INFLUENCE OF HEAT TREATMENT IN CREEP RESISTANCE OF TITANIUM ALLOY

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Abstract. *This study aimed to evaluate the resistance of a Ti-6Al-4V alloy in creep after heat treatments. It was used a Ti-6Al-4V alloy in cylindrical bars forms, forged condition and annealing at 190 °C for 6 hours and cooled in air. The microstructure of the alloy was evaluated after heat treatment and was submitted to creep tests at 600 °C and stress conditions from 125 to 319 MPa at constant load. Samples preparation for optical microscopy and scanning electron microscopy followed the usual methods of metallography. Three different conditions of heat treatments were utilized to obtain the following microstructures: Widmanstätten, Martensite and Bimodal. In the heat treated alloys the least sensitive oxidation microstructure is estimated through studies involving microstructural analysis, micro hardness and roughness. The alloy with Widmanstätten structure shows greater resistance to creep and oxidation with a longer life time in creep.*

Keywords: *creep, Ti-6Al-4V, heat treatment.*

1. INTRODUCTION

Titanium alloys are used in many industry sectors as a result of its excellent properties, e. g., high specific tension, good corrosion resistance, low specific mass and good oxidation resistance in temperatures lower than 600 ºC. These properties are decisive factors for alloys applications, particularly in aerospace industries. (Leyens and Peters, 2003).

Among titanium alloys the Ti-6Al-4V is the most important one, because holds important properties like good workability and machinability. (Sakai *et al.* 1988). However, the affinity with oxygen is one of the factors that limits its applications as components in structural materials at high temperatures. The good solid solubility of the oxygen in titanium results in materials losses and in the formation of a thin and fragile layer of considerable hardness during the exposition to air in high temperatures. (Welsch *et al.* 1988).

Aerospace industry absorbs about 75% of the world's titanium production, being the Ti-6Al-4V alloy one of the most versatile. One of the characteristics that most contributes to its growing use in the structural segments refers to its high melting point. Its use is concentrated in aerospace components in which the resistance to creep, fatigue and degradation are considered essentials. (Norris, 1994).

The heat treatment is a sequence of heating and cooling operations of a material in certain conditions with the purpose of improving special properties of the material. (Pereira, 1979). In a heat treatment the main factors that must be taken in concern are: heating, time in the treatment temperature, cooling and heating site atmosphere. The objective of the heating treatment is to change materials mechanical and structural characteristics according to its uses, like increasing or decreasing of hardness, mechanical resistance increasing, ductility and machinability improvements, heat and corrosion resistance, cutting properties and modifications in electromagnetic properties.

A creep resistance in the solid state is estimated by calculating the secondary strain rate and evaluated as a function of the load or tension applied. Thus, a static load is applied on a sample in elevated temperatures, measuring the strain as a function of time. (ASTM 1996). The selected alloy (Ti-6Al-4V) to microstructural evaluation after heat treatment was subjected to creep tests in air at 600 ºC, with constant load and conditions of 125 and 319 MPa. Complete creep test studies of the heat treated refractory alloy Ti-6Al-4V are scarce on the literature. The microstructural characterization had the objective of determine the existing phases, including characterization and quantification of the present inclusions, permitting a more detailed knowledge of the microstructure influence on creep resistance in air of the Ti-6Al-4V alloy.

2. MATERIALS AND METHODS

In this work, it was used a Ti-6Al-4V alloy in the form of cylindrical bars, acquired from the Company Multialloy Eng. Mat. Ltda., in forged condition, annealed at 190 ºC for 6 hours and cooled in air. The microstructural configuration of the resulting thermal and mechanical treatments is the condition for applications in aerospace industry. The characterization on the chemical composition of major elements (% p) is in according with the requirements of ASTM B265-89. (ASTM 1989).

2.1. Heat treatment

Ti-6Al-4V samples were used as shown in “Fig. 1”. It is used the refractory cylindrical furnace of Lindberg / Blue trade mark to the treatment of the samples. They were put in a quartz tube, and it was used a vacuum pump to remove air from the quartz tubes at the encapsulation. Argon was injected in the quartz tubes to avoid oxidation of the samples. A pickling solution of HF- 0.2 mL/HNO_3- 2 mL / H_2O-30 mL was used to wash the samples after the treatment. Water was used for the fast cooling of the samples.

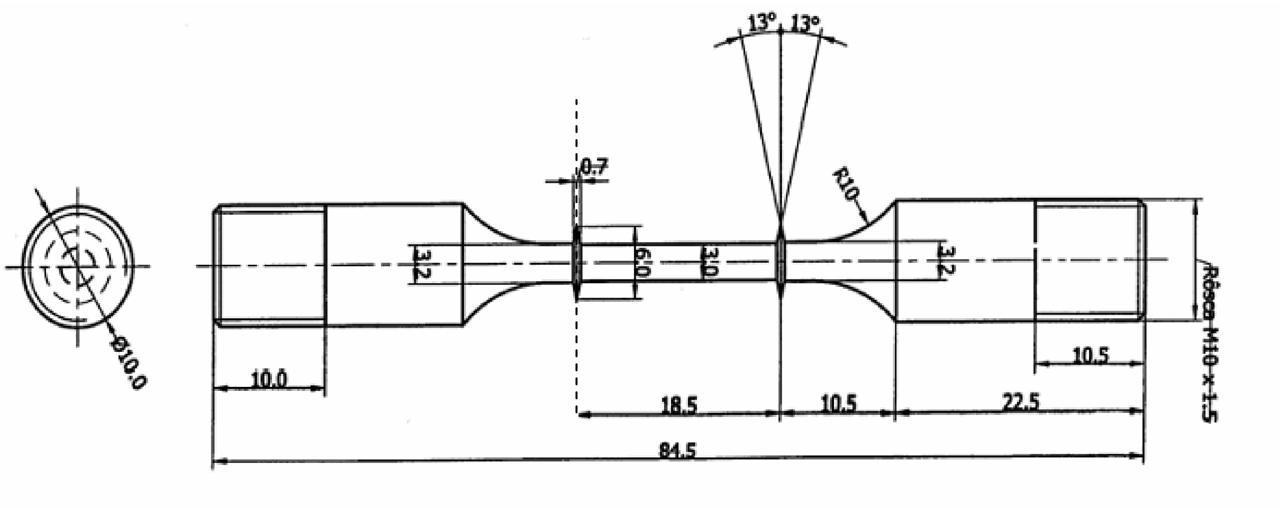

Figure 1. Schematic view of the specimen creep test.

2.1.1. Heat treatment #1

The samples were put in a quartz tube using a torch. The air inside the tube was removed with vacuum pump; after the air withdrawal argon gas was injected in for protection against oxidation.

The Lindberg / Blue furnace was heated up to 1050 °C and the samples were heated for 30 minutes. After this time samples were cooled inside the furnace at a rate of 6 Cº/ min until room temperature. The samples already at room temperature were removed from the furnace and put in acid solution.

2.1.2. Heat treatment #2

Similarly to the first heat treatment, the samples were encapsulated in a quartz tube and placed in inert atmosphere of argon. The Lindberg / Blue furnace was heated to 1050 °C and the samples were placed in the furnace and left for 1 hour. After the determined time, they were cooled in water until room temperature.

2.1.3. Heat treatment #3

The heat treatment 3 was conducted in two steps:

A. **1st Step:** The refractory furnace was heated up to 950 ºC, the samples were placed inside it and left for an hour. After this time they were cooled in water until room temperature, and after put in acid solution.
B. **2nd Step**: The samples were again encapsulated in inert atmosphere. The furnace was set to 600 ºC and the samples were put inside the furnace during 24 hours. After this time were removed and cooled in air until room temperature.

2.2. Oxidation study

It was estimate the microstructure more sensitive to oxidation after studies of microstructural analysis, micro hardness, roughness and weight gain. Initially the alloys were subjected to temperatures of 500, 600, 700 and 800 º C for 48 hours in the furnace with air atmosphere. After this period were measured the weight gain. The values of micro hardness were measured in the Futuretech FM model instrument with load of 300 g. The roughness was measured in a Surtronic 3 +, Taylor Hobson's trade mark which has a feeler with diamond tip of 5.0 µm.

2.3. Creep test

Creep tests were realized using MAYES machines. In the furnace were adapted electrical systems and controllers according to ASTM E139 standardization. (ASTM 1990). 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 accordance to the standard ASTM E139. (ASTM1990).

2.4. Metallographic preparation

The preparation of samples for analysis by optical microscopy and scanning electron microscopy followed the usual patterns of metallographic: hot pressing (150º C and 21 MPa), followed by hand sanding with sandpapers based on SiC, following 120, 240, 320 400, 600 and 1200 mesh. 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

3.1. Heat treatments

Different structures were obtained due to the conditions employed in each heat treatment. “Table 1” shows the conditions of heat treatments and the microstructures obtained. “Figure 2” shows an annealed Ti-6Al-4V alloy micrograph obtained by optical microscopy.

Table1. Relationship between heat treatment conditions and structure obtained.

Heat treatment conditions		**Obtained structure**
Treatment #1	Heating to 1050 ºC for 30 min.; Cooling rate of 6 ºC per minute in furnace.	Widmanstätten
Treatment #2	Heating to 1050 ºC for 1 h; Cooling in water.	Martensite
Treatment #3	Step 1: Heating to 950 ºC for 1 h; Cooling in water. Step 2: Heating to 600 ºC for 24h; Cooling in air.	Bimodal

The Ti-6Al-4V alloy was submitted to three different heat treatments for evaluation of different microstructures in the material. The structures obtained were Widmanstätten, Martensite and Bimodal, as shown in Table 1.

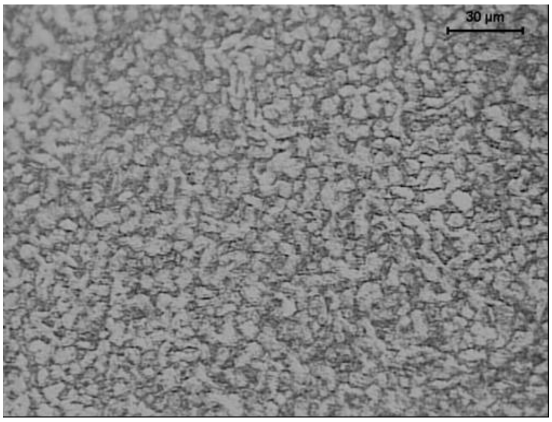

Figure 2. Ti-6Al-4V alloy micrograph as received.

Figure 2 shows the microstructure of the annealed Ti-6Al-4V alloy. It could be observed α grains (HCP) and dark regions that define the presence of β phase (BCC) along the grain boundaries of the alloy.

"Figure 3" shows images obtained by optical microscope from different structures obtained in the performed heat treatment.

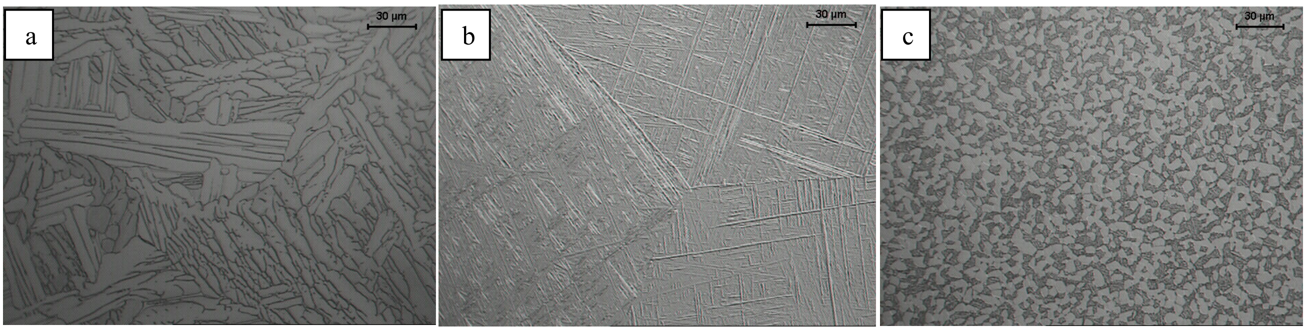

Figure 3**.** Structures obtained in heat treatments: a) Widmanstätten b) Martensite and c) Bimodal.

It could be observed in Figure 3 that the Widmanstätten structure has a larger average grain size, about 395 μm, and it can lead a greater resistance to oxidation of the alloy. Increasing grain size promotes decreasing in the activation energy of the grain boundary and then the material will react fewer with the environment.

3.2. Oxidation studies

"Figure 4" shows the weight gain measures of the microstructures obtained by heat treatment after 48 hours oxidation test.

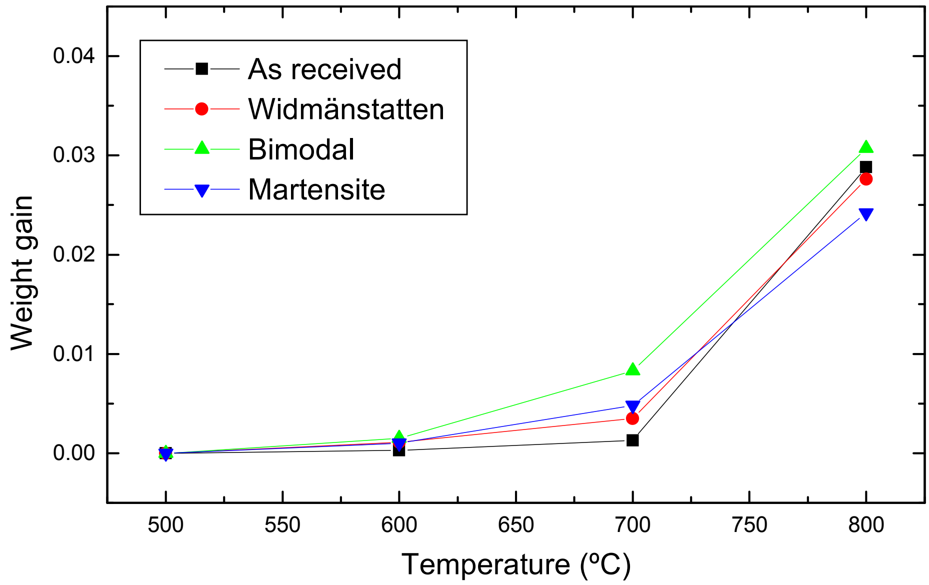

Figure 4. Weight gain measures of microstructures as a function of oxidation temperature for 48 hours.

Figure 4 shows the weight gain for the titanium alloy when exposed at temperatures up to 600º C. Nitrogen and other species are less sensitive than the oxygen used in these conditions. The absorbed oxygen is chemically combined with titanium alloys and other materials to form a layer of oxide on the surface, in addition to interstitial diffusion in the metal. (Pitt, 2004). The results at 500 and 600 ºC did not show a significant difference in weight gain among the other structures studied. At 700 and 800 ºC it is observed that the Bimodal structure shows a greater weight gain. At 800 ºC the structure of Martensite and Widmanstätten have a smaller weight gain indicating a tendency of these structures to a greater resistance to oxidation.

"Figure 5" shows the measures of micro hardness (HV) of microstructures obtained by heat treatment depending on the temperature of oxidation.

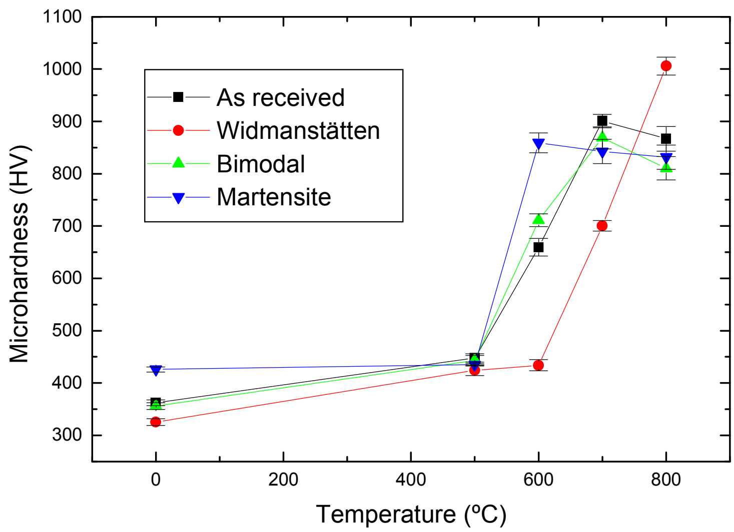

Figure 5. Micro hardness measures of the microstructures obtained by heat treatment.

The oxygen diffused in the alloy increases its hardness; the amount of oxygen is proportional to the hardness in titanium and can be estimated by measures of micro hardness. (Pitt, 2004). Based on this the results observed in Figure 5 show that the micro hardness increases with oxidation temperature, but there is not much difference in the behavior of different microstructures. However the structure of Widmanstätten at 800 °C gives a higher value as a function of the oxide layer formed. It is possible that the lower values of hardness at 500, 600 and 700 °C, in absolute values, results of thin layers of oxidation due to increasing of resistance to the process and the type of formed oxide. The metal combines with oxygen to form a long series of oxides with the stoichiometry of TiO to Ti_7O_{12}. (Abkowitz *et al.* 1995). Observing the values of micro hardness in the Widmanstätten structure it can be considered as the most resistant structure to oxidation.

"Figure 6" shows the measures of roughness (µm) of the microstructures obtained by heat treatment as a function of oxidation temperature.

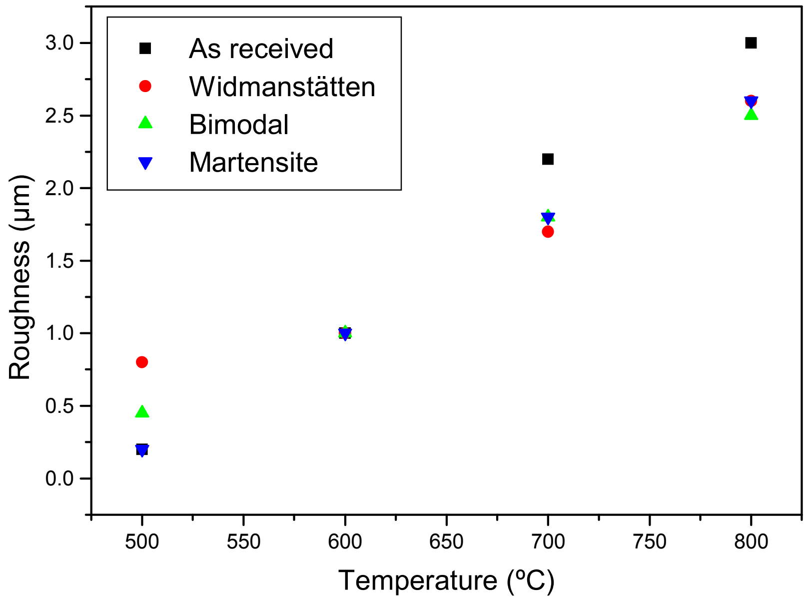

Figure 6. Roughness measures of the microstructures obtained by heat treatment as a function of oxidation temperature.

Measures of roughness shown in Figure 6 increase proportionally increasing the temperature for all the obtained structures in the heat treatment.

3.3. Creep tests

"Figure 7" shows the creep curves of the Ti-6Al-4V alloy at 600 ° C and 250 MPa of Widmanstätten and Martensite structures and as received, corresponding to the real strain ε as function of time t.

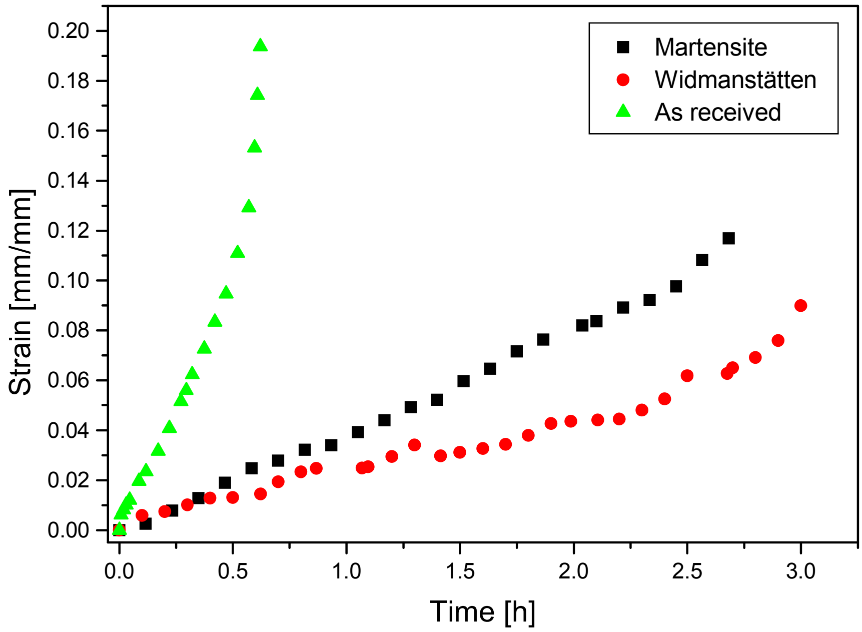

Figure 7. Creep curves of the Ti-6Al-4V alloy at 600 ºC and 250 MPa as received and with structures: Martensite and Widmanstätten.

"Figure 8" shows the creep curves of the Ti-6Al-4V alloy at 600ºC and 319 MPa of Widmanstätten, Martensite and Bimodal structures and as received, corresponding to the real strain ε as function of time t.

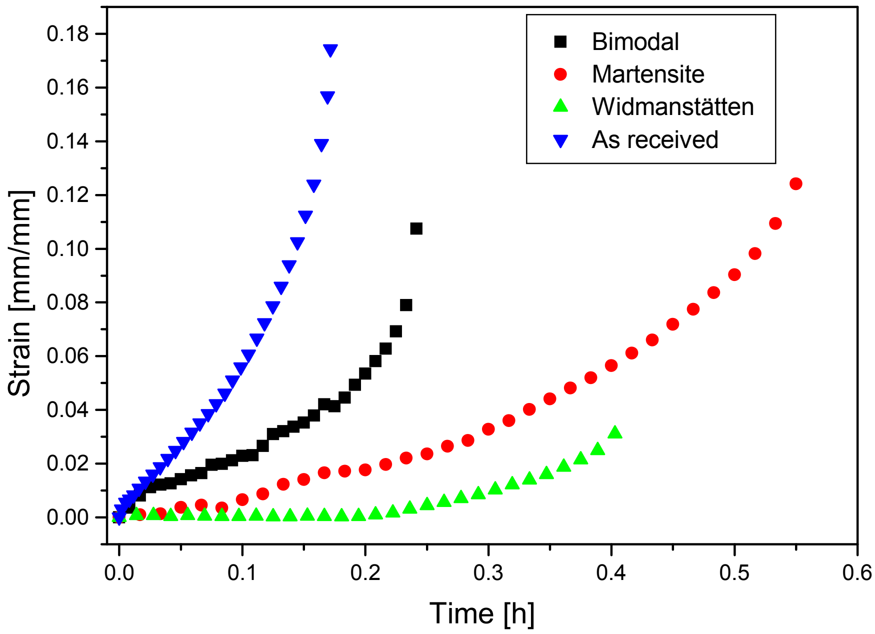

Figure 8. Creep curves of the Ti-6Al-4V alloy at 600ºC and 319 MPa, as received and with structures: Widmanstätten, Martensite and Bimodal.

The creep curves in Figure 7 and 8 show that the Widmanstätten structure at 600 °C, 250 MPa and 600 °C, 319 MPa, respectively, presents greater resistance to creep. 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. The results presented in Table 2 and 3 shows the highest values of t_p and the reduction of the steady-state creep rate demonstrate that the higher creep resistance of Ti-6Al-4V is observed in strain hardening Widmanstätten structure. "Figure 9" shows the creep curves of the Ti-6Al-4V alloy at 600ºC and 125, 222, 250 and 319 MPa of Widmanstätten structure, corresponding to the true strain ε as function of time t.

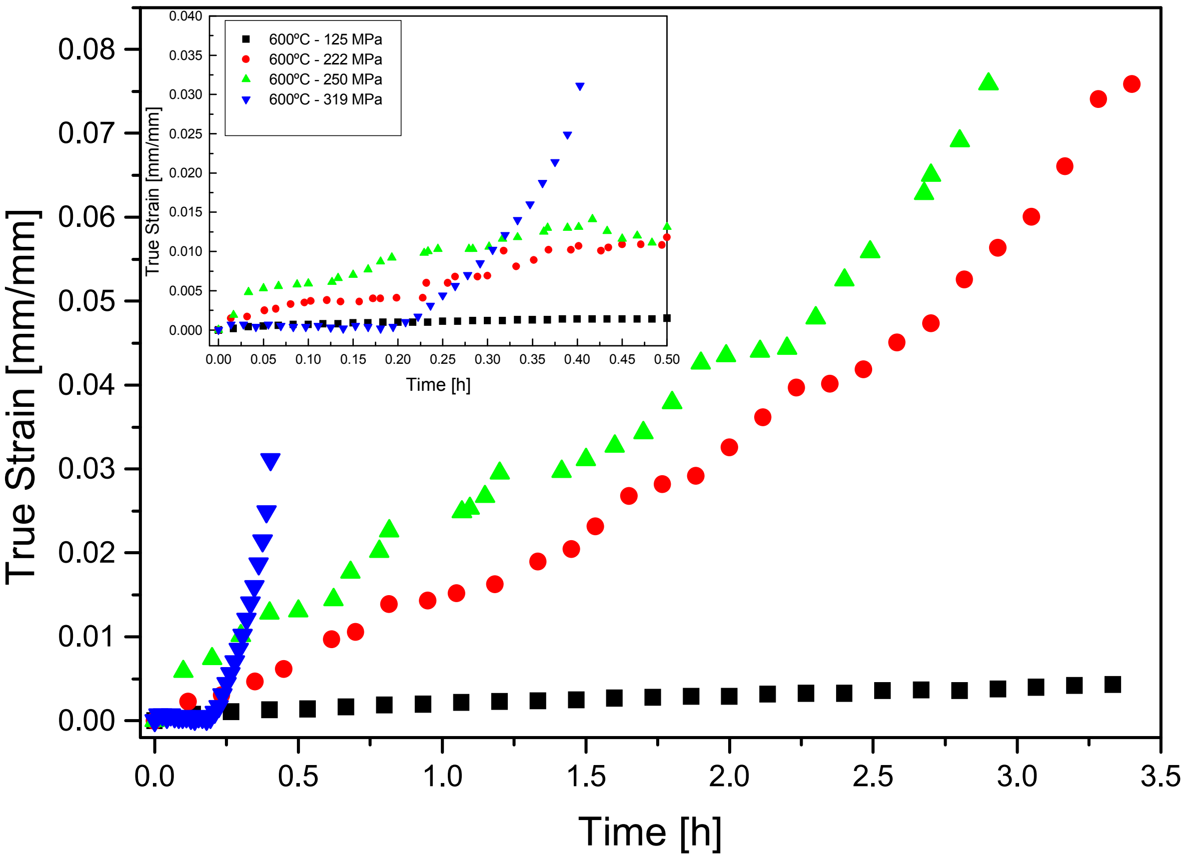

Figure 9. Creep curves of the Ti-6Al-4V at 600ºC and 125, 222, 250 and 319 MPa with Widmanstätten structure.

The following tables 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.

Table 4 shows that the highest values of t_p and during primary creep is dependent on the test stress.

Table 2. Creep data of Widmanstätten and Martensite at 600ºC and 250 MPa.

Treatment	**t_p (h)**	**$\dot{\varepsilon}_s$ (1/h)**	**t_f (h)**	**ε_f (mm/mm)**	**AR (%)**
Widmanstätten	0.29	0.0182	3.00	0.0899	9.67
Martensite	0.23	0.0405	2.68	0.1169	16.00
As received	0.05	0.1937	0.62	0.1938	75.83

Table 3. Creep data of Widmanstätten, Martensite and Bimodal at 600ºC and 319 MPa.

Treatment	**t_p (h)**	**$\dot{\varepsilon}_s$ (1/h)**	**t_f (h)**	**ε_f (mm/mm)**	**AR (%)**
Widmanstätten	0.03	0.0525	0.40	0.0311	11.33
Martensite	0.07	0.1135	0.55	0.1241	10.00
Bimodal	0.03	0.2201	0.24	0.1074	39.33
As received	0.02	0.5459	0.17	0.1742	62.99

Table 4. Creep data of Widmanstätten at 600 °C and different σ.

σ (MPa)	**t_p (h)**	**$\dot{\varepsilon}_s$ (1/h)**	**t_f (h)**	**ε_f (mm/mm)**	**AR (%)**
125	1.8	0.00058	-------	--------	--------
222	0.35	0.01793	3.40	0.0759	10.00
250	0.29	0.01820	3.00	0.0899	9.67
319	0.03	0.05250	0.40	0.0311	11.33

4. CONCLUSION

The creep properties of Ti-6Al-4V treated in three different conditions of heat treatments were investigated at 600 °C. The structures obtained were Widmanstätten, Martensite and Bimodal. High temperature exposure in Widmanstätten structure increases the creep resistance of the alloy at 600ºC in the range from 125 to 319 MPa.

In the heat treated alloys the least sensitive oxidation microstructure is estimated through the studies involving microstructural analysis, microhardness and roughness. The alloy with Widmanstätten structure shows greater resistance to creep and oxidation with a longer life time in creep.

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