (0.15)</td>
<td>2.22 (0.22)</td>
</tr>
<tr>
<td>15</td>
<td></td>
<td>5.05 (0.23)</td>
<td>4.25 (0.13)</td>
<td>4.04 (0.28)</td>
</tr>
<tr>
<td>18</td>
<td></td>
<td>8.27 (0.28)</td>
<td>6.42 (0.35)</td>
<td>5.80 (0.25)</td>
</tr>
</tbody>
</table>

Fig. 5 Fracture morphologies of laser-welded cast Ti joints (15 and 18 J) after constant elongation rate test in artificial saliva with and without 0.5% NaF addition.
cial saliva with and without 0.5% NaF. Joints welded with a smaller welding pulse energy presented a more-brittle fracture appearance. However, no significant differences in the fracture appearance were observed for Ti joints with the same welding pulse energy after CERT in artificial saliva with and without NaF. Fig. 6 shows the XPS analysis results of the fractured weld zone surface of a cast Ti joint (15 J) after CERT in 0.5% NaF-containing artificial saliva, revealing the presence of a Ti-F compound, $\text{Na}_2\text{TiF}_6$. Similar results, showing the presence of Ti-F compound on fractured surface, were also obtained for the laser-welded Ti joints (11 J) after FT in fluoride-containing artificial saliva (not shown).

Table 2 shows the mean fatigue life (in number of cycles) prior to the failure of the cast Ti joints with various Nd:YAG laser welding pulse energies after FT in different environments. The results showed that at higher welding pulse energies (15 and 18 J), the fatigue life of cast Ti joints exceeded $10^6$ cycles when tested in both air and corrosive solutions. Conversely, at the lower welding pulse energy (11 J), the presence of NaF in the artificial saliva decreased the fatigue life of Ti joints compared to that tested in either air or fluoride-free artificial saliva. It is noted that fracture in all of the test Ti joints occurred only at the welded metal after FT, if it occurred.

Fig. 7 shows the fracture morphologies of cast Ti joints (11 J) after FT in air (a) and in 0.5% NaF artificial saliva (b). Figs. 7(c) and 7(d) are higher magnifications of (a) and (b) respectively. At the lower magnification (Figs. 7(a) and (b)), no significant differences in fracture appearance were observed for Ti joints welded at a pulse energy of 11 J after FT in air and fluoride-containing solution. These samples show a brittle morphology on the fracture surface. Higher SEM magnification of Figs. 7(a) and (b) revealed that typical fatigue striations could be observed on the fractured surface of the Ti joints (Figs. 7(c) and (d)).

Results of two-way ANOVA for the fatigue be-

<table>
<thead>
<tr>
<th>Pulse energy (J)</th>
<th>Environment</th>
<th>Air</th>
<th>Artificial saliva</th>
<th>Artificial saliva+ 0.5% NaF</th>
</tr>
</thead>
<tbody>
<tr>
<td>11</td>
<td>1.2×10^6 (2.2×10^6)</td>
<td>3.7×10^4 (6.5×10^4)</td>
<td>1.9×10^4 (7.0×10^4)</td>
<td></td>
</tr>
<tr>
<td>15</td>
<td>&gt;10^6</td>
<td>&gt;10^6</td>
<td>&gt;10^6</td>
<td></td>
</tr>
<tr>
<td>18</td>
<td>&gt;10^6</td>
<td>&gt;10^6</td>
<td>&gt;10^6</td>
<td></td>
</tr>
</tbody>
</table>

Conversely, at the lower welding pulse energy (11 J), the presence of NaF in the artificial saliva decreased the fatigue life of Ti joints compared to that tested in either air or fluoride-free artificial saliva. It is noted that fracture in all of the test Ti joints occurred only at the welded metal after FT, if it occurred.

Fig. 7 shows the fracture morphologies of cast Ti joints (11 J) after FT in air (a) and in 0.5% NaF artificial saliva (b). Figs. 7(c) and 7(d) are higher magnifications of (a) and (b) respectively. At the lower magnification (Figs. 7(a) and (b)), no significant differences in fracture appearance were observed for Ti joints welded at a pulse energy of 11 J after FT in air and fluoride-containing solution. These samples show a brittle morphology on the fracture surface. Higher SEM magnification of Figs. 7(a) and (b) revealed that typical fatigue striations could be observed on the fractured surface of the Ti joints (Figs. 7(c) and (d)).

Results of two-way ANOVA for the fatigue be-

Table 2 Mean fatigue life (in number of cycles) prior to the failure of cast Ti joints with various Nd:YAG laser welding pulse energies after the constant elongation rate test in different environments (note: standard deviations are given in parentheses)

<table>
<thead>
<tr>
<th>Pulse energy (J)</th>
<th>Environment</th>
<th>Air</th>
<th>Artificial saliva</th>
<th>Artificial saliva+ 0.5% NaF</th>
</tr>
</thead>
<tbody>
<tr>
<td>11</td>
<td>1.2×10^6 (2.2×10^6)</td>
<td>3.7×10^4 (6.5×10^4)</td>
<td>1.9×10^4 (7.0×10^4)</td>
<td></td>
</tr>
<tr>
<td>15</td>
<td>&gt;10^6</td>
<td>&gt;10^6</td>
<td>&gt;10^6</td>
<td></td>
</tr>
<tr>
<td>18</td>
<td>&gt;10^6</td>
<td>&gt;10^6</td>
<td>&gt;10^6</td>
<td></td>
</tr>
</tbody>
</table>

Conversely, at the lower welding pulse energy (11 J), the presence of NaF in the artificial saliva decreased the fatigue life of Ti joints compared to that tested in either air or fluoride-free artificial saliva. It is noted that fracture in all of the test Ti joints occurred only at the welded metal after FT, if it occurred.

Fig. 7 shows the fracture morphologies of cast Ti joints (11 J) after FT in air (a) and in 0.5% NaF artificial saliva (b). Figs. 7(c) and 7(d) are higher magnifications of (a) and (b) respectively. At the lower magnification (Figs. 7(a) and (b)), no significant differences in fracture appearance were observed for Ti joints welded at a pulse energy of 11 J after FT in air and fluoride-containing solution. These samples show a brittle morphology on the fracture surface. Higher SEM magnification of Figs. 7(a) and (b) revealed that typical fatigue striations could be observed on the fractured surface of the Ti joints (Figs. 7(c) and (d)).

Results of two-way ANOVA for the fatigue be-

<table>
<thead>
<tr>
<th>Pulse energy (J)</th>
<th>Environment</th>
<th>Air</th>
<th>Artificial saliva</th>
<th>Artificial saliva+ 0.5% NaF</th>
</tr>
</thead>
<tbody>
<tr>
<td>11</td>
<td>1.2×10^6 (2.2×10^6)</td>
<td>3.7×10^4 (6.5×10^4)</td>
<td>1.9×10^4 (7.0×10^4)</td>
<td></td>
</tr>
<tr>
<td>15</td>
<td>&gt;10^6</td>
<td>&gt;10^6</td>
<td>&gt;10^6</td>
<td></td>
</tr>
<tr>
<td>18</td>
<td>&gt;10^6</td>
<td>&gt;10^6</td>
<td>&gt;10^6</td>
<td></td>
</tr>
</tbody>
</table>

Conversely, at the lower welding pulse energy (11 J), the presence of NaF in the artificial saliva decreased the fatigue life of Ti joints compared to that tested in either air or fluoride-free artificial saliva. It is noted that fracture in all of the test Ti joints occurred only at the welded metal after FT, if it occurred.

Fig. 7 shows the fracture morphologies of cast Ti joints (11 J) after FT in air (a) and in 0.5% NaF artificial saliva (b). Figs. 7(c) and 7(d) are higher magnifications of (a) and (b) respectively. At the lower magnification (Figs. 7(a) and (b)), no significant differences in fracture appearance were observed for Ti joints welded at a pulse energy of 11 J after FT in air and fluoride-containing solution. These samples show a brittle morphology on the fracture surface. Higher SEM magnification of Figs. 7(a) and (b) revealed that typical fatigue striations could be observed on the fractured surface of the Ti joints (Figs. 7(c) and (d)).

Results of two-way ANOVA for the fatigue be-

Table 2 Mean fatigue life (in number of cycles) prior to the failure of cast Ti joints with various Nd:YAG laser welding pulse energies after the constant elongation rate test in different environments (note: standard deviations are given in parentheses)

<table>
<thead>
<tr>
<th>Pulse energy (J)</th>
<th>Environment</th>
<th>Air</th>
<th>Artificial saliva</th>
<th>Artificial saliva+ 0.5% NaF</th>
</tr>
</thead>
<tbody>
<tr>
<td>11</td>
<td>1.2×10^6 (2.2×10^6)</td>
<td>3.7×10^4 (6.5×10^4)</td>
<td>1.9×10^4 (7.0×10^4)</td>
<td></td>
</tr>
<tr>
<td>15</td>
<td>&gt;10^6</td>
<td>&gt;10^6</td>
<td>&gt;10^6</td>
<td></td>
</tr>
<tr>
<td>18</td>
<td>&gt;10^6</td>
<td>&gt;10^6</td>
<td>&gt;10^6</td>
<td></td>
</tr>
</tbody>
</table>

Conversely, at the lower welding pulse energy (11 J), the presence of NaF in the artificial saliva decreased the fatigue life of Ti joints compared to that tested in either air or fluoride-free artificial saliva. It is noted that fracture in all of the test Ti joints occurred only at the welded metal after FT, if it occurred.

Fig. 7 shows the fracture morphologies of cast Ti joints (11 J) after FT in air (a) and in 0.5% NaF artificial saliva (b). Figs. 7(c) and 7(d) are higher magnifications of (a) and (b) respectively. At the lower magnification (Figs. 7(a) and (b)), no significant differences in fracture appearance were observed for Ti joints welded at a pulse energy of 11 J after FT in air and fluoride-containing solution. These samples show a brittle morphology on the fracture surface. Higher SEM magnification of Figs. 7(a) and (b) revealed that typical fatigue striations could be observed on the fractured surface of the Ti joints (Figs. 7(c) and (d)).

Results of two-way ANOVA for the fatigue be-

Table 2 Mean fatigue life (in number of cycles) prior to the failure of cast Ti joints with various Nd:YAG laser welding pulse energies after the constant elongation rate test in different environments (note: standard deviations are given in parentheses)

<table>
<thead>
<tr>
<th>Pulse energy (J)</th>
<th>Environment</th>
<th>Air</th>
<th>Artificial saliva</th>
<th>Artificial saliva+ 0.5% NaF</th>
</tr>
</thead>
<tbody>
<tr>
<td>11</td>
<td>1.2×10^6 (2.2×10^6)</td>
<td>3.7×10^4 (6.5×10^4)</td>
<td>1.9×10^4 (7.0×10^4)</td>
<td></td>
</tr>
<tr>
<td>15</td>
<td>&gt;10^6</td>
<td>&gt;10^6</td>
<td>&gt;10^6</td>
<td></td>
</tr>
<tr>
<td>18</td>
<td>&gt;10^6</td>
<td>&gt;10^6</td>
<td>&gt;10^6</td>
<td></td>
</tr>
</tbody>
</table>
behavior showed that both welding pulse energy and test environment had statistically significant influences on fatigue life (welding pulse energy: $P < 0.0001$; test environment: $P < 0.01$). Further comparison with Tukey’s test revealed that 11 J was significantly different from 15 J and 18 J (factor: welding pulse energy); however, there were no significant differences between the fluoride-free and fluoride-containing solutions (factor: test environment). Furthermore, in terms of cracking susceptibility, both welding pulse energy and test environment also had statistically significant influences on tensile strength ($P < 0.0001$) and elongation ($P < 0.0001$). Tukey’s test revealed that for the factor of welding pulse energy, all the three kinds of welding energy were significantly different; likewise, for the test environment factor, the fluoride-free and fluoride-containing solutions were significantly different.

DISCUSSION

From the micrographs of weld zones shown in Figs. 3 and 5, it could be seen that the laser welding process did not create a significant heat-affected zone (HAZ) in the joint neighborhood. It has been reported that laser welding is a fast procedure with quasi-instantaneous metal heating and cooling (up to $10^5 \degree C/s$)[14], and hence no significant HAZ is formed. Similar results have been reported previously[2,15].

Studies by Wiskott et al.[2,16] showed that the presence of a fine acicular structure can provide the welded metal with higher cracking resistance. Therefore, as shown in Table 1, a decrease in tensile strength as the welding pulse energy increased was due to the coarsening of acicular structure in the weld zone with increasing welding pulse energy (Fig. 4). On the other hand, the tensile strength of laser-welded Ti joints with different welding pulse energies (581 MPa for 11 J; 563 MPa for 15 J; 543 MPa for 18 J) was higher than that of the non-welded cast Ti (440 MPa), although the fracture of laser-welded Ti joints occurred only at the point of welding after CERT. This was related to the fact that the cross-sectional area of the fractured weld zone of Ti joints with different welding pulse energies (2.4 mm$^2$ for 11 J; 3.3 mm$^2$ for 15 J; 4.3 mm$^2$ for 18 J) was much smaller than that of non-welded cast Ti (7.1 mm$^2$). In other words, during CERT, the weld zone with a smaller cross-sectional area would endure a higher stress with respect to the non-welded region (i.e., base metal) of Ti joints. Therefore, it is expected that the fracture location of Ti joints with a higher
welding pulse energy (>18 J) may occur at the non-welded region, instead of the weld zone, when a higher proportion of the cross-sectional area of the joint is welded. However, this needs further investigations. The above statement could also explain the finding that the fracture location occurred at the weld zone of Ti joints (11 J) after FT in various test environments.

On the other hand, the presence of Na$_2$TiF$_6$ on the fractured surface of cast Ti joint after CERT in acidic fluoride-containing environment (Fig. 6) implied that fluoride ions could destroy the protective passive film on Ti weld zone via the formation of Ti-F compound. In an acidic medium, NaF can induce hydrofluoric acid (HF)\textsuperscript{15,16}. Then, HF reacts with titanium oxides and locally dissolves the protective oxide layer by forming Ti-F compound on Ti surface\textsuperscript{13,19}. In other words, fluoride ions can form a soluble complex with titanium ions derived from the protective titanium oxide layer. Without the protective oxide layer, acid corrosion can take place and Ti metal surface will be locally attacked. This might result in the acceleration of crack initiation on Ti weld zone during CERT and FT. As the crack propagated inwards to the weld zone, a new crack tip would be generated and then attacked by the acidic fluoride solution. This process would occur repeatedly during crack propagation, with the former process assisting the latter. Therefore, the presence of 0.5% NaF in acidic artificial saliva was detrimental to the mechanical properties of cast Ti joints under the same welding pulse energy, leading to decreases in tensile strength, elongation, and fatigue life (Tables 1 and 2).

In this study, Tukey’s test revealed that no significant differences were found between the fluoride-free and fluoride-containing artificial saliva for the fatigue life of laser-welded cast Ti joints. Similar results were also obtained by Zavanelli et al.\textsuperscript{20}. In this study, however, the presence of fluoride ions in artificial saliva significantly increased the cracking susceptibility, i.e., decreased the tensile strength and elongation, of laser-welded cast Ti joints.

From the fracture morphologies of cast Ti joints after CERT in artificial saliva with and without fluoride (Fig. 5), a more-brittle fracture morphology of Ti joints at the lower welding pulse energy was related to the presence of a finer lamellar/acicular structure in the weld zone as compared to Ti joints of higher welding pulse energies, as shown in Fig. 4. On the other hand, from the fracture morphologies of cast Ti joints after FT (Fig. 7), a slightly more-brittle fracture morphology was presented on the Ti joint tested in the 0.5% NaF environment compared to that tested in air. This was thought to have been due to the fluoride-enhanced corrosion occurring at the crack tips of the Ti joint during FT in the fluoride-containing environment as mentioned earlier. This assumption was supported by the detection of a Ti-F compound by XPS on the fractured weld zone surface after FT in the 0.5% NaF-containing artificial saliva. This also explained the decrease in fatigue life of laser-welded cast Ti joints (11 J) in the fluoride-containing solution compared to that tested in fluoride-free solution.

Note that some large pores (some nearly 100 μm in diameter) were observed along the welded fusion line on the fracture surface of cast Ti joints shown in Figs. 3, 5, and 7. This might suggest gas trapping as a consequence of the continuous argon gas spray throughout the Nd:YAG laser welding procedure. The presence of large pores in laser-repaired Ti sections has been reported previously\textsuperscript{25} and appears to be the most important factor in controlling the strength of the welded joint. On the other hand, larger pores (around a few tens to hundreds μm in diameter) observed along the welded fusion line of the weld zone with the higher welding pulse energy seemed to have no detrimental influence on the breaking susceptibility of Ti joints tested in open air\textsuperscript{21}. In this study, based on the CERT and FT results (Tables 1 and 2), the presence of large pores in the weld zone did not exert a significant influence on both the crack susceptibility and fatigue behavior of cast Ti joints in any test environment.

It is important to note that the environment is a critical factor in determining fatigue properties. Thus, fatigue analyses, which are typically carried out in air at room temperature, are not always relevant to the practical conditions in the oral cavity\textsuperscript{21,22}. Pröbster et al.\textsuperscript{23} demonstrated that acidic fluoride agents can cause severe surface damage to Ti, while a NaF-containing environment with a neutral acidity does not cause any damage. In other words, acidity of the solution is more important than the fluoride ion concentration. Therefore, from the perspective of clinical relevance, there should be correct and representative simulation of the oral environment for in vitro dental studies. In this study, the prepared fluoride-containing artificial saliva had a pH of 5.6 and 0.5% NaF (around 2400 ppm of fluoride ions), simulating a slightly acidic oral environment and the use of fluoride-containing toothpastes or dental prophylactic gels.

The corrosion resistance of Ti is highly dependent on the stability of the surface oxide film, and the presence of fluoride ions reduces the protective surface oxide film\textsuperscript{24}. Lucas and Lemons\textsuperscript{25} reported that this oxide film on Ti provides corrosion resistance under static conditions, but is not sufficiently stable under stressed conditions. Könnem et al.\textsuperscript{26} reported that Ti is susceptible to hydrogen damage, and that crack propagation is relatively fast in the stressed specimens in a fluoride-containing solution. A previous report also showed a reduction in the fatigue life of Ti in wet environments as compared to that in
open-air environment\textsuperscript{20}. However, there is no detailed information regarding the corrosion cracking and corrosion fatigue properties of cast Ti after laser welding. The results of this study showed an increase in cracking susceptibility and a reduction in fatigue life of laser-welded cast Ti joints in the presence of a corrosive solution, especially one containing fluoride ions.

It is estimated that the number of deflections (due to the removal from mouth and adjustment into position) for removable partial denture frameworks is probably below 1500 per year\textsuperscript{21}. Referring to the abovementioned literature\textsuperscript{31} and the data in Table 2, the fatigue life of Nd:YAG laser-welded cast Ti joint at a welding pulse energy of 11 J was estimated to be 13 and 25 years in an oral environment with and without fluoride ions, respectively. On the other hand, for other laser-welded Ti joints at higher welding pulse energies (15 and 18 J), fatigue fracture might not occur in all environments.

CONCLUSION

Welding pulse energy and test environment were found to exert significant influences on the cracking susceptibility and fatigue life of Nd:YAG laser-welded Ti joints. Nd:YAG laser welding procedure at a lower welding pulse energy (11 J) decreased elongation and fatigue life, but increased the tensile strength, of cast Ti joints in all tested media compared to that at higher welding pulse energies (15 and 18 J). Further, when compared to the controls (tested in the open air), artificial saliva negatively affected the cracking susceptibility and fatigue life of cast Ti joints regardless of whether or not it contained 0.5% NaF – but the negative influence was greater in fluoride-containing artificial saliva. This was because fluoride ions could destroy the surface passive film on laser-welded cast Ti joints through the formation of Ti-F compound. Based on the results in this study, we would suggest that the local application of fluoridated dentifrices be abstained from clinical patients with Nd:YAG laser-welded Ti joints produced at a lower welding pulse energy.

ACKNOWLEDGMENTS

The authors would like to thank Chung Shan Medical University, Taiwan for the partial financial support (Grant No. CSMU 92-OM-B-050).

REFERENCES

19) Oda Y, Kawada E, Yoshinari M, Hasegawa K, Okabe T. The influence of fluoride concentration on the corrosion of titanium and titanium alloys. Jpn J Dent...


