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## Effect of blasting on the fatigue life of Ti-6Al-7Nb and stainless steel AISI 316 LVM

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### Abstract

Among metallic biomaterials, titanium, its alloys and stainless steel AISI 316 LVM are of high importance and are widely used as surgical implants. Since these implants are designed to remain inside the body for a long time and are subjected to fluctuating high loads, it is important to consider fatigue properties. There are a variety of surface treatments that are used in metallic surgical implants with different aims: to improve biological response, wear properties, to modify surface finish, etc. In this work, the effect of blasting, that is one of the most commonly used surface treatments, on the fatigue life of Ti-6Al-7Nb alloy and stainless steel AISI 316 LVM was studied. Topography, roughness, surface defects, and microstructural changes were examined. Rotating bending (R=-1) fatigue tests were carried out in air, at room temperature, with a frequency of 43 Hz.  $\sigma_a - N_f$  curves were obtained for each material and surface condition. The fracture surface of tested samples were also characterized.

Even though blasting introduced stress raisers, it improved the fatigue behavior of both Ti-6Al-7Nb and AISI 316 LVM samples.

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## 1. Introduction

Metallic biomaterials are extensively used as biomaterials for surgical implants on a wide range of applications such as joint and dental implants, spinal fixations, and implants for osteosynthesis (plates, screws, nails, etc). The excellent biocompatibility, good mechanical properties and high corrosion resistance make titanium, titanium alloys, and stainless steel AISI 316 LVM (Hanawa, 1999; Niinomi, 2008) attractive among biomaterials. Furthermore, titanium and its alloys facilitate osseointegration that means a direct functional and structural connection between the living bone and the implant surface (Niinomi, 2007). In recent years, Ti-6Al-7Nb has been preferred to Ti-6Al-4V in order to avoid possible allergic reactions in patients due the presence of vanadium in the latter alloy.

### Nomenclature

|      |  |
|------|--|
| Ti   | Ti-6Al-7Nb alloy   |
| SS   | Stainless Steel AISI 316 LVM                               |
| Ti-M | Ti-6Al-7Nb alloy as machined surface condition             |
| Ti-B | Ti-6Al-7Nb alloy as blasting surface condition             |
| SS-M | Stainless Steel AISI 316 LVM as machined surface condition |
| SS-B | Stainless Steel AISI 316 LVM blasting surface condition    |

Within the broad range of surface treatments used in surgical and dental implants, blasting, anodizing, acid etching, ceramic coating, and porous coating are some of the most popular ones (Fathi and Doostmohammadi, 2009; Le Guéhenec et al., 2007). Blasting, in particular, is widely used due to its practical and versatile application in implants for surgery. It is used for cosmetic and cleaning purposes, to homogenize appearance, and to remove shines and marks from prior machining and forming. In case of titanium and its alloys, it can also be used to improve osseointegration by increasing the surface roughness. For cosmetic and cleaning purposes, the use of glass particles for blasting is the most common in Argentina, due to its low cost. However, to improve osseointegration, generally alumina particles are used, as they are effective to increase roughness and improve performance (Aparicio, 2004).

Surgical implants are subjected to cyclic loads during their lifetime, and therefore, the fatigue endurance of the material plays a very important role when trying to estimate long-term performance of the device (Gil et al., 2006). Furthermore, the characteristics of the surface of an implant can affect the fatigue properties. The relevant factors are:

- The surface roughness or the presence of stress raisers at the surface,
- The presence of residual stresses at the surface ,
- The fatigue strength of the surface material.

The presence of a stress raiser, such as a notch, may accelerate the initial stage of crack nucleation (Stage I), due to increased local stress at the root of the notch. On the contrary, the introduction of superficial compressive residual stresses can significantly increase fatigue strength. Furthermore, superficial strain hardening can enhance the fatigue strength of the surface. The blasting of the surface can affect the three factors listed above, therefore, it becomes important to investigate the effect of blasting on fatigue life.

The aim of this work is to study the effect of blasting on the fatigue life of Ti-6Al-7Nb and stainless steel AISI 316 LVM.

## 2. Materials and methods

### 2.1. Materials

The materials used for the study, were Ti-6Al-7Nb and stainless steel AISI 316LVM rods of 5 mm diameter. Rotating bending fatigue samples were machined from the rods. The material characterization was carried out by metallographic analysis, static tensile test and Vickers microhardness measurements. For each material, the microstructural characterization was carried out on a transversal and a longitudinal section.

Metallographic samples were prepared. Ti-6Al-7Nb samples were etched with Kroll's reagent, and AISI 316 LVM samples were etched with glyceresia (ASTM Standard E407, 2007). A Shimadzu ® UH-1000KNA universal testing machine was used for the static tensile testing. For each material, ultimate tensile strength, yield strength and elongation were obtained. A Shimadzu HMV-2000 microhardness tester was used to obtain Vickers microhardness. The obtained results for the microhardness were an average of five measurements.

### 2.2. Blasting treatment

The blasting treatments on the Ti-6Al-7Nb fatigue specimens were performed by using irregular alumina particles, while glass microspheres were used for the AISI 316 LVM. The blasting parameters were: pressure of 5bar, treatment duration of 45s, sample velocity of 25 rpm, sample-nozzle distance of 10cm, and nozzle inclination angle of 90°. In both cases, the machining condition was used as reference. The treatment parameters and particles used are in accordance with current industrial practices.

The topography of the surfaces of the samples was characterized before and after blasting by scanning electron microscopy (SEM) using a Philips® SEM 505. Furthermore, longitudinal sections of the surface treated specimens were also analyzed in order to characterize surface damage. Additionally, energy dispersive spectroscopy (EDS) was used to confirm the presence of blasting particles embedded in the surface. Surface roughness was characterized by measuring the Ra, Rt, and Sm parameters using a TSK® Surfcom 1400 3D roughness tester.

### 2.3. Fatigue test

Rotating bending fatigue tests were carried out ( $R = -1$ ) using a M.O.P Type CT 8/30 fatigue machine. The test configuration corresponds to a cantilever beam, loaded in an extreme. The tests were carried out in air, at room temperature, with a frequency of 43Hz. (2580 rpm). A criterion of infinite life of 107 cycles was adopted. The samples were tested with constant stress amplitude at different load levels. The maximum stress applied to the samples was 70% of the ultimate tensile strength of each material. The geometry and dimension of the sample are shown in Fig 1.

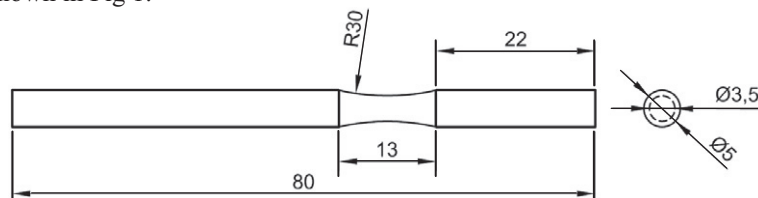


Fig. 1. Dimensions and geometry of the specimen used for the fatigue tests (in millimeters)

For each surface condition, 12 to 15 specimens were tested.  $\sigma_a - N_f$  curves were plotted based on the results of these tests. Fracture surfaces were characterized by stereomicroscopy and scanning electron

microscopy. Influence of surface damage on crack nucleation was evaluated on longitudinal sections of the tested specimens.

### 3. 3. Results and discussion

#### 3.1. Material characterization

The microstructure analysis revealed that both Ti and SS show a preferred grain orientation in the rolling direction. A higher level of plastic deformation was observed on SS than on Ti specimens. The microstructure of the SS specimens, showed austenitic grains ( $\gamma$ -phase) without neither delta ferrite ( $\delta$ -phase) nor martensite ( $\alpha'$ -phase). The microstructure of Ti revealed a dispersion of fine grained beta phase in an alpha phase matrix. The values obtained from the static tensile and the microhardness tests can be observed in Table 1. The SS showed the highest values of strength and hardness. The hardness value obtained for SS corresponds to an extra hard condition according to ASTM F138 classification. Hardness classification for Ti does not exist in the relevant standard (ASTM F1295). This standard suggests that mechanical requirements of the material for the hot rolled condition may be established by agreement between the supplier and the purchaser.

Table 1. Mechanical properties

| Material | $\sigma_{UTS}$ (MPa) | $\sigma_{0.2}$ (MPa) | E (GPa) | A(%) | MHV <sub>(1N)</sub> |
|----------|----------------------|----------------------|---------|------|---------------------|
| Ti       | 1063                 | 852                  | 101     | 10   | 347                 |
| SS       | 1436                 | 1383                 | 188     | 9,5  | 410                 |

#### 3.2. Treatment characterization

Fig. 2 shows SEM images of the surface of Ti-B and SS-B samples after the blasting treatment. The Ti-B sample shows signs of plastic deformation and tearing of the material, without signs of prior machining marks (Fig. 2a). Furthermore, EDS analysis the presence of embedded alumina particles. The SS-B samples also showed signs of plastic deformation, however to a lesser extent than in the previous case and similarly no prior machining marks were observed (Fig. 2b). The EDS analysis showed the presence of glass particle fragments. The presence of particles embedded in the surface of the blasted samples is a consequence of the high kinetic energy levels imparted to the particles during the treatment.

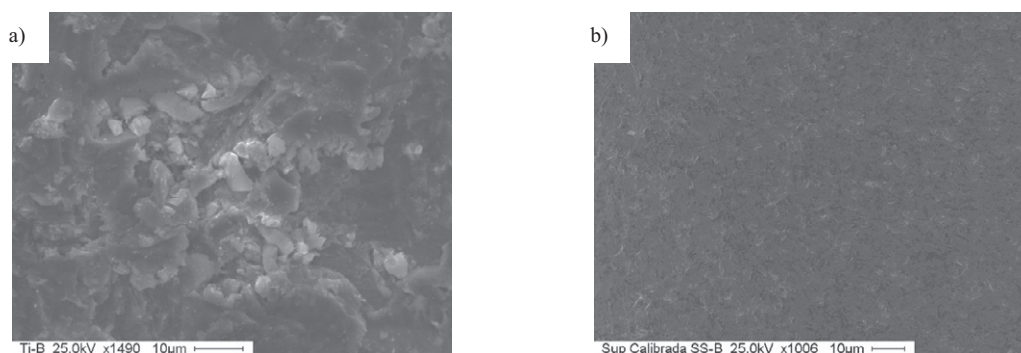


Fig. 2. Topography obtained from the blasting treatment a) Ti-B; b) SS-B

The results of the surface roughness measurements are reported in Table 2 for each condition. In both cases, blasting significantly increased the values of roughness parameters. In case of Ti-B samples, Ra increased by approximately 400%. The achieved value is within the optimal range to promote osseointegration (Cooper, 2000). For the SS-B samples, the increase of Ra parameter was in the order of 250%. The results obtained from the roughness measurements are consistent with the topography characteristics observed by SEM. The differences in the measured roughness values can result from various reasons. Firstly, glass particles with spherical geometry and low hardness tend to generate lower surface roughness (Ra) than harder and irregular alumina particles (Mohammadi et al. 2007). Moreover, the higher hardness and mechanical strength of the SS samples limit the ability of the material to suffer further plastic deformation by the particles. Both of these arguments could explain the production of a smoother surface with lower Ra and Rt values in the SS samples. As height related parameters of roughness (Ra and Rt) were reduced, horizontal parameters (Sm) increased, indicating more separation between peaks and valleys (Aparicio et al., 2004).

It is worth noticing that microstructural analysis of the longitudinal sections of treated samples revealed severely deformed subsuperficial grains, suggesting a low degree of strain hardening. Differences between the as machined and blasted conditions were not significant. Again, the high mechanical strength and low elongation of both materials limit their ability to suffer further plastic deformation during blasting.

Table 2. Measured roughness parameters

| Sample | $R_a \pm D$ ( $\mu\text{m}$ ) | $R_t \pm D$ ( $\mu\text{m}$ ) | $S_m \pm D$ ( $\mu\text{m}$ ) |
|--------|-------------------------------|-------------------------------|-------------------------------|
| Ti-M   | 0.57±0.09                     | 4.44±0.93                     | 75.59±11.00                   |
| Ti-B   | 2.25±0.14                     | 16.45±1.26                    | 125.36±23.21                  |
| SS-M   | 0.49±0.08                     | 3.50±0.35                     | 61.70±5.42                    |
| SS-B   | 1.21±0.04                     | 10.07±0.97                    | 148.72±32.19                  |

### 3.3. Fatigue testing

Fig. 3a and 3b show the  $\sigma_a - N_f$  curve for each material and surface condition. Blasting treatment improved the fatigue endurance of both materials. The increase of the fatigue strength was 14% of the ultimate tensile strength of the material for Ti-B samples, and 10% for the SS-B samples.

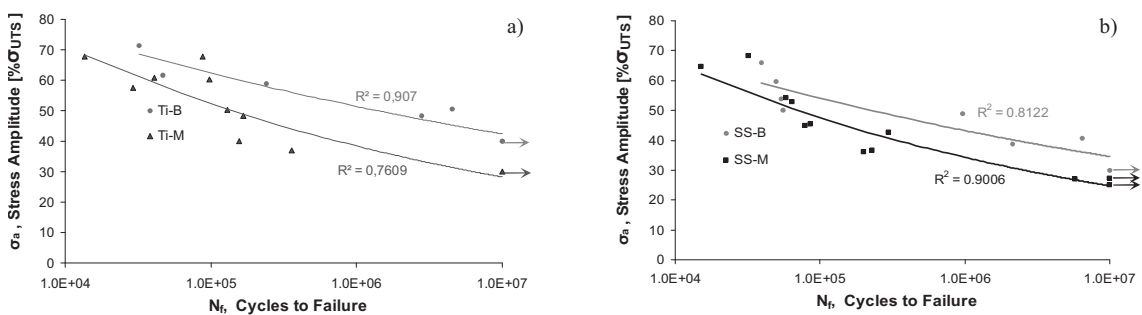


Fig. 3.  $\sigma_a - N_f$  curves. a) Ti-6Al-7Nb; b) AISI 316 LVM

The experimental data were fitted by using the Basquin equation. The coefficients obtained for each condition are shown in equations (1) to (4):

$$Ti - M : \sigma_a = 2371.8 * N_f^{-0.1341} \quad (1)$$

$$Ti - M : \sigma_a = 1645.8 * N_f^{-0.084} \quad (2)$$

$$SS - M : \sigma_a = 3158.8 * N_f^{-0.1417} \quad (3)$$

$$SS - B : \sigma_a = 2217.3 * N_f^{-0.097} \quad (4)$$

### 3.4. Surface damage and fracture surface characterization

Surface damage was characterized for all the studied conditions and it was found that blasting treatment generated irregularities that acted as effective stress raisers for fatigue crack nucleation. The longitudinal sections of tested samples showed cracks nucleated from the defects generated by the blasting treatment, for both Ti-B and SS-B samples, as it is shown in Fig. 4. These subcritical cracks were found in the vicinity of the fracture section. Unless the density of subcritical cracks decrease with the stress level, the SS samples showed a higher density than the Ti samples.

Compressive residual stresses introduced by blasting could shift the maximum local stress slightly off the surface, towards the center of the cross-section, being possible to find subsuperficial crack initiations (Pazos et al., 2010). This effect depends on the level of the introduced residual stress, the higher the level the higher the possibility of subsuperficial origins. However, in this work, crack initiation was always located at the surface. The strain hardening produced by blasting could be related with the amount of plastic deformation near the treated surface (Jiang et al., 2006). In this work, the microstructural analysis of longitudinal section of Ti-B and SS-B samples revealed a hardly visible deformed layer, in agreement with the treatment parameters used, specially the short treatment time (Verpoort and Gerdes, 1989).

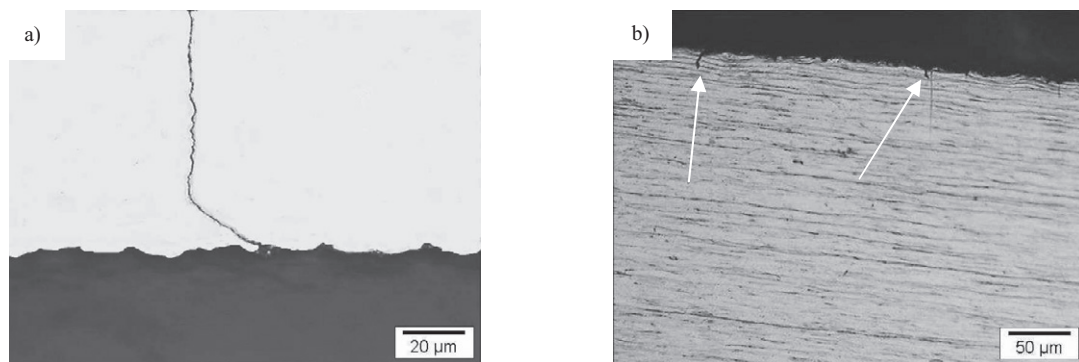


Fig. 4. Crack nucleated from surface defect. a) Ti-B 60% $\sigma_{UTS}$ , b) SS-B 60% $\sigma_{UTS}$  (the arrows show subcritical cracks)

Fracture surfaces of Ti and SS samples showed different topographic characteristics, Fig. 5. For each material, the stress level did not show effects on these topographic characteristics. As it was expected, surface treatment did not have an influence on the crack propagation mechanisms. Both Ti-M and Ti-B samples had their final fracture zone located opposite to the crack origin and were rotated counterclockwise with respect to it, these results were consistent with those reported for samples tested in rotating bending at low nominal

stress (ASM Metal Handbook, 1996). On the other hand, both in specimens SS-M and SS-B, the final fracture zone was located towards the center, furthermore multiple crack initiations were observed. These topographic features are generally found in specimens tested in rotating-bending at high nominal stress (ASM Metal Handbook, 1996). However, in the current experiments, multiple crack initiations have been exhibited also in samples tested at low stress levels. It could be elaborated that the stress raisers generated by blasting increase the local stress level and are responsible for the multiple crack initiations, however this behaviour has been observed on untreated samples as well. To a first approximation, the relative notch sensitivity of a given material may be estimated from the yield- to tensile-strength ratio (Hertzberg, 1996). In this work, SS evidenced a  $\sigma_{0.2}/\sigma_{UTS}$  ratio of 0.96 while Ti a ratio of 0.80. Materials with limited deformation capacity, would notch weaken (Hertzberg, 1996). Hence, the multiple crack initiations observed could be linked to the mechanical properties of the "extra hard" AISI 316 LVM condition.

Fig. 5b and 5d show details of the topographies of the stable and unstable propagation zones of Ti and SS samples. It highlights the presence of striations and secondary cracks in the stable propagation zone and the presence of typical dimples of a ductile fracture in the unstable propagation zone.

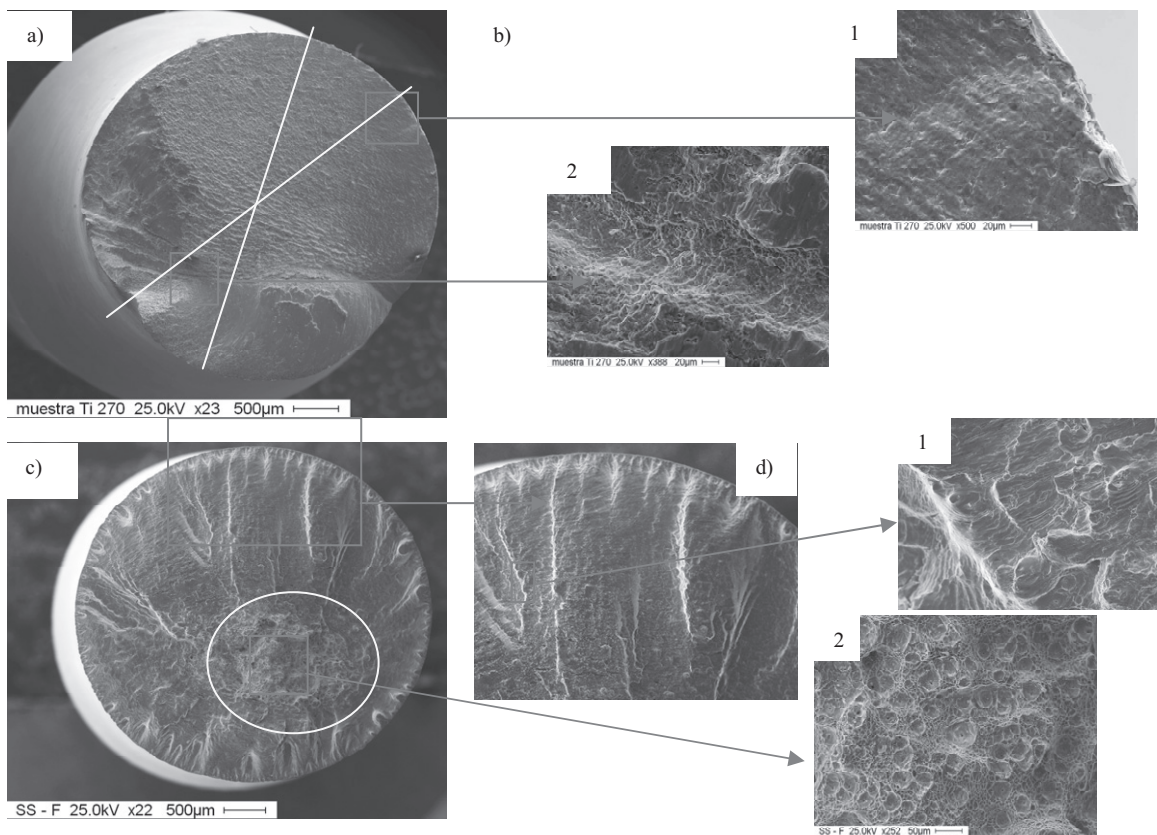


Fig. 5. Fracture surface a) Ti 70% $\sigma_{UTS}$ ; c) SS 70% $\sigma_{UTS}$ ; b) and d) Topographic characteristics; 1) initiations and stable propagation zone, and 2) unstable propagation zone

Fatigue life of surface treated materials is inherently influenced by the duration of the crack initiation stage, however, the surfaces treatment does not affected the propagation stage. Depending on the introduced modifications, surface treatments can accelerate or delay the nucleation of fatigue cracks. In this context, a

delayed crack initiation implies a higher number of cycles to failure for the same stress level. Increased surface roughness or surface damage normally result in inferior fatigue life. On the other hand, compressive residual stresses and strain hardening on the surface tend to improve fatigue endurance. In this work, blasting treatments increased surface roughness and also generated surface defects that acted as stress raisers and were preferential sites for crack nucleation. Nevertheless, blasted samples showed better fatigue behavior than untreated samples. Therefore, the introduction of residual stresses and local strain hardening associated with blasting would have outweighed the adverse effect associated with an increased roughness and the presence of stress raisers. As a result, crack nucleation was delayed on blasted samples, and an overall superior fatigue life was achieved.

#### 4. 4. Conclusions

The used blasting treatments improved the fatigue behavior of both Ti-6Al-7Nb and stainless steel AISI 316LVM. The improvement in fatigue strength of Ti samples was approximately 14% of the tensile strength of the material, while for SS samples, the improvement was 10%. Although the surface defects introduced by the treatment acted as stress concentrators and served as crack nucleation sites, the introduction of residual stresses and strain hardening associated with this type of treatment outweighed their detrimental effect.

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