Microstructural Analysis of Ti-6Al-4V alloy after Plasma Immersion Ion Implantation (PIII)

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Abstract

The search for alloys with improved high-temperature specific strength and creep-resistance properties for aerospace applications has led in the last decades to sustained research activities to develop new alloys and/or improve existing ones. Titanium and its alloys are excellent for applications in structural components submitted to high temperatures owing to their high strength to weight ratio, good corrosion resistance and metallurgical stability. Its high creep resistance is of great importance in enhancing engine performance. However, the affinity by oxygen is one of main factors that limit its application as structural material at high temperatures. Materials with adequate behavior at high temperatures and aggressive environmental became a scientific requirement, technological and economically nowadays. The objective of this work is the mechanical and microstructural characterization of the Ti-6Al-4V alloy after treatment by nitrogen Plasma Immersion Ion Implantation (PIII) process. The aim of this process is the improvement of superficial mechanical properties of the Ti-6Al-4V alloy. The selected alloy after ionic implantation process by plasma immersion was submitted to creep tests at 600 ºC, in constant load mode at 250 and 319 MPa. The techniques used in this work were optical microscopy and scanning electronic microscopy. The fractograph analysis of the samples tested in creep shows narrowing phenomena and microcavities. The creep results show the significant increase of material resistance, it can be used as protection of oxidation in high temperatures applications.

1 – Introduction

Ti-6Al-4V alloy is one of the mostly used titanium alloys in aeronautical and biomedical applications because of its excellent combination of mechanical, toughness, corrosion resistance, and chemical stability properties [1-2]. However, the affinity by oxygen is one of main factors that limit its application as structural material at high temperatures. The high solid solubility of oxygen in titanium results in material loss and in the formation of hard and brittle layer during elevated temperature air exposure. [3]. The development of titanium alloys with the objective of improving the creep properties have been observed, although the surface oxidation limits the use of these alloys in temperatures up to 600°C [4]. In recent works was verified the increasing of the superficial properties of the alloy through the PIII nitrogen treatment [5-7]. Based on those results this work aims to evaluate the creep resistance of the Ti-6Al-4V alloy with superficial treatment of PIII.

2 – Experimental

The material used in this work was obtained in Multialloy Eng. Mat. Ltda, forged and annealed at 190ºC during 6 hours and cooled in air. The creep specimens of Ti-6Al-4V alloy are presented in Figure 1. Two samples were treated by PIII treatment (Figure 2) at Instituto Nacional de Pesquisas Espaciais (INPE). In this treatment the samples were put in a vacuum reactor with pressure of $76x10^{-3}$ Pa and implanted by nitrogen ions during 120 minutes. The frequency used was 400 Hz, pulse of 40 μs and voltage varying between 7 and 10 kV. All the samples of Ti-6Al-4V alloy, untreated and treated by PIII, were submitted to creep tests at 600°C and 250 at 319 MPa under constant load mode at Instituto Tecnológico de Aeronáutica (ITA/CTA). Creep tests were realized using MAYES machines. Antares Software was used to collect the data on the elongation of the samples and the measuring of temperature in pre determined periods of time. It was used a transducer-type LVDT Schlumberger D 6.50 to obtain measures of elongation and it was used Cromel-Alumel thermocouple type AWG24 to control temperature. The creep tests were realized in accord to the standard ASTM E139 [8].

Figure 1 – Creep specimens.

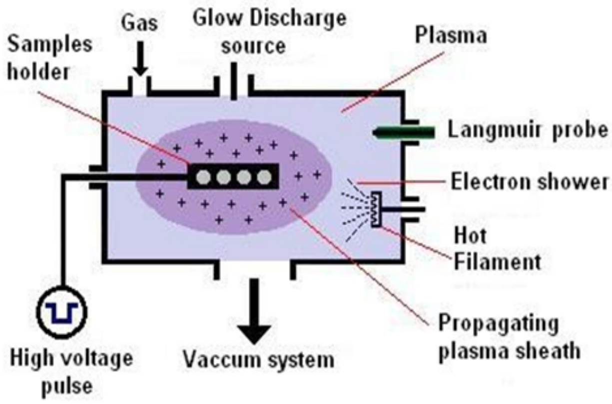

Figure 2 – Schematic diagram for PIII apparatus.

The preparation of samples for analysis by optical microscopy and scanning electron microscopy followed the usual patterns of metallographic: hot pressing (150ºC and 21 MPa), followed by hand sanding with sandpapers based on SiC, following 120, 240, 320 400, 600 and 1200 #. The polishing was done with a solution of colloidal silica (OP-S). The SEM images were obtained in the backscattering electron mode, whose main mechanism of contrast is related to differences in average atomic number between the phases present. Through the analysis by SEM were studied the main characteristics of the fracture surfaces. An optical microscope Leica model DMRXP and the scanning electron microscope model LEO 435 VPI trade mark also were used.

3 – Results and Discussions

Figure 3a shows the microstructure of the annealed Ti-6Al-4V alloy. It could be observed α grains (CH) and dark regions that define the presence of β phase (BCC) along the grain boundaries of the alloy. Ti-6Al-4V PIII treated micrograph (Figure 3b) shows similar structure than untreated alloy with thin structure and small grains. Using XRD analysis was possible to observe the Ti_2N phase (Figure 4). The PIII treatment produces a thin layer of Ti_2N with 0.6 nm of thickness.

Figure 5 shows AFM analysis. The roughness increasing in PIII treated alloy, probably due to the ions sputtering on the surface, this was verified in recent works in literature [9-10]. The sample treated by PIII presents average roughness (rms) of 1.94 nm, while the untreated sample the value is 0.09 nm.

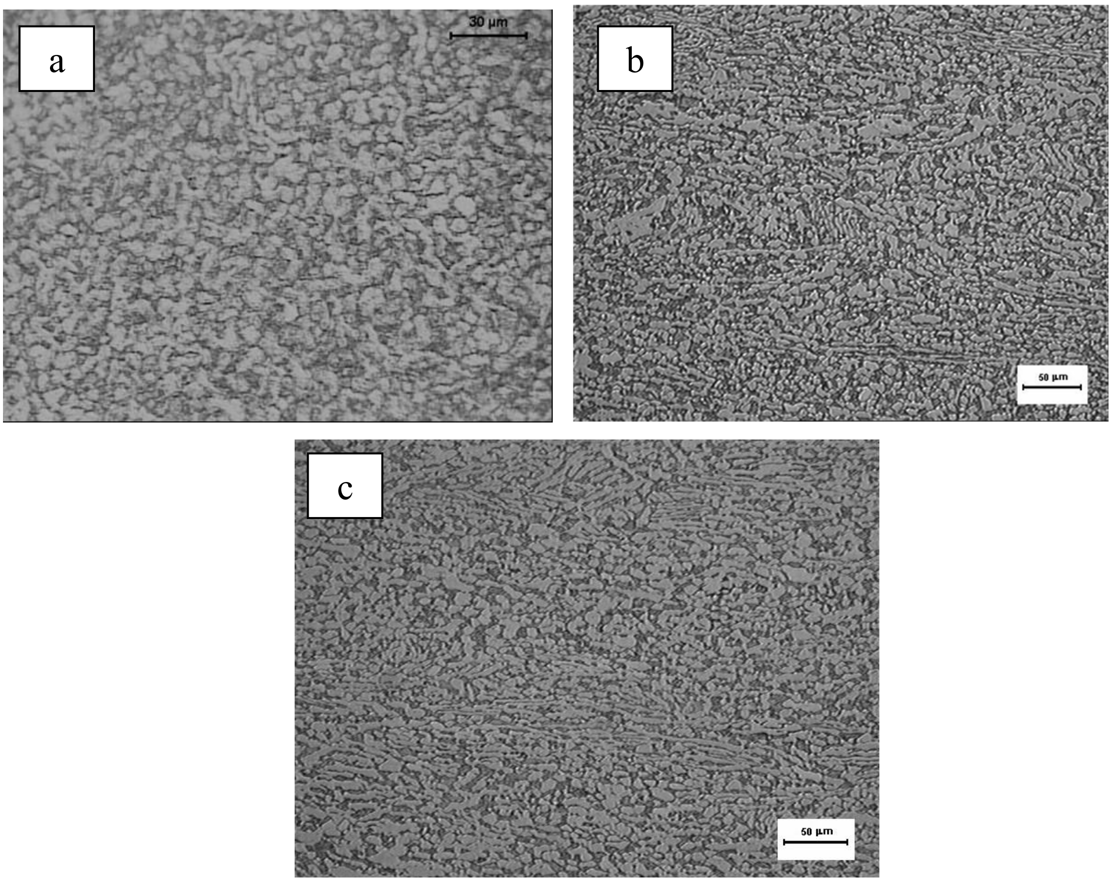

Figure 3 - Micrograph analysis of Ti-6Al-4V alloy (a) untreated; (b) treated by PIII after creep test (600ºC; 250 MPa) and (c) treated by PIII after creep test (600ºC; 319 MPa)

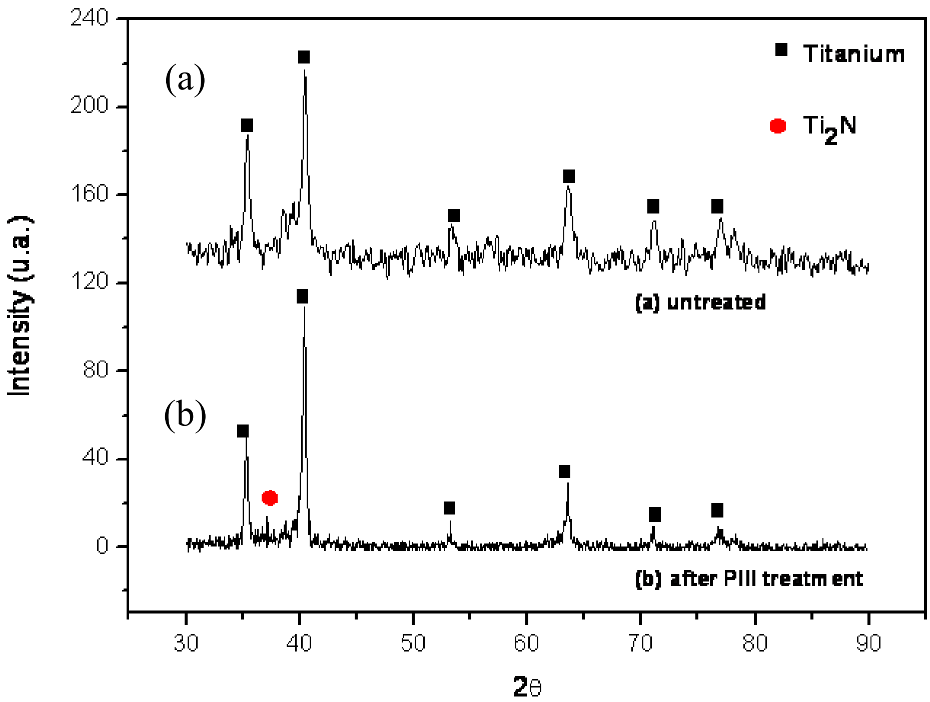

Figure 4 - XRD analysis of Ti-6Al-4V alloy (a) untreated and (b) treated by PIII.

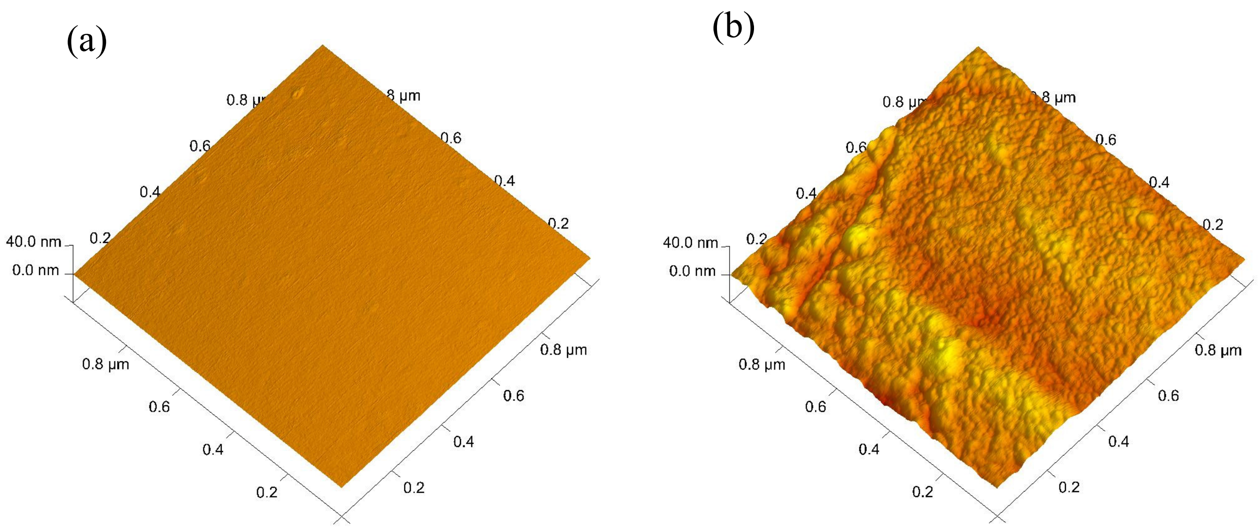

Figure 5 - AFM analysis of Ti-6A-4V alloy (a) untreated sample (b) treated sample.

Figure 6 presents the creep curves obtained by Ti-6Al-4V treated by PIII and untreated at 600°C and 250 MPa and 319 MPa. The Ti-6Al-4V alloy shows a normal curve of creep stages consisting of primary, secondary and ternary well defined. There is a relatively short initial period of decreasing primary creep rate that is associated with hardening due to the accumulation of dislocations. However, most of the creep life is dominated by a constant creep rate that is thought to be associated with a stable dislocation configuration due to recovery and hardening process.

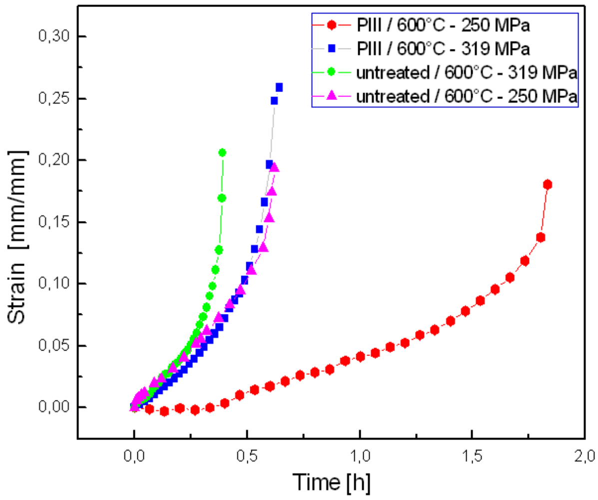

Figure 6 - Creep curves of Ti-6A-4V alloy obtained at 600 ºC, 250 and 319 MPa.

Table 1 show that the highest values of t_p and during primary creep are dependent on the test stress. This resistance is a relation of the superficial hardness obtained in this treatment. The following results present in Table 1 show the relationship of the main experimental parameters obtained at 600°C from experimental curves. When σ is the applied stress, $\dot{\varepsilon}_s$ is the stationary creep rate, obtained from the slope of the linear creep curve (secondary stage). The value of t_p is the constant relative time to primary time, obtained in the final stage of primary and / or in the beginning of secondary stage. The value t_f is the final time of fracture, ε_f correspond to the fracture strain and AR the percentage reduction in area at fracture. Results presented at Table 1 suggest the high t_p values and the reduction of stationary creep rate demonstrate the high creep resistance when it's treated by PIII treatment.

Table 1 – Creep test parameters.

treatment	σ (MPa)	t_p (h)	$\dot{\varepsilon}_s$ (1/h)	t_f (h)	E_f (mm/mm)	AR (%)
untreated	250	0.03	0.1906	0.62	0.1938	75.83
PIII	250	0.27	0.0615	1.83	0.1807	29.33
untreated	319	0.01	0.5698	0.39	0.1742	62.99
PIII	319	0.11	0.1925	0.60	0.1964	25.67

The Figures 7 and 8 show images obtained by SEM to fractograph evaluation of the alloy treated by PIII after creep test. It can be evidenced microcavities development and dimples.

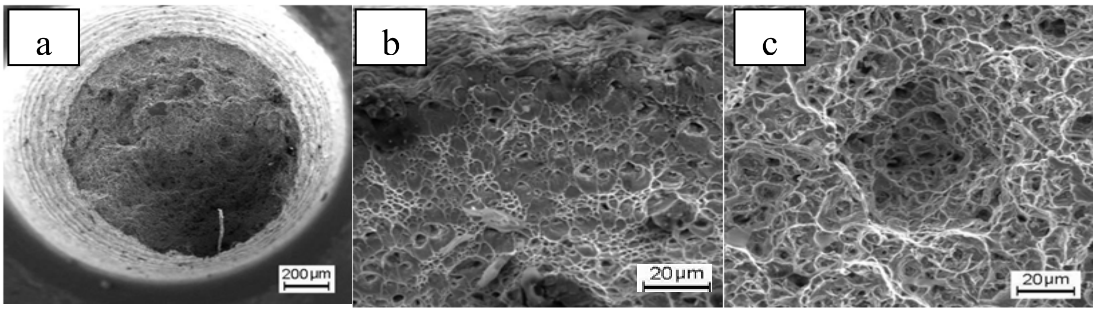

Figure 7 - Fractograph analysis of Ti-6Al-4V alloy treated by PIII after creep test at 600ºC and 250 MPa. (a) general view, (b) lateral view and (c) center view.

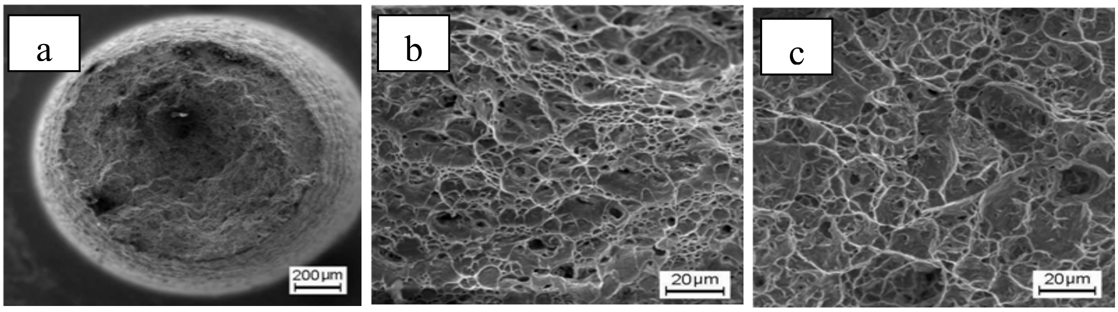

Figure 8 - Fractograph analysis of Ti-6Al-4V alloy treated by PIII after creep test at 600ºC and 319 MPa. (a) general view, (b) lateral view and (c) center view.

4 – Conclusions

The PIII treatment produces a thin layer of Ti_2N with 0.6 nm of thickness. The sample treated by PIII presents average roughness (rms) of 1.94 nm, while the untreated sample the value is 0.09 nm. The creep properties of Ti-6Al-4V treated in PIII treatment were investigated at 600°C. High temperature exposure in the treated alloy increases the creep resistance of the alloy at 600ºC in the range from 250 and 319 MPa. The alloy with PIII treatment shows greater resistance to creep and oxidation with a longer life time in creep. The fractograph evaluation of the alloy treated by PIII after creep test. It can be evidenced microcavities development and dimples with equiaxed shape. The ductile fracture is identified.

5 – References

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