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Effect of thermal treatments on tensile strength of commercially cast pure titanium and Ti-6Al-4V alloys

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Heating titanium structures is assumed to relieve tensions induced by the casting process as well as possibly optimizing some mechanical properties. The aim of this investigation was to evaluate the effect of thermal treatments on tensile strength of commercially pure titanium (CP Ti) and Ti-6Al-4V alloy. Thirty dumbbell rods, with diameters of 3.0 mm at the central segment and lengths of 42 mm, were cast for each metal using the Rematitan System. CP Ti and Ti-6Al-4V specimens were randomly divided into three groups of ten: a control group that received no thermal treatment and two test groups. One (T1) was heated at 750 °C for 2 h and the other (T2) was annealed at 955 °C for 1 h and aged at 620 °C for 2 h. Tensile strength was measured with a universal testing machine (MTS model 810). Tensile strength means and standard deviations were statistically compared using a Kruskal-Wallis test at a $\alpha = 0.05$ significance level. No statistically significant differences in tensile strength were observed among CP Ti groups. For the Ti-6Al-4V alloy, the control and T1 groups revealed statistically higher tensile strengths when compared to the T2 group, with no significant difference between the control and T1 groups.

1. Introduction

The rising cost of gold has encouraged development of dental casting alloys. Titanium is currently the metal of choice. Commercially pure titanium (CP Ti), available in four grades (1-4 ASTM), is based on the incorporation of small amounts of oxygen, nitrogen, hydrogen, iron and carbon during purification procedures [1]. Titanium has been increasingly used in dental applications because of its excellent biocompatibility, high corrosion resistance, low density, high strength/weight ratio, high ductility, low thermal conductivity, and adequate mechanical properties [2–5]. The most commonly used titanium alloy is Ti-6Al-4V due to its desirable mechanical characteristics [1, 6, 7].

Reports in the literature suggest that when titanium structures are submitted to elevated temperatures, the tensions induced by the casting process may be relieved, reverting the material to its original structure. In addition to relieving stress, thermal treatments optimize particular properties such as fracture toughness, fatigue strength, and hardness [1].

The high melting point of titanium (approximately 1700 °C for CP Ti) and the relatively low mold temperature (approximately 430 °C) generate a considerable thermal declive during the casting process [8]. Thus, the use of a thermal treatment that relieves the tension caused by the casting process may be of great relevance for the improvement of the clinical performance of the structures of the titanium prostheses. In addition, the use of thermal treatments that improve determined properties, such as resistance to traction, may result in a greater clinical applicability and longevity of the titanium composites.

At room temperature, CP Ti has a hexagonal close-packed (hcp) crystal structure referred to as the "alpha" phase. This structure transforms to a body-centered cubic (bcc) crystal structure, called "beta" phase, at 883 °C. Whilst this phase change can directly influence properties of titanium, its influence on the efficiency of thermal treatments during casting is not known.

Use of titanium in prosthetic restorations is relatively recent, thus there are few studies available

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discussing their behavior, such as wear factors and masticator strain, within the diverse oral environment. Consequently, studies on procedural techniques such as thermal treatments are required, particularly those concerning the application of titanium and titanium alloys in dental practice. The purpose of this study was to evaluate the effect of thermal treatments on the tensile strengths of CP Ti and Ti-6Al-4V cast alloys.

2. Experimental procedures

Both metals were manufactured by the RMI Company in Ohio, USA. Compositions are given in Table I.

Thirty dumbbell rods with a 3.0 mm diameter at the central segment and a length of 42 mm [9], obtained from a stainless steel mold, were cast for each metal in a Rematitan System (Rematitan, Dentaurum J. P. Winkelstroeter KG, Pforzheim, Germany). Wax patterns were connected to a V-shaped sprue and placed in the investment material (Rematitan Plus, Dentaurum J. P. Winkelstroeter KG, Pforzheim, Germany), which was mixed and heated according to the manufacturer's instruction.

Castings were divested and air-abraded with 50 μm aluminum oxide. Specimens were then separated from the sprues using a precision saw (Isomet 1000, Buehler, Lake Bluff, Illinois, USA). After radiographing for eventual porosity or voids [6], defective specimens were eliminated, and only intact specimens were included in the test. Specimens were then machined with a fine finishing stone (Dentaurum J. P. Winkelstroeter KG, Pforzheim, Germany) in a headpiece air turbine (Dabi Atlante SA Industria Médico Odontológicas, Ribeirão Preto, Brazil). Finally the specimens presented a diameter of 3.0 ± 0.3 mm at the central segment and a length of 42 mm.

CP Ti and Ti-6Al-4V alloy specimens were randomly divided into three groups of ten which received the following thermal treatments: control—no thermal treatment; treatment 1 (T1)—heated at 750 °C for 2 h; and treatment 2 (T2) annealed at 955 °C for 1 h and aged at 620 °C for 2 h. Heating and cooling took place in a furnace (FV-1 MP, E. D. G. Equipments and Controls Ltda., São Carlos, Brazil) in an argon atmosphere. Following the thermal treatments, the dimensions of the specimens were measured with a digital paquimeter (500-144B model, Mitutoyo Sul América Ltda, Suzano, SP, Brazil), with a precision of 0.01 mm, and no dimensional alteration was observed.

Tensile strength was measured using a universal testing machine (MTS model 810, MTS System Corporation, Minnesota, USA) at a crosshead speed of 1.0 mm/min. Tensile strength means and standard deviations were statistically compared using a Kruskal-Wallis non-parametric test at a $\alpha = 0.05$ significance level.

Microstructures were analyzed using an optical microscope and the fracture surface was analyzed with a scanning electron microscope (SEM) in order to characterize the effect of the thermal treatments on tensile strength.

TABLE I Metal composition (%)

	N	C	H	Fe	O	Al	V	Ti
CP Ti Grade 2	0.02	0.08	0.007	0.18	0.15	–	–	Balanced
Ti-6Al-4V Grade 5	0.02	0.01	0.003	0.22	0.17	6.2	3.8	Balanced

3. Results

The Kruskal-Wallis test presented no statistically significant differences in tensile strength among the control specimens and the treated CP Ti specimens (Table II). Conversely, the control and T1 specimens of the Ti-6Al-4V alloy presented statistically higher tensile strengths compared to the T2 specimens of the same material. There was no significant difference among control and T1 specimens of the titanium alloy (Table III).

Figs 1 and 2 illustrate the optical microscopy analysis of the experimental groups.

For the CP Ti alloy, the control group presented an equiaxial alpha grain structure (Fig. 1(a)). Compared with the control group, treatment 1 provoked just a slight disarrangement of grains (Fig. 1(b)). In contrast, treatment 2 caused evident microstructural changes (Fig. 1(c)).

For the Ti-6Al-4V material, the control and T1 groups presented a granular structure with a mixture of equiaxial grains and inter-granular β (Fig. 2(a) and (b)), whilst the T2 group demonstrated a typical Widmanstatten structure, with White plates (α phase) and dark regions (β phase) (Fig. 2(c)).

Figs 3 and 4 present the analysis of the fracture surface with scanning electron microscopy (SEM).

The fractured surface of all specimens, regardless of treatment, were analyzed by scanning electron microscopy (SEM). The CP Ti control group presented ductile fractures with surface dimples of varying sizes. T1 specimens revealed mixed fractures with dimples

TABLE II Tensile strength means (MPa) and standard deviations for treatments of CP Ti

	Thermal treatment		
Materials	Control	T1	T2
CP Ti	537.32[a] (SD 29.19)	550.82[a] (SD 43.21)	474.85[a] (SD 109.91)

[a]Values followed by the same letter were not statistically different ($P > 0.05$).

TABLE III Tensile strength means (MPa) and standard deviations for treatments of Ti-6Al-4V

	Thermal treatment		
Materials	Control	T1	T2
Ti-6Al-4V	916.42[a] (SD 22.62)	930.29[a] (SD 24.63)	753.61[b] (SD 36.76)

[a]Values followed by the same letter were not statistically different ($P > 0.05$).

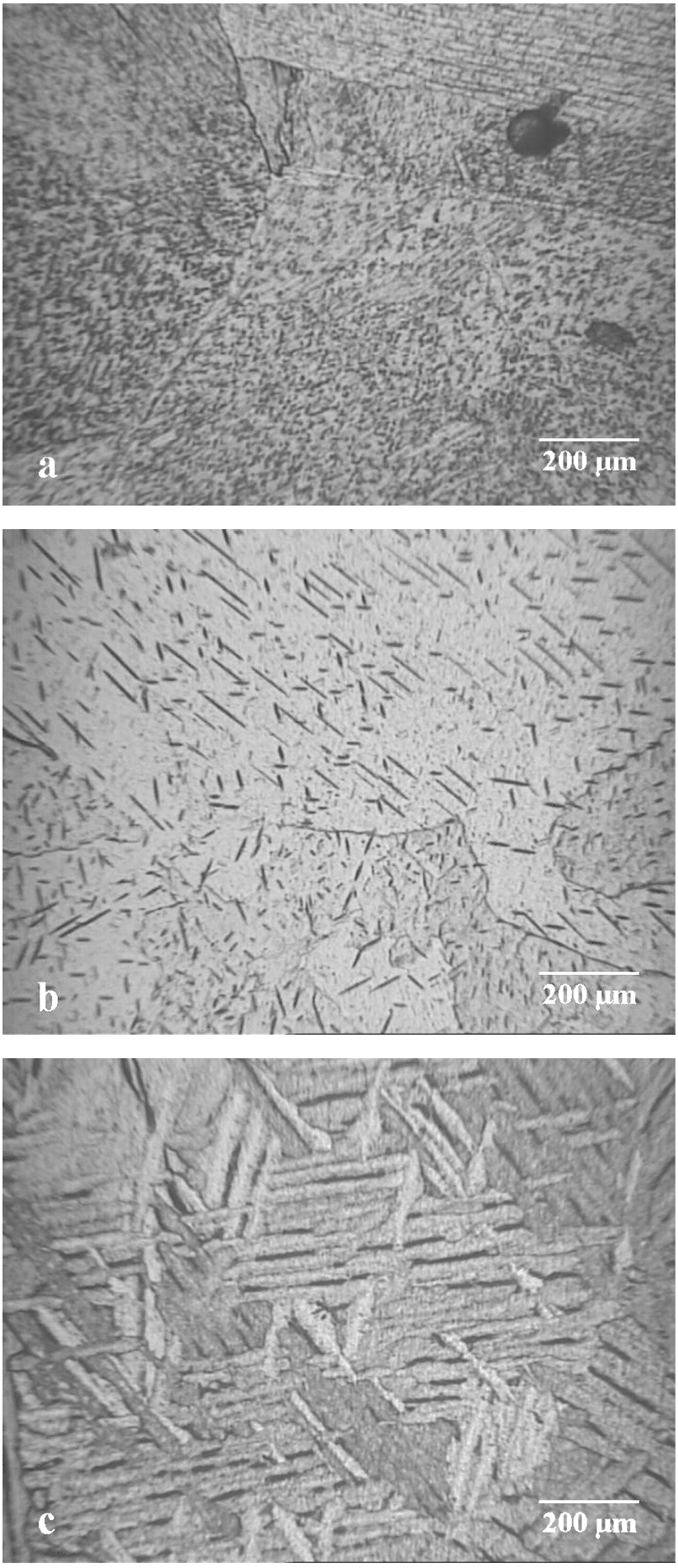

Figure 1 Optical micrographs for CP Ti: (a) control, (b) T1 and (c) T2.

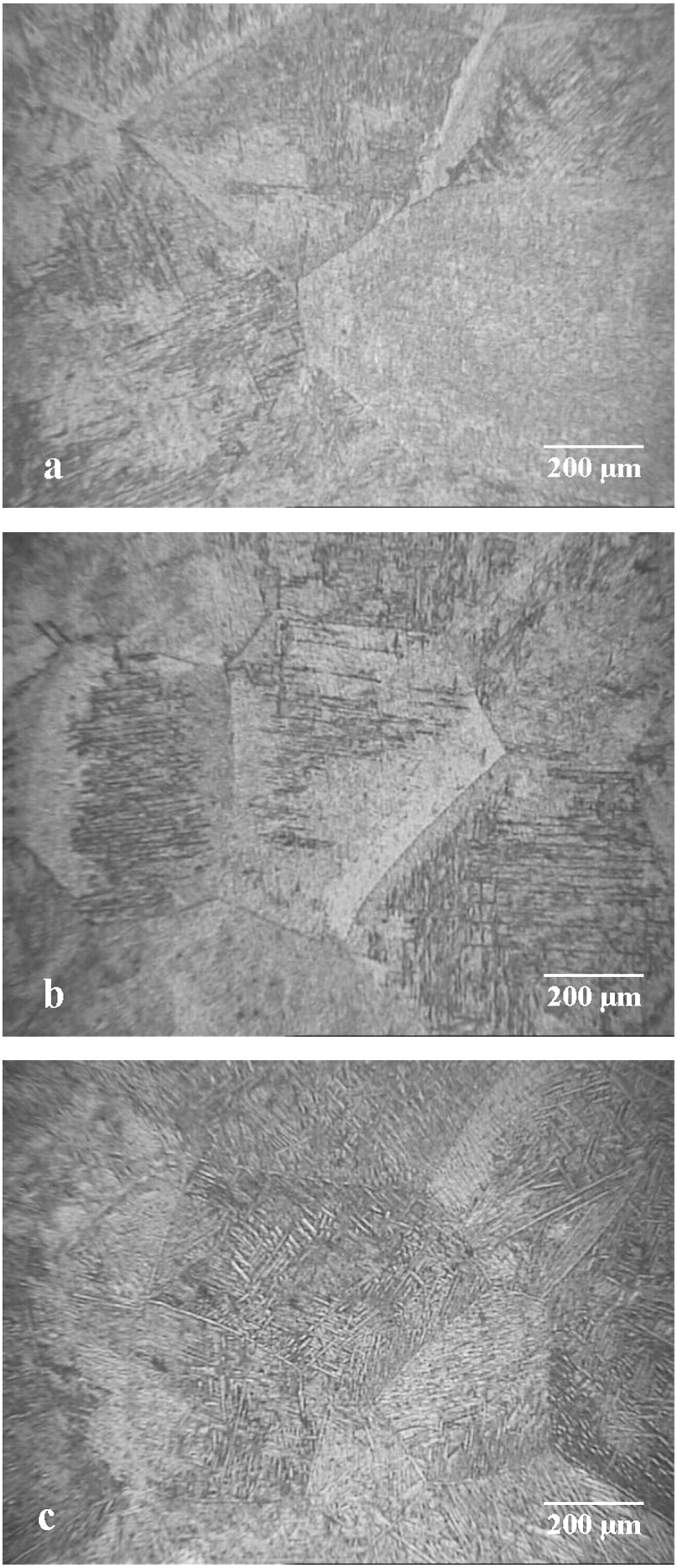

Figure 2 Optical micrographs for Ti-6Al-4V alloy: (a) control, (b) T1 and (c) T2.

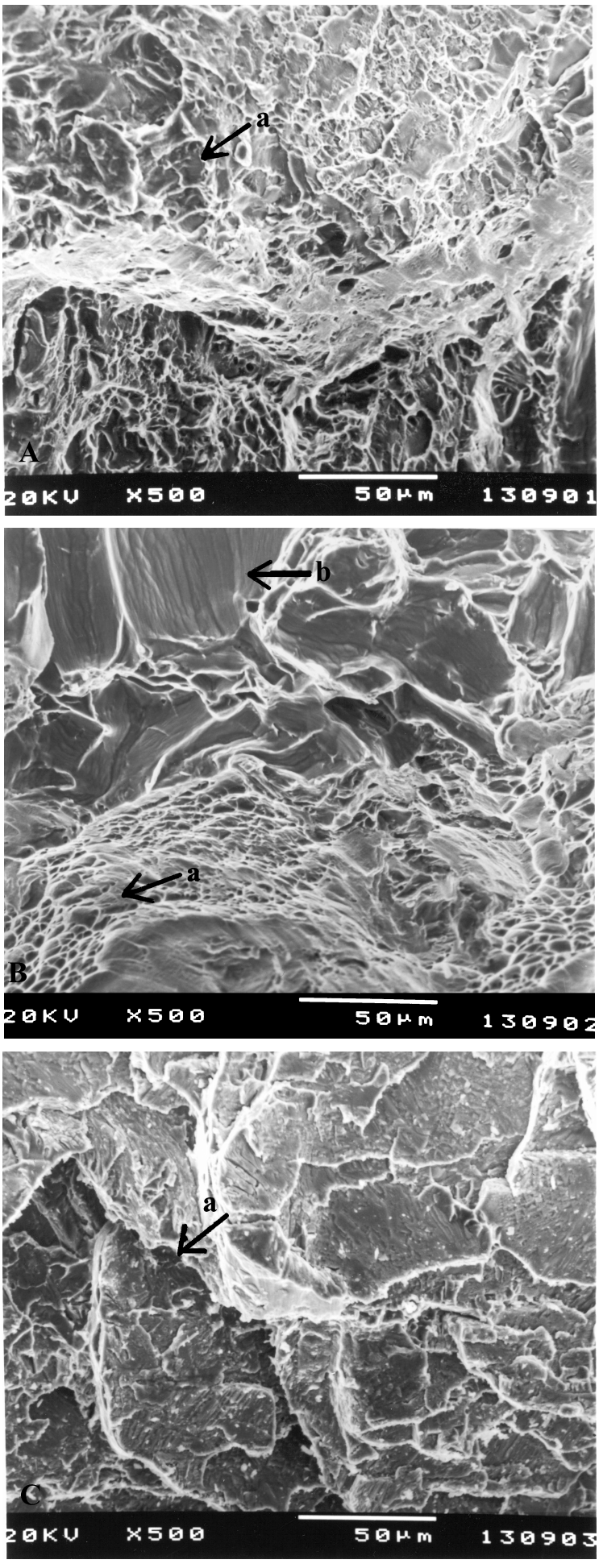

Figure 3 SEM micrographs for CP Ti: (A) control— (a) dimples, (B) T1— (a) dimples, (b) striation area, and (C) T2— (a) inter-granular fracture.

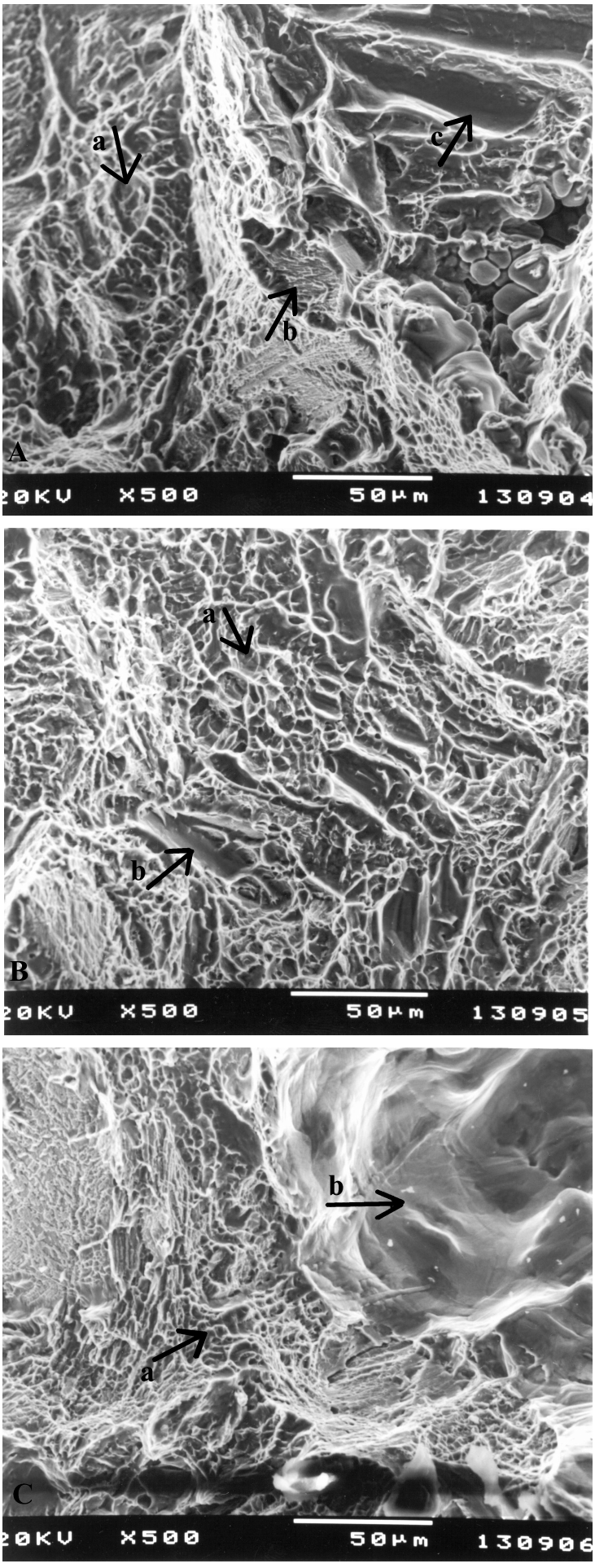

Figure 4 SEM micrographs for Ti-6Al-4V (A) control— (a) dimples, (b) micro voids and (c) striation area, (B) T1— (a) dimples and (b) striation area, and (C) T2— (a) dimples and (b) striation area.

typical of ductile fractures and striation areas that are particular to brittle fractures. T2 specimens did not present any ductility characteristics; however, there was evidence of inter-granular brittle fractures (Fig. 3).

The Ti-6Al-4V control group presented mixed fractures that were predominantly ductile with dimples, micro voids and striation areas. T1 specimens presented ductile fractures with a tendency for striation. T2 specimens were similar to CP Ti T1 specimens in that mixed fractures were found with several dimples and striation areas and a predominance of brittle fractures (Fig. 4).

4. Discussion

Several studies have attempted to characterize properties of pure titanium and titanium alloys after treatments involving various lengths of time at different temperatures as well as diverse cooling methods [10–17]. Two thermal treatments were evaluated in this study: 750 °C for 2 h (T1) and annealing at 955 °C for 1 h + aging at 620 °C for 2 h (T2). Both treatments were performed in a furnace with argon-controlled atmosphere.

Average tensile strength of control groups (no thermal treatment) was 537.32 MPa for CP Ti and 916.42 MPa for Ti-6Al-4V alloy. These values are similar to those of Donachie Jr. (550 and 907 MPa, respectively) [1].

Statistical evaluation showed no significant difference among treatments of CP Ti (Control—537.32 MPa, T1—550.82 MPa and T2—474.85 MPa). With regard to the Ti-6Al-4V alloy, the T2 group (753.61 MPa) was statistically different from the other groups (Control—916.42 MPa and T1—930.29 MPa), which were statistically similar.

Similar studies [10, 16, 18, 19] have attempted to assess the effects of different temperature and cooling conditions on the mechanical properties and microstructure of pure titanium and titanium based alloys. Greater tensile strength was associated with rapid cooling in water or oil [15]. This greater value could be explained by the β phase transforming into a stronger α phase after quenching in water above 800 °C. The grain size is related to the velocity at which the material is cooled, and, in general, rapid cooling promotes finer structure and better properties such as yield stress, ductility and crack propagation strength [1, 18].

Results of the present study indicate that several variables can influence mechanical properties of titanium alloys after thermal treatment. Factors such as alloy phase (α, $\alpha + \beta$ or β); treatment temperature in the field of $\alpha + \beta$ or β phase; temperature duration as well as cooling rate or method determine the properties obtained. Examination by optical microscopy (Fig. 1 and 2) illustrates apparent changes caused by the thermal treatment in both CP Ti and Ti-6Al-4V alloy.

Control CP Ti (Fig. 1(a)) presented an equiaxial alpha grain structure. Treatment one (T1) exposed CP Ti specimens to temperatures in the α field phase. After which the structure remained relatively globular (equiaxial) with slight disarrangement of grains (Fig. 1(b)). Considering that the T1 treatment temperature (750 °C for 2 h/furnace cooling) was below that of the phase transformation temperature (883 °C), this slight change in microstructure was justifiable. On the other hand, when the T2 treatment was exposed to annealing temperature in the β phase field, which is associated with aging, there were evident microstructural changes (Fig. 1(c)). Statistical analysis showed that changes in CP Ti structure after the different thermal treatments were not enough to significantly modify tensile strength values. According to the literature, CP Ti possesses an alpha structure, which can be stress relieved and annealed; however, strength cannot be increased through thermal treatments [1].

Concerning the Ti-6Al-4V alloy, the T2 group had significantly lower mean tensile strength (753.61 MPa) compared to control (916.42 MPa) and T1 (930.29 MPa) specimens. The latter two were statistically similar. Reports indicate greater tensile strength values after annealing and aging. However, a probable justification of the smaller value obtained after the T2 treatment is the cooling method employed. Better results are normally obtained with this temperature range when specimens are rapidly cooled by quenching in water. Thus, the β phase structure present at the annealing temperature may be maintained or partially transformed during cooling by martensite transformation or nucleation and growth [1].

In contrast, with slow cooling, appreciable diffusion may occur during cooling, and decomposition of the altered beta phase futing aging may not provide effective strengthening [1].

It is important to point out that, in the present study, the method of slow cooling was chosen due to the reduced dimensions of the specimens. According to Donachie [1], for products of small section size, air or fan cooling are preferred because it minimizes distortion.

Another explanation for our findings is offered by Markovsky [14]: the author advocated that when two $\alpha + \beta$ cast titanium alloys are submitted to a rapid thermal treatment, tensile and fatigue properties are improved due to the formation of a heterogeneous β phase at high temperatures. The homogeneous β phase is formed during the conventional treatment where the α particles are at the same position after annealing, as in the initial lamellar structure. Markovsky suggested that this occurrence could be responsible for the small deviation in the mechanical properties of the casting alloys.

The similar behavior presented by the control and T1 groups for the Ti-6Al-4V alloy in the present study can be explained by optical microscopy analysis (Fig. 2(a) and (b)), which revealed both groups to have a granular structure with a mixture of equiaxial grains and inter-granular β phase. This finding demonstrates that treatment 1, performed in the phase α field (750 °C), was not sufficient to cause the appearance of irregular grains, which provide a stronger structure than the equiaxed structure [1].

In contrast, the T2 treatment of the titanium alloy presented a typical Widmanstatten structure, showing white plates (α phase) and dark regions (β phase) in each grain (Fig. 2(c)). The inferior behavior of the T2 group may be explained by this structure, which, from the pattern of distorted platelets, is detrimental to fracture toughness, negatively influencing crack propagation [1].

In summary, diverse temperatures, cooling methods and pressure conditions as well as atmospheric control can be used for complex phase transformation in titanium-based alloys. These considerations, however, make the understanding of the results obtained complicated, limiting us to suggestions which may explain the effects caused by the different thermal treatment used. Accordingly, further studies in areas such as chemical, physical, mechanical and biological behavior are needed to improve thermal conditions of treatments before prosthetic employment.

5. Conclusions

Within the limits of this study, the following conclusions can be drawn: tensile strength of CP Ti was not influenced by thermal treatments. With the Ti-6Al-4V alloy, however, the control and T1 groups revealed statistically similar strengths, and higher values for the T2 group).

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