The effect of shot blasting and heat treatment on the fatigue behaviour of titanium for dental implant applications

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Short title: Fatigue fracture of shot-blasted titanium

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Abstract
Objectives. The effect of the shot-blasting treatment on the cyclic deformation and fracture behaviour of a commercial pure titanium with two different microstructures; equiaxed (α-phase) and acicular (martensitic α’-phase) has been investigated. Methods. The fatigue tests were carried out in artificial saliva at 37°C. Cyclic deformation tests have been carried out up to fracture and the fatigue-crack nucleation and propagation have been analysed. Residual stresses were determined by means of X-ray diffraction. Results. The results show that the shot blasting treatment improves the fatigue life in the different microstructures studied. The equiaxed-phase has improved mechanical properties compared to the acicular one. Shot blasting, despite it has not exhaustive variable control because of the nature of the treatment, improves the fatigue life by the fact that the initiation site of the fatigue crack changes from the surface of the specimen to the interior of the shot blasted specimen. This is a consequence of the layer of compressive residual stresses that the treatment generates in titanium surfaces. The acicular morphology of the martensite favours the crack propagation along the interface of the α’ plates. Significance. Shot blasting, which is widely used on titanium dental implants in order to favour their osseointegration, can also improve their fatigue resistance.

Keywords
Titanium, dental implants, fatigue resistance, shot blasting, residual stresses
Introduction
The design of an oral implantable device has to always take into consideration the cyclic loading during the life in service of the implant, and therefore the fatigue endurance of the materials used will play a very important role when trying to estimate the long term performance of the device. Thus the assessment of the fatigue behaviour of implantable alloys has been taking greater importance. Wrought cobalt-chromium, Titanium and Ti-6Al-4V alloys show both a similar fatigue endurance, when evaluated by means of rotary bending fatigue tests (about 550 MPa) [1], and when tested in corrosion fatigue in torsion [2]. A relevant aspect is that the elastic modulus of titanium (110 GPa) is about half that of stainless steel (200 GPa) and that of cobalt-chromium-molybdenum alloys (235 GPa). This is a very important point to be taken into account when considering the load transfer into the bone when a dental implant, a joint prosthesis, or an osteosynthesis device is to be designed.

Table 1 summarizes the fatigue endurance for some of the alloys previously discussed. It should be pointed out that the fatigue strength limit is reduced in all cases when the material is tested in saline solution in relation to tests performed in air. It should be also pointed out that both in air and in saline solution; titanium shows the highest fatigue strength limit.

Moreover, Titanium and Titanium alloys can be strengthened and their mechanical properties may be varied by controlling their composition and by means of thermomechanical processing techniques [3-8]. Titanium and Titanium alloys are also resistant to general corrosion, pitting and crevice corrosion, which may occur in other alloys as a result of the aggressive attack of body fluids [9-11].

Because of all these properties, commercially pure Titanium (c.p. Ti) is widely used as dental implant material [12-13]. But clinical success is achieved not only because of implant material but also because of other properties [14]. Among them, one of the most important is surface implant quality, which refers to its mechanical, physicochemical and topographic properties [15-16]. In this sense it is known that an increased implant-surface roughness enhances the in vitro behaviour of osteoblasts [17-19]. Moreover, a better long-term in vivo response is achieved [20-24]. Osseointegration of the implant is meant to be achieved by bone ingrowth into the roughness of the titanium surface [16,25].

Shot blasting is one of the most frequently used treatments for obtaining a rough surface of a dental implant [16, 26]. The materials of the shot particles, which are bombarded on dental implant surface, are chemically stable materials that will not stimulate negative responses of the biological behaviour of the implant [27]. Shot blasting induces a residual stress layer in the treated material because of the local plastic deformation of the metal [28]. Despite the variables of shot blasting are not exhaustively controlled, as for shot peening [29], the stressed superficial layer is in compression and, consequently, an increase in the fatigue resistance of the shot-blasted dental implant is expected.

The influence of the shot blasting treatment on the fatigue and fracture properties of titanium with different microstructures has been studied in this work.
Materials and Methods
The c.p. Ti Grade III used in the present work was kindly donated by Klockner, S.L. The material came as cylindrical rods of 12 mm diameter, forged at 950°C, subsequently annealed at 700°C during 2 hours and then cooled in air. The chemical composition of the alloy is shown in Table 2. The metallographic microstructure corresponds to equiaxial α-grains (Figure 1). Both the composition and the microstructure satisfy the ASTM F67-00 [30] standard for unalloyed Ti for surgical implant applications.

Tensile specimens with a ratio diameter to gauge length of 1/5 and fatigue specimens were machined. A first batch of specimens was kept as the as-received material whilst second and third batches of specimens were heat treated. These specimens were kept during 1 hour in a tubular furnace with argon atmosphere at 1050°C, which is a temperature just above the β-transus for the Ti, and they were then cooled in water at 20°C. The resulting microstructure corresponds to acicular α'-martensite (Figure 2). The specimens of the third and fourth batches were shot blasted producing a roughness surface for the specimens in the two microstructures (equiaxed α-phase and acicular α'-martensite).

The c.p. Ti Grade III discs were shot blasted with particles of Al2O3 and 600 μm in mean size, using 0.2 MPa of blast pressure.

Qualitative surface roughness was observed by scanning electron microscopy (SEM), and quantitative surface roughness was determined by means of a profilometer with a diamond tip. Rₐ and other surface parameters were calculated. X-Ray dispersion energy was used for semiquantitative surface chemical composition measurements.

The hardness distribution in a cross-section of shot blasted specimens was measured using a Vickers microhardness tester with a load of 100 gf and 15 s of indentation.

Residual stresses were measured with a diffractometer incorporating a Bragg-Bentano configuration. The measurements were done for the family of planes (213), which diffractions at 2θ = 139,5°. The elastic constants of Ti at the direction of this family of planes are EC = (E/1+ν)(213) = 90,3 (1,4) GPa. Eleven Ψ angles, 0° and five positive- and five negative-angles were evaluated. The position of the peaks was adjusted with a pseudo-Voigt function using appropriate software (WinplotR), and then converted to interplanar distances (dΨ) using Bragg’s equation. The dΨ vs sen²Ψ graphs and the calculation of the slope of the linear regression (A) were done with appropriate software (Origin). The residual stress is: \( \sigma = EC \times \frac{1}{d_0} \times A \); where \( d_0 \) is the interplanar distance for \( \Psi = 0° \).

The tensile specimens were tested in a universal screwdriven testing machine of 100kN capacity at a cross-head speed of 1mm/min. The fatigue specimens were cyclically deformed in tension-compression under strain control \( R_e = -1 \), in a servohydraulic testing machine of 100kN capacity using a container with artificial saliva at 37°C. The chemical composition of the artificial saliva used is shown in Table 3. The strain rate was always kept constant at 6.5 \( 10^{-3} \) s⁻¹. The total strain amplitude used was +/- 7 \( 10^{-3} \). The deformed and fracture specimens were observed by means of SEM.
Results and Discussion
The surface roughness of the as-machined metal for $\alpha$-phase and $\alpha'$-martensite are $R_a = 0.30 (0.04) \mu m$ and $R_a = 0.33 (0.03) \mu m$, respectively. The shot blasted and $\alpha$-phase specimens have $R_a = 4.2 (0.8) \mu m$, and the shot blasted and $\alpha'$-martensite have $R_a = 3.8 (0.7) \mu m$. As expected, the shot blasted specimens have statistically significant ($p<0.001$; t-Student) and higher surface roughness than the as-machined ones. Non-statistically significant differences in $R_a$-values between equiaxed- and acicular-phase c.p. Ti were found. The roughness obtained (chemical composition and size of shot particles and shot-blasting pressure) for the shot-blasted c.p. Ti was determined as optimal for in vitro and in vivo response in previous works [19,24].

The hardness distribution at the above cross-section of the materials studied is presented in Figure 3. The maximum hardness of 525 (13) HVN and 516 (26) HVN for equiaxed and acicular microstructures, respectively, with shot blasting treatment were measured near the surface. Hardness decreased gradually with increasing distance from the surface. The average hardness of the matrix for the equiaxial phase is 205 (9) HVN and for the acicular phase is 234 (11) HVN; so that the increase in hardness by shot blasting is, approximately, 300 HVN for the two microstructures.

Figure 4 is an example of $d_{\Psi}$ vs $\text{sen}^2 \Psi$ graph for a shot blasted $\alpha$-phase c.p. Ti specimen showing the linear trend of the results with a negative slope. The linearity confirms the validity of the calculated value for the residual stresses and the negative slope indicates that the residual stresses are compressive. Figure 5 shows an example of $d_{\Psi}$ vs $\text{sen}^2 \Psi$ graph for an as-machined $\alpha$-phase c.p. Ti specimen. In this case the results give a two-slope configuration, with a slope for the positive $\Psi$-angles and the other for the negative $\Psi$-angles. This two-slope configuration can be attributed to a surface with a high shear strain like the machined titanium. However, the slope of the curve is also negative indicating the compressive nature of the residual stresses induced by the machining. The values for the residual stresses are summarized in Table 4. As expected, the compressive stresses induced by shot blasting on c.p. Ti are statistically significant ($p<0.001$, t-Student) and highly different from those induced on as-machined samples; but there are not statistically significant differences between equiaxed and acicular phase, both as-machined or shot blasted. All these results approve the values obtained in hardness tests because the higher the hardness, the higher the compressive residual stresses. According to this, a compressive layer with a depth showed by the hardness profiles (Figure 3) might be expected.

The different materials have been tested in unidirectional tension in order to obtain their monotonic stress-strain curves. The relevant parameters are listed in Table 5, where each value have been obtained from the average of two different tests for each material. A significant result is that acicular microstructures reduce significantly the ductility of the alloy.

Table 6 shows the number of cycles to failure ($N_f$) and the cumulative plastic strain ($\varepsilon_{\text{cum}}$) for the $\alpha$- and $\alpha'$- microstructures without and with shot blasting treatment. It can be observed that the as-machined microstructure of the equiaxed phase presents longer life.
fatigue than the acicular microstructure. This is because the interface of the acicular phase is a fast way for the propagation of the fatigue crack, as can be observed in Figure 6.

The fatigue behaviour of the samples submitted to shot blasting treatment is better due to the compressive effect of the residual stresses on the surface that makes difficult the crack nucleation. This fact can be observed in Figures 7 (as-machined specimen) and 8 (shot blasted specimen), where the crack grows from the surface and from 15-μm beneath the surface, respectively. As a consequence, an improvement of the fatigue behaviour of the shot blasted treatment for the two microstructures studied (α-equiaxed and α'-acicular) is obtained. The crack propagation is very similar for the four specimens and can be observed in Figure 9, where the crack propagation in the fracture surface with the grooves of each cycle can be seen.

The four types of specimens are cyclically deformed at different total strain amplitudes, and cyclic softening is observed in all cases. The maximum stress reached at every total strain amplitude is higher for the equiaxed than for the acicular microstructures. The highest rate of softening has been observed for the acicular phase.

Consequently, shot blasting treatment increases the implant surface roughness by impingement, at high pressure, of small abrasive particles, which results in local plastic strain. This fact will produce a firmer and earlier fixation and a better osseointegration of a dental implant as explained in the Introduction. Besides, this impingement produces an increase of the surface hardness due to the compressive loads of the impact of the particles. Despite the variables of the shot blasting treatment are not exhaustively controlled, the value of the residual compressive stresses on the surface layer affected by the treatment provokes that the crack nucleation site changes from the specimen surface (for the as-machined metal) to the specimen interior (for the shot-blasted metal). This change is postulated to result in a significant modification of fatigue properties of dental implants made of c.p. Ti.

**Conclusion**

Shot blasting of c.p. Ti dental implants not only improves the osseointegration of the implants because of the increase in the metal surface roughness but also improve their fatigue life because of the layer of compressive residual stresses that is formed.

**Acknowledgements**

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References
Table 1 Fatigue strength of some metals used for implants, in MPa

<table>
<thead>
<tr>
<th>Material</th>
<th>In air</th>
<th>In saline solution</th>
</tr>
</thead>
<tbody>
<tr>
<td>316L Stainless steel (annealed)</td>
<td>260-280</td>
<td>230-270</td>
</tr>
<tr>
<td>Cast CoCrMo alloy</td>
<td>310</td>
<td>240-280</td>
</tr>
<tr>
<td>Wrought CoCrMo alloy</td>
<td>550</td>
<td>475</td>
</tr>
<tr>
<td>Wrought commercial pure Ti</td>
<td>300</td>
<td>240</td>
</tr>
<tr>
<td>Ti-6Al-4V</td>
<td>605</td>
<td>500</td>
</tr>
</tbody>
</table>

Table 2 Chemical composition of the c.p. Ti used in this work

<table>
<thead>
<tr>
<th>Element</th>
<th>Weight %</th>
</tr>
</thead>
<tbody>
<tr>
<td>Nitrogen</td>
<td>max. 0.05</td>
</tr>
<tr>
<td>Carbon</td>
<td>max. 0.10</td>
</tr>
<tr>
<td>Hydrogen</td>
<td>max 0.0125</td>
</tr>
<tr>
<td>Iron</td>
<td>max. 0.30</td>
</tr>
<tr>
<td>Oxygen</td>
<td>max. 0.35</td>
</tr>
<tr>
<td>Titanium</td>
<td>max. balance</td>
</tr>
</tbody>
</table>

Table 3 Chemical composition of the artificial saliva

<table>
<thead>
<tr>
<th>Compound</th>
<th>Composition (g/dm³)</th>
</tr>
</thead>
<tbody>
<tr>
<td>K₂HPO₄</td>
<td>0.20</td>
</tr>
<tr>
<td>KCl</td>
<td>1.20</td>
</tr>
<tr>
<td>KSCN</td>
<td>0.33</td>
</tr>
<tr>
<td>Na₂HPO₄</td>
<td>0.26</td>
</tr>
<tr>
<td>NaCl</td>
<td>0.70</td>
</tr>
<tr>
<td>NaHCO₃</td>
<td>1.50</td>
</tr>
<tr>
<td>Urea</td>
<td>1.50</td>
</tr>
<tr>
<td>Lactic acid</td>
<td>until pH=6.7</td>
</tr>
</tbody>
</table>

Table 4 Surface residual stresses calculated at the four different types of c.p. Ti studied.

<table>
<thead>
<tr>
<th>Material</th>
<th>σ (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>α-phase as-machined</td>
<td>-77.2 (5)</td>
</tr>
<tr>
<td>α'-martensite as-machined</td>
<td>-69.3 (4)</td>
</tr>
<tr>
<td>α-phase shot blasted</td>
<td>-220.0 (3)</td>
</tr>
<tr>
<td>α'-martensite shot blasted</td>
<td>-205.1 (8)</td>
</tr>
</tbody>
</table>

Table 5 Mechanical properties obtained from the tensile tests on the different materials studied

<table>
<thead>
<tr>
<th>Material</th>
<th>Maximum strength (MPa)</th>
<th>Yield Stress 0.2% (MPa)</th>
<th>Ductility (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>α-phase as-machined</td>
<td>460 (30)</td>
<td>155 (23)</td>
<td>46 (7)</td>
</tr>
<tr>
<td>α'-martensite as-machined</td>
<td>570 (40)</td>
<td>175 (12)</td>
<td>26 (5)</td>
</tr>
<tr>
<td>α-phase shot blasted</td>
<td>480 (39)</td>
<td>168 (25)</td>
<td>39 (4)</td>
</tr>
<tr>
<td>α'-martensite shot blasted</td>
<td>587 (34)</td>
<td>189 (23)</td>
<td>19 (3)</td>
</tr>
</tbody>
</table>

Table 6 Number of cycles to fatigue-failure (Nf) and cumulative plastic strain (εcum) for the different c.p. Ti studied

<table>
<thead>
<tr>
<th>Material</th>
<th>Nf</th>
<th>εcum</th>
</tr>
</thead>
<tbody>
<tr>
<td>Titanium alpha</td>
<td>35115 (1200)</td>
<td>3.7 (0.8)</td>
</tr>
<tr>
<td>Titanium acicular</td>
<td>24447 (1348)</td>
<td>2.6 (0.3)</td>
</tr>
<tr>
<td>Titanium alpha with blasting</td>
<td>51578 (2890)</td>
<td>2.9 (0.5)</td>
</tr>
<tr>
<td>Titanium acicular with blasting</td>
<td>37600 (1001)</td>
<td>1.9 (0.4)</td>
</tr>
</tbody>
</table>
Figure 1 Microstructure of the equiaxed α-c.p. Ti.

Figure 2 Microstructure of the acicular α’-martensite

Figure 3 Hardness distribution at the above cross-section of a sample of the different types of materials studied
Figure 4. Example of $d_\Psi$ vs $\text{sen}^2\Psi$ graph for a shot blasted $\alpha$-phase c.p. Ti specimen

Figure 5. Example of $d_\Psi$ vs $\text{sen}^2\Psi$ graph for an as-machined $\alpha$-phase c.p. Ti specimen
Figure 6 Crack propagation in the acicular c.p. Ti microstructure

Figure 7 Crack nucleation on the surface of the specimen as-machined

Figure 8 Crack nucleation beneath the surface of the shot-blasted specimen
Figure 9 Fatigue crack propagation representative for all the materials studied.
Captions
Table 1. Fatigue strength of some metals used for implants, in MPa.
Table 2. Chemical composition of the c.p. Ti used in this work.
Table 3. Chemical composition of the artificial saliva.
Table 4. Surface residual stresses calculated at the four different types of c.p. Ti studied.
Table 5. Mechanical properties obtained from the tensile tests on the different materials studied.
Table 6. Number of cycles to fatigue-failure (Nf) and cumulative plastic strain (\(\varepsilon_{cum}\)) for the different c.p. Ti studied.

Figure 1. Microstructure of the equiaxed \(\alpha\)-c.p. Ti.
Figure 2. Microstructure of the acicular \(\alpha'\)-martensite.
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