

## DEFORMATION AND FRACTURE OF AN ALPHA/BETA TITANIUM ALLOY

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### Abstract

Titanium alloys are used in the aero-spatial, energy and biomaterial industries among others and exhibit high specific strength and fracture toughness. Their mechanical properties show a strong dependence on the microstructure, especially on the size and morphology of the constituent phases. An experimental evaluation was done to a better understanding of that influence using some techniques like as transmission electron microscopy (TEM), both low and high resolution (HR), scanning electron microscopy (SEM), coupled to electron back-scattering diffraction (EBSD), X-ray diffraction (XRD) and optical microscopy (OM). Some in-situ TEM deformation studies were also done. The alloy was submitted to two heat treatment conditions to get different phases distribution. An hcp phase (alpha) in coexistence with a bcc phase (beta) was observed after both treatments as well the occurrence of twins, stacking faults and dislocations arrangements. The work then discusses the influence of these features on the overall alloy strength.

Keywords:  $\alpha/\beta$  titanium alloy; Ti-6Al-4V; transmission electron microscopy

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## 1. Introduction

The class of  $\alpha+\beta$  titanium alloys are the most common in the various types of titanium alloys. Commercial Ti-6Al-4V is best known for having compact hexagonal crystal structure (alpha phase) and body-centered cubic (beta) present in its microstructure at room temperature, combining strength and corrosion resistance and formability with machinability. With excellent combinations of strength / weight ratio and excellent corrosion resistance, titanium alloys have been a great attraction for applications in complex industrial components.

As cast titanium alloys exhibit beta ( $\beta$ ) grains for large regions with fine lamellar structures within the grain, and consequently, modest mechanical properties. The application of fast annealing reverses the structure of titanium alloys, causing a change in the arrangement of phases within the beta grains, thereby increasing the mechanical strength. The decline in the gross structure of the cast by rapid annealing and subsequent increase in strength by the martensite formation are results from the rapid cooling from  $\beta$  and ( $\alpha + \beta$ ) fields [1].

The transformation of martensite is directly related to other reactions that depend on the microstructure and chemical composition of the alloy. To evaluate the occurrence and influence of martensite and other morphologies of the  $\alpha$  phase, formed within the ( $\alpha + \beta$ ) and  $\beta$  fields, some parameters are important such as the transformation temperature, the cooling rate, the mechanical properties and corrosion resistance for biomaterials. Monotonic tensile tests, hardness and impact tests and the immersion of the material with different treatment conditions in corrosive media, provide data related to the optimum condition of heat treatment [2].

This paper undertakes an assessment of micromechanisms of deformation and fracture of titanium Ti-6Al-4V alloy in order to study the influence and behavior of the different phases, using electron microscopy.

## 2. Experimental

Ti-6Al-4V alloy was received in the form of extruded bars of 6 mm in diameter. The initial state of the material is annealed at 800°C for two hours.

### 2.1. In situ Transmission Electron Microscopy

A scheme of the sample prepared for analysis of in situ tensile loading in TEM is shown in Fig.1. Tensile tests of Ti-6Al-4V heat treated at 1000°C for one hour and cooled in water have been performed at room temperature with a Gatan straining holder and TEM observations were made with a Jeol 200C electron microscope.

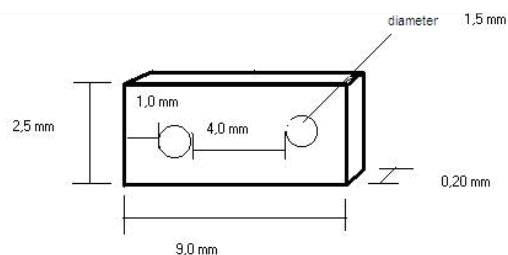


Fig. 1 Scheme of specimen for in situ TEM tensile test

The sample after cutting was submitted to the following steps: mechanical thinning, sample grinding with 600 grit emery paper up to a thickness of 0.2 mm, and final thinning and drilling in a twin jet electropolishing device. Samples with thickness of 0.2 mm were coated with amorphous carbon through the system of "sputtering" and the analysis carried out in two electron microscopes JEOL 200C and JEM 2100 HRTEM.

A region of the specimen was chosen which shows the beginning of the deformation experienced by the sample during the in situ tensile test, and 0.05 mm displacements were applied from the initial position, generating cumulative shifts of 0.05mm, 0.10 mm, 0.15 mm, 0.20 mm and 0.25 mm.

## **2.2. Optical Microscopy and Scanning Electron Microscopy**

The specimen were cut with diamond disc and then its surfaces were grinded with different particle size silicon carbide (SiC) papers. After grinding the samples were polished with diamond paste. To reveal the structure a chemical attack was carried out, using a mixture of 10 mL of hydrofluoric acid (HF), 10 mL nitric acid (HNO<sub>3</sub>) and 30 mL of lactic acid (C<sub>3</sub>H<sub>6</sub>O<sub>3</sub>). In revealing the structure of the heat treated samples, chemical attack was carried out using a mixture of 10 mL of hydrofluoric acid (HF), 10 mL nitric acid (HNO<sub>3</sub>) and 85 mL of water; before SEM analysis the samples were coated with amorphous carbon, for observation of the main phases present. The following equipments were used: Olympus (model BX 60) Optical Microscope, a system of digital image capture and Philips XL-30 Scanning Electron Microscope (SEM) with EDS.

## **2.3. Transmission Electron Microscopy and High Resolution Transmission Microscopy**

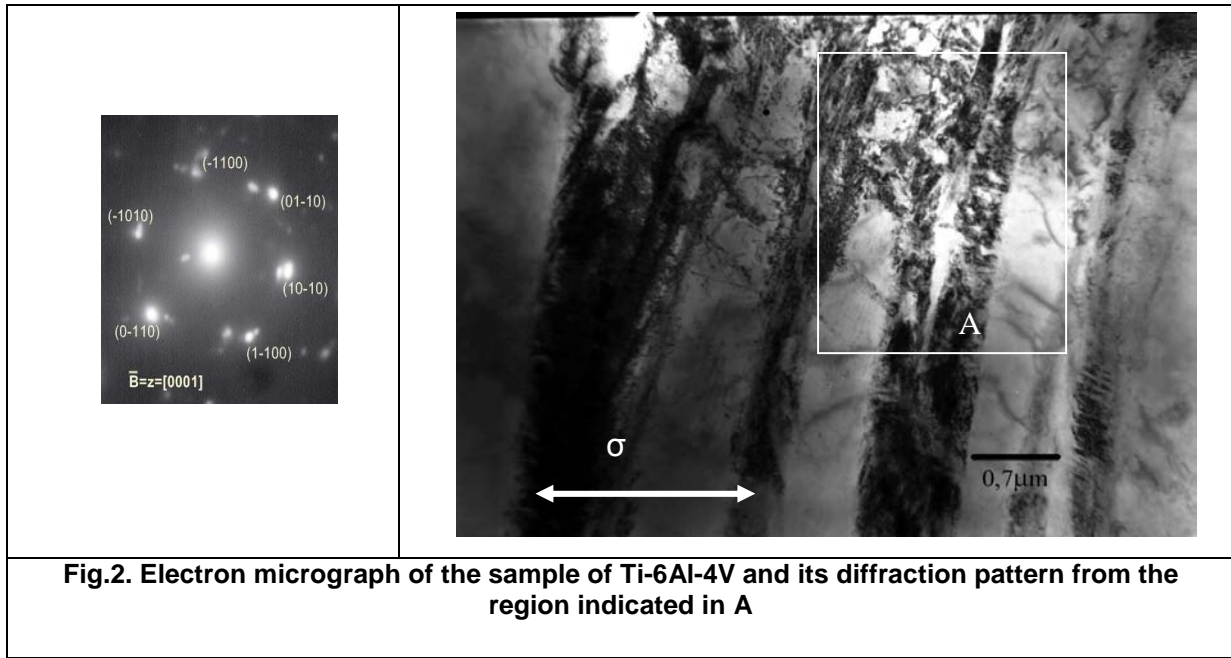
Some 0.5 mm thickness sections were cut from the samples, with a diamond disc and 3 mm diameter disc specimens were machined by electroerosion. The samples were grinding with 600 grit emery paper to reach a thickness of 0.2 mm and in sequence were subjected to final thinning to the adequate thickness and drilling. They were then coated with amorphous carbon via a sputtering system and analyzed in both TEM equipments.

## **3. Results and discussion**

### **3.1. In situ Transmission Electron Microscopy analysis**

To investigate the nature of deformation modes and the associated elementary processes, TEM was largely used. In situ tensile tests were carried out at room temperature. The dislocation dynamics and deformation micromechanisms under stress and temperature can only be studied by this technique [3].

Ti-6Al-4V dislocation substructure and its diffraction pattern are shown in Fig.2. The double ended arrow indicates the applied stress ( $\sigma$ ) direction.

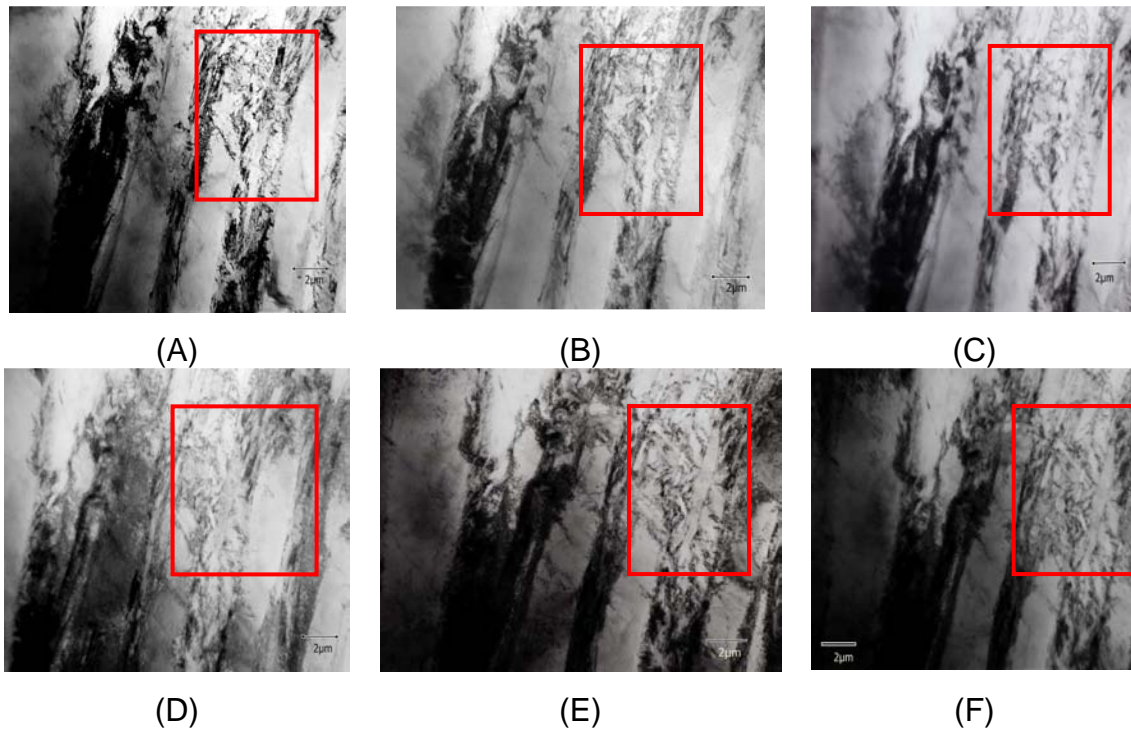


**Fig.2. Electron micrograph of the sample of Ti-6Al-4V and its diffraction pattern from the region indicated in A**

As a result of the diffraction pattern in the region A of the sample on the alpha phase (hcp), it can be observed slip in the hcp alpha phase of prismatic and basal planes  $(\bar{1}100)$  and  $(10\bar{1}0)$ , direction of observation  $\bar{B} = [0001]$ . Different regions of the alpha phase and beta phase can be observed in the vertical lamellae in Fig.2.

The possibility of slipping occurring in prismatic, pyramidal or basal planes is generally associated with the analysis of local shear stresses. One of the difficulties of identifying the deformation mechanisms in pure titanium or its alloys is related to the complex competition between the different slip systems [4].

Fig. 3 (A-F) shows that after applying the displacement, the density of dislocations become more intense as the applied stress increases. It was noted that in addition to the slipping of planes, deformation of grains also occurred.



**Fig. 3** Electronic micrographs of Ti-6Al-4V sample on the analysis of in situ traction for different shifts: (A) 0.05mm, (B) 0.10 mm, (C) 0.15 mm, (D) 0.20 mm, (E) and (F) 0.25 mm.

### 3.2. Optical Microscopy and Scanning Electron Microscopy analysis

In analysis of the specimen surface by optical microscopy the presence of two phases was observed. The microstructure is a mixture of ( $\alpha + \beta$ ) and  $\beta$  phase evenly distributed in the  $\alpha$  matrix. An acicular martensitic microstructure was revealed, ie, the structure shows the presence of thin lamellae resembling needles, characteristic of alpha structure, as can be seen in Fig. 4.

Studies on the microstructural evolution in alloy Ti-6Al-4V by thermo-mechanical process, at temperatures of 850 °C, 900 °C, 950 °C and 1000 °C were previously done and authors report that the deformation behavior and transformation of primary lamellar alpha phase during the heat treatment is very complex [5]. There is also report that there are two possible mechanisms for the separation of shorter alpha-type plates. One of these mechanisms occurs during hot deformation promoting the emergence of low and high angles between the  $\alpha$  plates, ranging from a few degrees to about 30°. The  $\beta$  phase can then come into  $\alpha$  phase in this region along the contours [6].

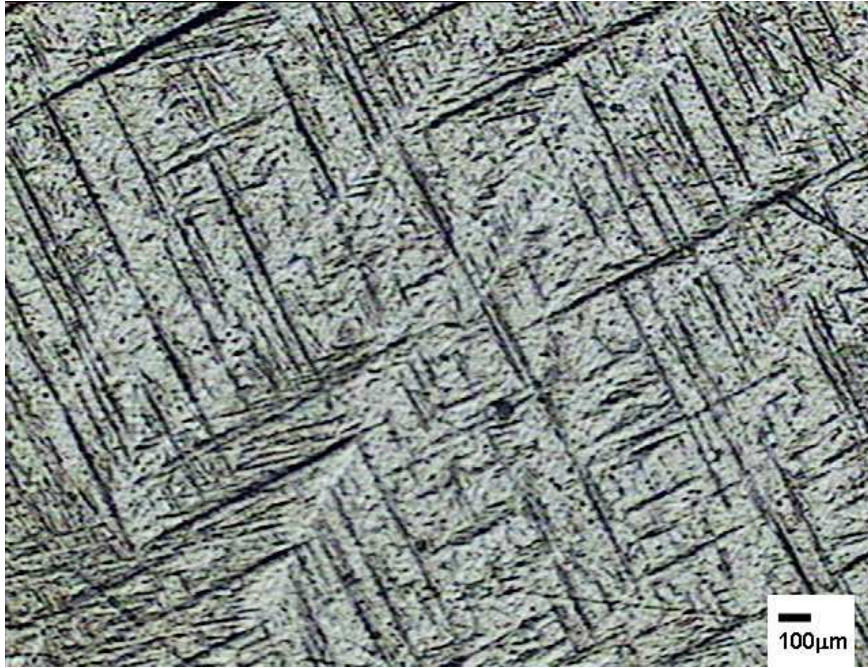


Fig. 4. Optical micrograph of the sample of Ti-6Al-4V heat treated at 1000 ° C for two hours and cooled in water submitted to surface chemical attack

The presence of the  $\beta$  phase homogeneously distributed in the  $\alpha$  matrix is shown Fig. 5 for Ti-6Al-4V annealed at 800°C for two hours.

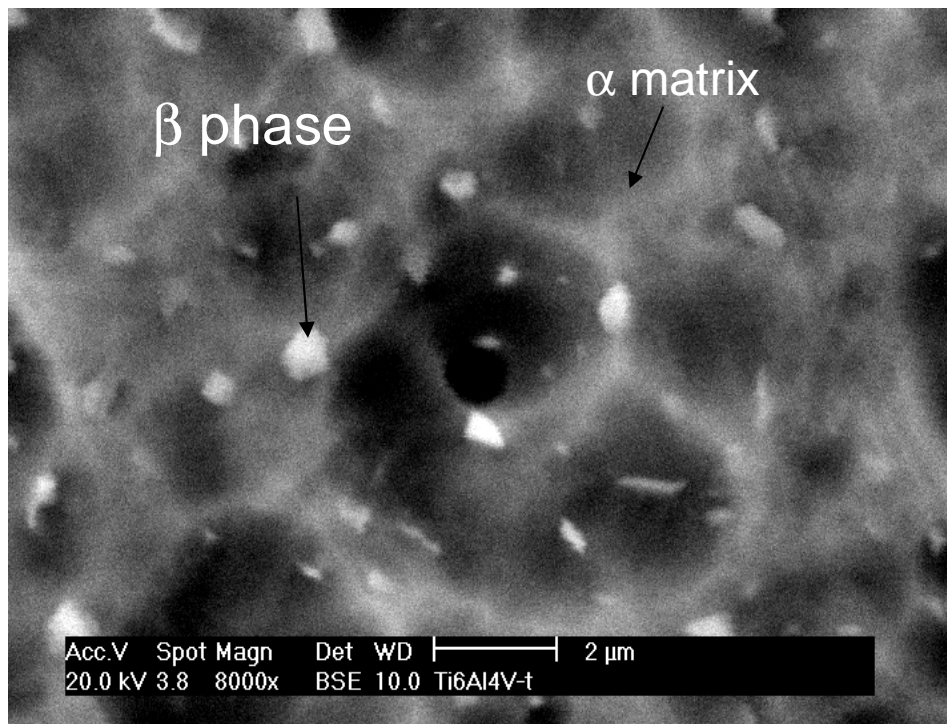


Fig 5. SEM image of microstructure of the sample Ti-6Al-4V annealed at 800°C for two hours

Fig. 6 shows the  $\alpha$  phase and its crystallographic orientation distribution in Ti-6Al-4V. This analysis is obtained by electron backscattered diffraction (EBSD) in the SEM, and the basal planes (0001) are indicated by red color.

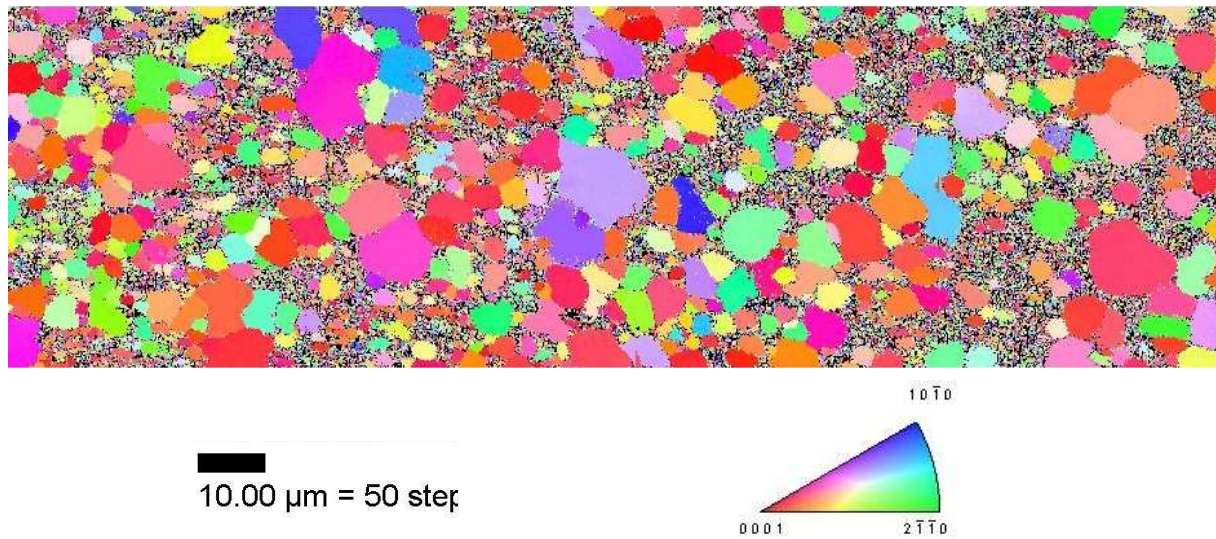


Fig 6. SEM/EBSD image of Ti-6Al-4V microstructure annealed at 800°C for two hours

The presence of large areas with steps of the same orientations on the fracture surface of the alloy, as seen in Figure 7, reveals the heterogeneously grain structure (a few large grains across the sample thickness) and the failure mode associated with the tensile loading.

The morphology of steps on the surface of fracture depends on the loading orientation, the effectiveness of the slip process in the grains and the texture of titanium alloy [7].

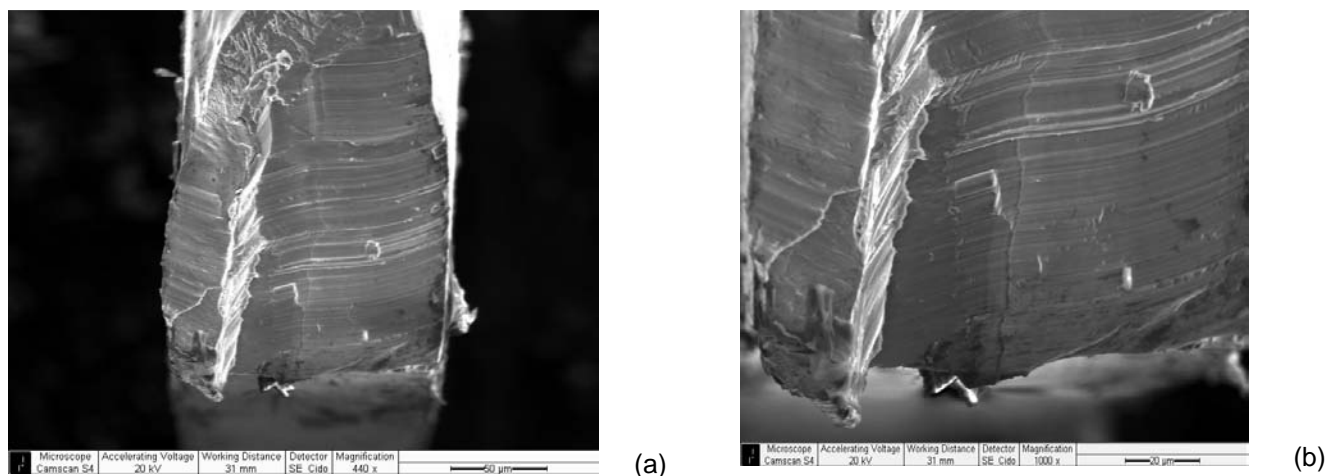
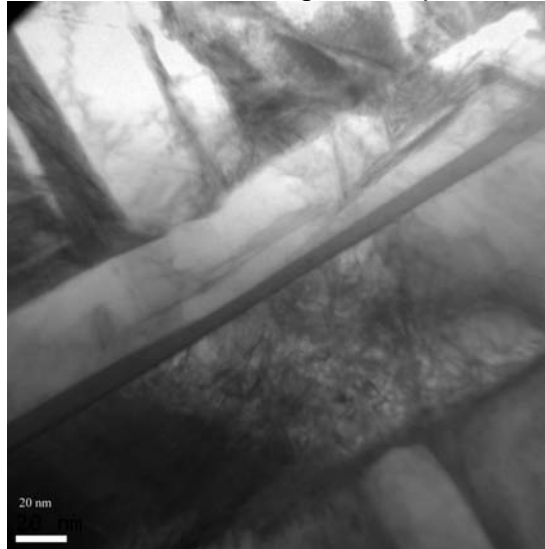


Fig 7. SEM micrograph of Ti-6Al-4V, after heat treatment at 1000 °C/1h and rapid cooling in water at two different magnifications

### 3.3. Transmission Electron Microscopy Analysis (Conventional and High Resolution)

Samples of Ti-6Al-4V in two different conditions: the as-received condition, i.e. heat treated at 800° C for two hours and the other annealed at 1000°C for one hour, submitted to the same water cooling were prepared for analysis in the TEM. The following electron micrographs show the change experienced by the alloy after heat treatment.

Fig. 8 shows overlapping planes with the presence of dislocation areas. This behavior can be attributed to the cooling rate imposed on the sample.



**Fig 8. Electron micrograph of the sample of Ti-6Al-4V, after heat treatment 1000 °C/1h and rapid cooling in water.**

In Fig. 9a, observed by high resolution TEM, it is clear the orientation of crystal planes of Ti-6Al-4V. Note the presence of sub-grains in the dark regions (indicated by arrows). By measuring the angles between the planes, values ranging from 60° to 70° are found. This variation can be explained by the proximity to regions where there is the presence of sub-grains. Some example of twinning in the  $\alpha$  phase is shown in Fig.9b.

In the case of Ti-6Al-4V exists factors, in the martensitic structure such as the prior existence of dislocations and  $\alpha'/\alpha'$  interfaces which can reduce the activation energy required for nucleation of (1011) twins [2]. It is believed that this observation can not be done in tests with pure titanium.



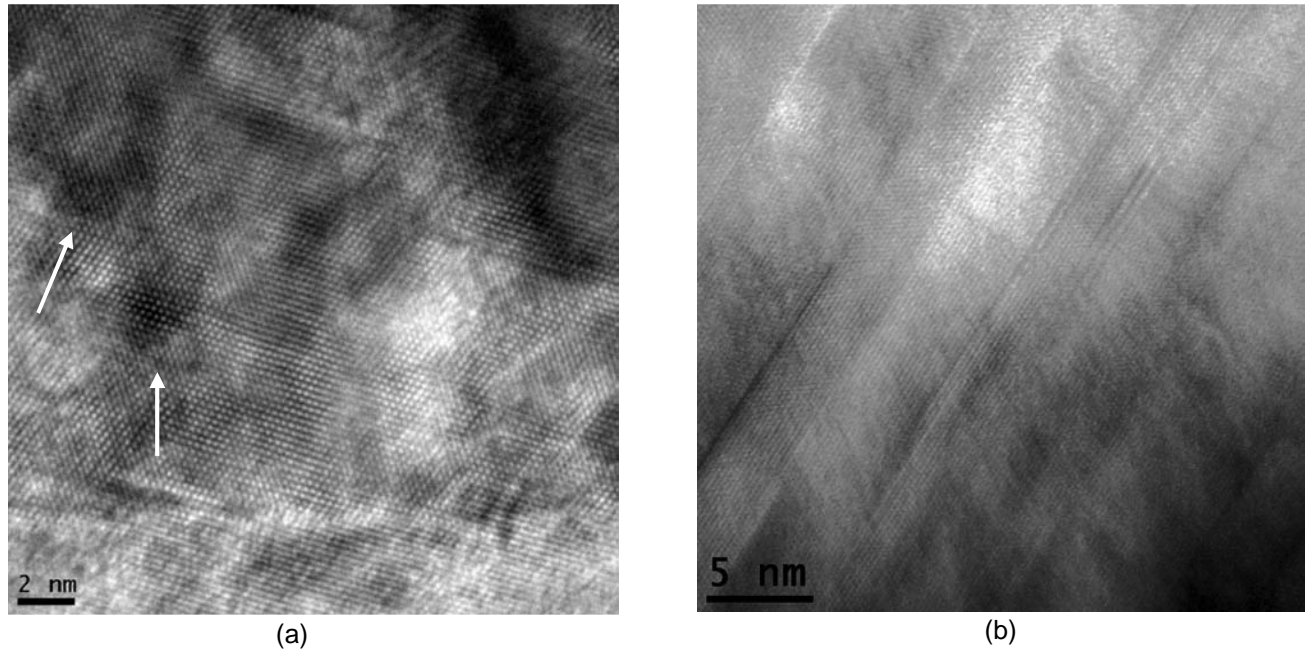


Fig 9. Electron micrograph of the sample of Ti-6Al-4V, after heat treatment 1000 °C/1h and rapid cooling in water.

#### 4. Conclusion

In situ studies of deformation and fracture by TEM techniques allowed the determination of the presence of alpha phase with lamellar morphology formed heterogeneously in the  $\beta \rightarrow \alpha$  transition and the formation of secondary  $\alpha$  phase, formed during cooling, from the ( $\alpha + \beta$ ) field, applied to the sample. In situ tensile test allowed to observe the presence of twins and dislocations, generated by the sliding of prismatic and basal planes in the  $\alpha$  phase since these planes require low critical shear stress to slip. Lamellar microstructure formed during cooling from the  $\beta$  field, was observed and it contributes to the conversion of part of the secondary ( $\alpha + \beta$ ) structure, which was trapped between the lamellae of  $\alpha$  field. For the different conditions of heat treatment applied to the alloy, the variations were observed between the amount of phases  $\alpha$  and  $\beta$ , in relation to the initial microstructure of the material.

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