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Artigo Original de Pesquisa

Influence of heat treatment and mechanical cycling on hardness and fracture analysis of Ti35Nb5Zr casting alloy

Ana Lúcia Roselino Ribeiro[1,2], Glícea Maria Silva Bizinoto[1], Marcela Fernandes de Lima[1], Luís Geraldo Vaz[1*]

[1]Faculdade de Odontologia de Araraquara, UNESP Univ Estadual Paulista, Araraquara, SP, Brasil
[2]FACIT – Faculdade de Ciências do Tocantins, Araguaína, TO, Brasil

Abstract

Titanium alloys are used in Dentistry due to their excellent mechanical, physical and chemical properties. However, it is necessary to find a biocompatible alloy to substitute the Ti-6Al-4V alloy. The presented suggestion was to study mechanical properties of a new titanium alloy without toxic elements. The Ti-35Nb-5Zr (%wt) alloy was produced by arc melting method. The tests were Vickers hardness, tensile strength and mechanical cycling, according to technical standards for titanium biomaterials. Hardness tests were performed in three different stages: before and after heat treatment at 1000°C for 1h, and after mechanical cycling test. The fracture analysis was evaluated by scanning electron microscopy. The mean values of Vickers hardness were 230.3±27.06HV before and 462.6±59.44HV after heat treatment (P<0.0001). The mean hardness values were different among the samples after mechanical cycling test. The comparison of hardness mean values after heat treatment and after dynamic test shows a decrease of the results. The maximum tensile strength mean value, obtained from tensile test, was 3,440N. It was observed, microscopically, the presence of dimples after mechanical cycling test. It was concluded that the hardness values were different at each step and the predominant fracture was the ductile type.

Keywords: Alloys; Hardness tests; Tensile strength; Titanium.

INTRODUCTION

In the Medical and Dentistry areas, the use of synthetic materials to substitute or to increase the biological tissues was always a great concern. For this purpose, several devices are made from metals, ceramics, polymers and composites. Indeed, they are not new materials at all times, but materials which new properties can be achieved by different chemical compositions or manufacturing processes.

Titanium and its alloys are very used as biomaterials due to a good combination of mechanical, chemical and physical properties such as low density, high mechanical resistance, corrosion resistance and biocompatibility[1-3].

Besides, the elastic modulus of the implants developed to substitute or interact with bone is a very important factor to be considered. This modulus should be similar to the bone modulus for promoting bone healing and remodeling[4,5]. The commercially pure titanium has elastic modulus around 104 GPa[6] and the cortical bone, between 10 and 30 GPa[4,6].

To improve the titanium elastic modulus it is suggested to add metallic elements to titanium (Ti), as the addition of niobium (Nb) and zirconium (Zr), since the first one acts as β-stabilizer, which improves the material mechanical characteristics, and the second one is used as ω phase suppressor and it has chemical properties similar to Ti.

It is known that Ti alloys combined with Nb and/

or Zr have good mechanical properties[7-13], corrosion resistance[14-16] and biocompatibility[2,10,11,15,17-21]. In addition, the elements Nb and Zr don't cause adverse reactions with the tissues around the implant[22].

In the literature there are studies related to Ti-Nb-Zr alloys[8,11,12]. However, it's required more research to find an ideal material, with proper mass ratio and melting process, regarding better mechanical, chemical and physical properties.

In the present study the Ti-35Nb-5Zr (wt%) alloy was assessed by Vickers hardness in different stages: before and after the heat treatment (1h - 1000°C) and after mechanical cycling test, and by fracture analysis.

MATERIAL AND METHOD

In this work the Ti-35Nb-5Zr alloy (wt%) was arc melted and machined as discs (6mm x 5mm) and as samples (according to ASTM E606[23] and ASTM E8 M[24]) as cited before by Ribeiro et al.[25] (2009). After that, the samples and discs were heat treated at 1000°C for 1h.

Hardness tests were achieved in three different stages. Four discs (1TT-4TT) were analyzed before and after the heat treatment at 1000°C for 1h and eight samples (A-H) were analyzed after the mechanical cycling test.

Vickers hardness analysis was carried out in eight regions of each disc and in four regions of each part of the fractured sample, with load of 500gf applied for 15s (Micromet 2100, Buehler, Lake Bluff, Illinois, USA).

Tensile test was carried out in a servo-hydraulic machine (Material Test System - MTS 810; MTS System Corporation, Minneapolis, USA), equipped with Test Star II with 10kN load cell at a constant speed of 1mm/min, to determine the tensile strength in five samples. Mechanical cycling test was carried out in the same MTS machine set to a frequency of 5Hz with 10,000 cycles and load with a value of 60% of the ultimate tensile strength mean, in eight samples. At this moment, the samples that eventually had not been fractured during the dynamic test were fractured like in the tensile test to be used in the hardness test.

The fracture surfaces were examined using scanning electron microscopy – SEM (Model T-330 A JEOL-JSM, Tokyo, Japan) and fractography analysis was performed according to literature[26].

The results of hardness were analyzed with t-student test (P≤0.05) and Tukey test (P<0.05).

RESULTS

The mean values of Vickers hardness were 230.3±27.06HV before and 462.6±59.44HV after the heat treatment. These results were statistically different when analyzed by t-student test (P<0.0001). Figure 1 shows the hardness values after the mechanical cycling test (A-H). Figure 2 shows the difference between hardness values after heat treatment (1TT-4TT) and after mechanical cycling test (A-H).

The maximum tensile strength mean value, obtained from tensile test, was 3,440N and 60% of this value (2,064N) was used for the accomplishment of the mechanical cycling test. After the mechanical cycling test, one sample (F) did not support the 10,000 cycles at a frequency of 5Hz. Figure 3 shows the fractured surface of the sample (A) that supported the dynamic test, and it was later fractured by tensile test, and Figure 4 shows the sample (F) that has been fractured during the dynamic test.

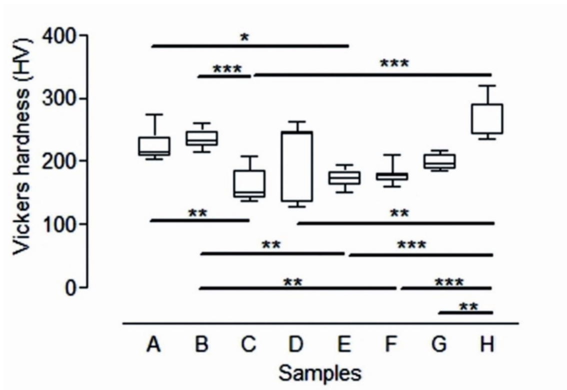

Figure 1. Distribution of Vickers hardness values (HV) for 8 distinct samples (A-H) after the mechanical cycling test. The variance analysis result was significant (P<0.0001). Comparing 2 x 2 by Tuckey test, the statistical results can be observed in the figure (* = P<0.05; ** = P<0.01, *** = P<0.001).

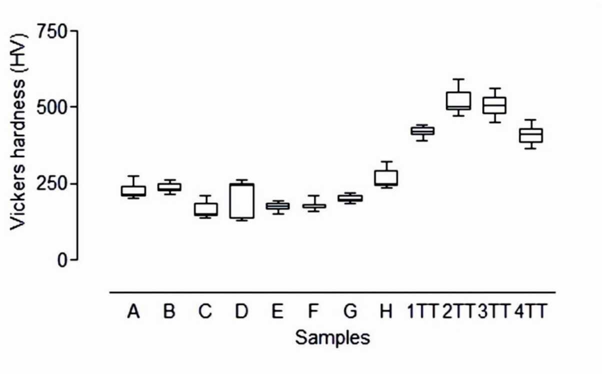

Figure 2. Distribution of Vickers hardness values (HV) for 12 distinct samples after mechanical cycling test (A-H) and heat treatment (1TT-4TT). The variance analysis result was significant (P<0.0001). Comparing 2 x 2 by Tuckey test, the results were statistically different (P<0.001) for all samples.

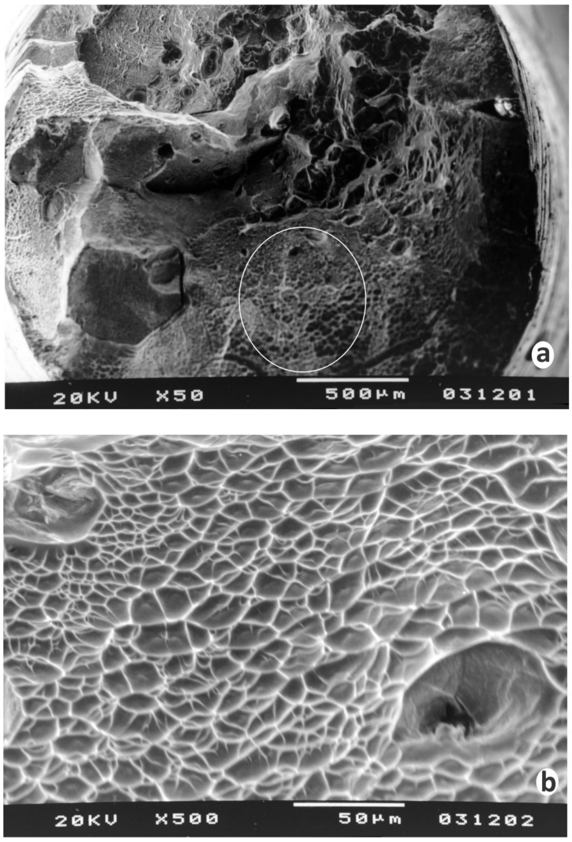

Figure 3. SEM images of sample A (a) 50x and (b) 500x regarding the circle, showing the dimples presence, after mechanical cycling test and tensile test.

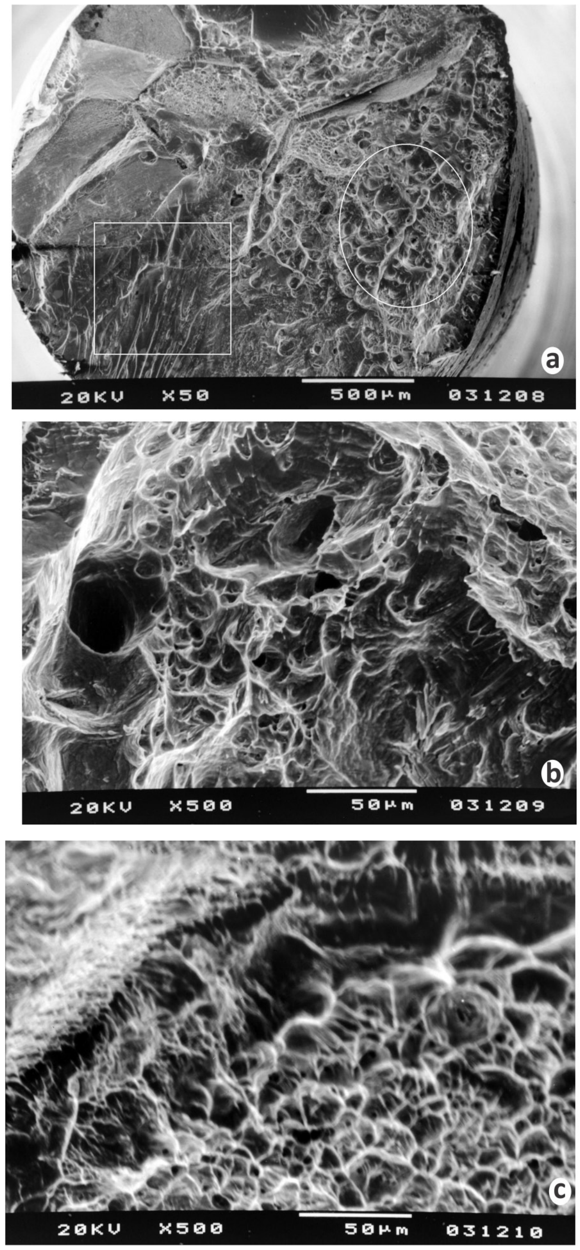

Figure 4. SEM images of sample F (a) 50x and (b) 500x regarding the circle, showing the dimples presence and (c) 500x regarding the square, showing the cleavage presence, after mechanical cycling test.

DISCUSSION

The heat treatment at 1000°C for 1h led to an increase of the hardness mean values of the Ti-35Nb-5Zr discs. This fact shows that the structure of the discs changed and, probably, it means a phase modification of the alloy.

According to those findings, some studies with Ti and Ti alloys presented similar results when the materials were heat treated as Ribeiro et al.[27] (2005), Kobayashi et al.[9] (1998) and Rocha et al.[28] (2006) that studied commercially pure titanium (cpTi), Ti-Zr-Nb alloys and, cpTi and Ti-Al-V, respectively. However, Elias et al.[7] (2006) found that the hardness value of Ti-Nb-Zr alloy decreased after heat treatment. In addition, it is important to note that those studies differ in the heat treatment type, regarding time, temperature and cooling method.

After the mechanical cycling test, the mean va-lues of hardness were statistically different among the samples. That result is, possibly, due to the machine process and to the repetitive loads that the samples supported on the dynamic test that, probably, modified the forces distribution unevenly, and then, it changed the samples structure.

The comparison of hardness mean values after heat treatment and after dynamic test shows a decrease of the results and, again, it is possibly justified by the no uniform distribution of forces after mechanical cycling test.

As result of fracture analysis, it was observed, macroscopically, on the samples that supported the mechanical cycling test a section reduction and after the forced rupture, a cup-cone fracture. Microscopically, it was observed by SEM the presence of dimples on the samples that supported the dynamic test (Figure 3). These are properties of ductile materials, that is, they have plastic deformation. It is important since that fracture is predominantly verified in pure metals and it characterizes a material that can be easily conformed.

One sample (F) has not supported the cycles set in the dynamic test and it was verified that its rupture was different from the others samples. Microscopically, it was observed, by SEM, mixed fracture with presence of dimples and cleavage (Figure 4).

Probably, the sample F failed due to limitations in the casting process. In this study, the casting of the Ti-35Nb-5Zr ingots was a manual method and it was carefully remelted until it could be observed a homogeneity ingot. However, porosity induced by casting can happen and it is a critical characteristic in the materials performance front to the mechanical cycling test[15,29].

As final observation, this study showed some important characteristics of Ti-35Nb-5Zr alloy, but it is important to improve the casting process and to assess its microstructure before and after the heat treatment to verify if there is any phase transformation.

CONCLUSIONS

•The Vickers hardness mean value had increased after heat treatment at 1000°C for 1h and it had decreased after mechanical cycling test.

•The predominant fracture was of the ductile type.

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*Autor de Correspondência:
Luís Geraldo Vaz
Departamento de Materiais Odontológicos e Prótese
Faculdade de Odontologia de Araraquara
UNESP – Universidade Estadual Paulista
Rua Humaitá, 1680
14801-903, Araraquara, SP, Brasil.
Phone: +55-16-33016420
Fax: +55-16-33016406
e-mail: lugervaz@foar.unesp.br

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