International Congress of Science and Technology of Metallurgy and Materials, SAM - CONAMET 2013

Thermomechanical Processing of Alloys. Case of Study Ti Gr. 4

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Abstract

Titanium alloys are characterized by great versatility arising from the ability to obtain a wide spectrum of properties by controlling the alloying elements and thermo-mechanical treatments that determine the microstructure. The determining factors for the workability of titanium alloys are related to the structure of the alpha phase (HCP) and the strong dependence of the alpha and beta phases on processing variables such as temperature, strain and strain rate. Therefore, proper selection and control of these parameters in each deformation step is critical to obtain an optimum combination of mechanical properties and microstructure of the final product.
The tested material is titanium commercially pure (Ti CP (Ti Gr4)), which despite being an alpha alloy, it is seen that small amounts of iron can change the microstructural evolution of the alloy.

1. Introduction

1.1 Hot workability

Hot workability of a metal or alloy is the capability to be deformed under conditions of high temperature and relatively high strain rates. The two characteristics that govern hot workability are deformation resistance and ductility. In turn, these depend on the process and metallurgical variables. Within the variables of material that affect workability are mechanisms of fracture, the localized flow and metallurgical considerations (grain size and structure).

Pure metals and single-phase alloys have the best ductility, while alloys with low melting point phases tend to be difficult to deform and have a restricted working range. In general, when increasing the solute content of the alloy, increases the possibility of forming low melting point phases and also of second phases, which results in a decreased region of good workability.

During the plastic forming of ingot or continuous casting products and the subsequent deformation steps, segregation, second phase particles, inclusions and the same crystal structure (grains) are aligned in the direction of largest flow of material (lines flow), which produce directional variations in properties such as strength, ductility, impact resistance and fatigue. This

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anisotropy in the properties is higher between the longitudinal direction of plastic forming and the cross section. (Merlone et al.(1993))

The process-dependent parameters are deformation (strain), strain rate, temperature, friction, stress state and yield criteria. (Dieter et al. (2003))

The plastic deformation aims to give the desired shape to the material and improve or control the properties of the product. The strain rate is the speed at which the deformation takes place. The yield point is strongly affected by the strain rate, so that it becomes an important variable in determining the workability and in some trials difficult to control.

Hot working refers to deformation under conditions of temperature and strain rate such that recovery processes occurring simultaneously with the deformation. While the temperature increases, the mechanical strength decreases and the ductility increases, above a certain temperature structural changes may alter this behavior (biphasic zones, precipitates, etc.).

Friction is a key factor in any industrial deformation process between the workpiece and the tooling that applies the deformation force. The relative movement is impeded by the contact under pressure. The existence of friction increases the value of the resistance to deformation and makes the deformation more inhomogeneous, increasing the tendency to fracture.

The knowledge of the metallurgical phenomena described that take place during hot forming has led to consider these processes as thermomechanical treatments in which strains are combined with temperatures and strain rates at which are performed to obtain a product with appropriate structure to further use. (Merlone et al.(1993) and Dieter et al. (2003))

1.2 Titanium and its alloys

Pure titanium exhibits an allotropic transformation at 882°C, at which it changes from a body-centered cubic structure (BCC) (high temperature beta phase) to a hexagonal close-packed structure (HCP) (low-temperature alpha phase). The transformation temperature is strongly influenced by substitutional and interstitial elements.

Alloying elements can be classified as alpha stabilizers, or beta stabilizers. Alpha stabilizers such as aluminum, oxygen and nitrogen, increase the temperature at which the alpha phase is stable. Moreover beta stabilizers such as molybdenum and vanadium, stabilize the beta phase at lower temperatures. The minimum temperature at which there is 100% beta phase is known as beta transus and has fundamental importance in hot working and heat treatment of titanium alloys. Alpha stabilizers tend to increase the beta transus while beta stabilizers tend to lower it. Below the beta transus titanium is a mixture of alpha and beta or 100% alpha if it contains no beta stabilizer. (Donachie (2000)) The interstitial elements such as oxygen, nitrogen and carbon elevate the mechanical strength of titanium. Regarding the beta stabilizers they can be classified into two groups: isomorphous and eutectoids. Tin and zirconium are considered inert from the point of view of phase stabilization although they are transformation retardants and hardening agents. (Lutjering et al. (2007))

Substitutional alloying elements such as tantalum and vanadium play an important role in the control of the microstructure and properties of titanium alloys. Tantalum, vanadium and niobium are isomorphous beta and do not form intermetallic. Eutectoid systems are formed with chromium, iron, copper, nickel, palladium, cobalt, manganese and other transition metals. These are incorporated into the alloys by isomorphous beta stabilizers to stabilize the beta phase, and prevent the formation of intermetallic during service at elevated temperature. (Donachie (2000))

Iron forms an eutectic system with titanium, however there is no literature (Polmear (1995) and Totten et al. (2010)) which suggests that iron behaves as an alloying isomorphic because the eutectic reaction, at 600°C, (Collins (2003) and ASM Handbook (2003)) is very slow and does not manifest itself during heat treatments or during the service. Figure 1 shows a micrograph which shows temperature beta phase intermetallic Ti_2Fe instead. (Lutjering et al. (2007))
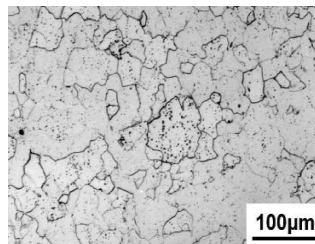

Fig. 1: Titanium Gr. 3 with 0.15% Fe, the dark spots are beta phase due to the iron content. (Joshi (2006) and (Lutjering (2007))

However times to be detected Ti_2Fe precipitation are extremely long and, as mentioned above, this system acts as if it were an isomorphic.

Titanium alloys are classified according to the present phases at room temperature after processing: (ASM Handbook (2003))

- Unalloyed titanium or titanium commercially pure (Ti CP)
- Alpha and "Near-alpha"
- Alfa+beta

- Beta metastable and "Near-Beta"

Alpha alloys are considered those that contain only alpha stabilizing elements or neutral elements. By adding a small amount of beta stabilizing elements is obtained the "Near Alpha". Alpha+Beta alloys are most used at room temperature, having from 5 to 40% volume of beta phase. For alloys with higher percentage of beta stabilizers, martensitic transformation is suppressed and all the alloy consists of beta phase. (Leyens et al. (2003)) However some Alpha alloys containing iron in composition have small amounts of beta at room temperature. (Lutjering et al. (2007))

Titanium commercially pure (Ti CP) is characterized by having lower mechanical resistance and has greater resistance to corrosion. It can improve its mechanical strength with the addition of oxygen which significantly increases the strength and decreases the ductility. Other elements such as carbon and iron are considered impurities and appear in the manufacturing process. (Leyens et al. (2003)) However, some authors consider iron as an alloying element whose function is to control the grain growth. (Lutjering et al. (2007)) Ti CP alloy includes four grades covering a range of strength of 240-740 MPa.

Alpha and "Near Alpha" alloys have a relatively high content of alpha stabilizers and a low content of beta stabilizers. They have good resistance to high temperature creep. (Lutjering et al. (2007))

Both alpha alloys and titanium commercially pure (Ti CP) do not respond to heat treatment, because the properties of these alloys are not changed appreciably with the microstructure and there are no precipitates that may cause an increase in resistance. On the other hand "Near-Alpha" alloys have some response to heat treatments because they contain a higher content of beta stabilizing elements. (Leyens et al. (2003))

Alpha-beta alloys are characterized by a good balance between mechanical properties, corrosion resistance and workability. These alloys have the highest strength because they respond to heat treatments. However, they possess low weldability and lower corrosion resistance than the alpha alloys. (Lutjering et al. (2007))
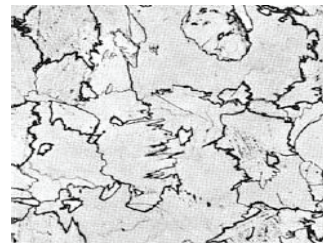
Beta metastable alloys have excellent forgeability because the beta phase has a BCC structure. Also they possess good hardenability because of its high content of beta alloying. The latter allows to develop high hardening through aging processes at significant thicknesses. (Leyens et al. (2003)) Compared with alpha-beta alloys, beta alloys have higher density, lower creep resistance and lower ductility in aged condition. However, the fracture toughness is greater. (Lutjering et al. (2007))

1.3 Microstructural development of titanium alloys

The microstructure of titanium alloys depends on thermo-mechanical parameters and chemical composition. The microstructure of titanium is mainly described by the size, shape and distribution of alpha and beta phases. The two extreme cases are laminar structure, generated by a cooling from the beta field and equiaxed structure resulting from a process of recrystallization. (Polmear (1995) and Totten et al. (2010)) Both types can be fine or coarse depending on the process parameters. The temperature and the cooling rate have a remarkable influence on the microstructure. (Donachie (2000) and Hayashi et al. (1994))
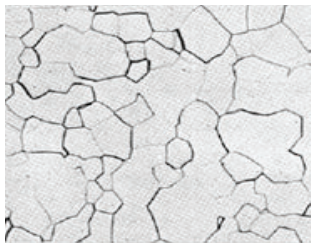
Annealed at 800°C, 1 hr.(100x)   Annealed at 1000°C, 1 hr. y water quenching (100x)

Fig. 2: Effect of temperature on the microstructure of titanium commercially pure (Ti CP). (Donachie (2000))

Ti CP is also affected by the annealing temperature and cooling rate. Figure 2 shows the variation of the microstructure due to treatments under and above the beta transus temperature. (Donachie (2000))

Although titanium commercially pure in an alpha alloy, small amounts of iron can change the microstructural evolution of the alloy. Both the microstructure and mechanical properties are the result of various parameters in each stage of the thermomechanical processing (See Figure 3).
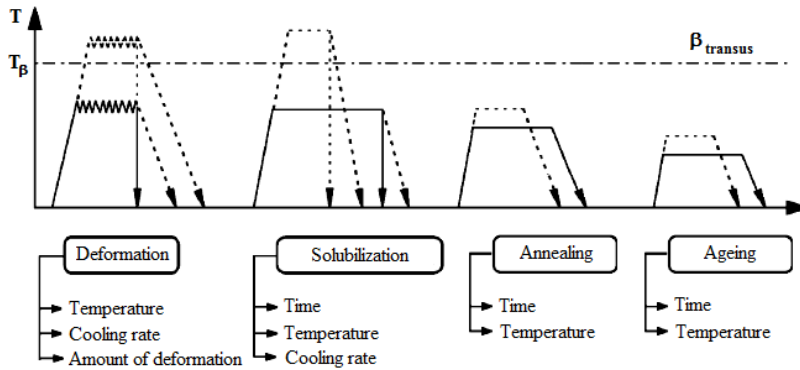
Fig. 3: Outline of process thermomechanical. (Donachie et al. (2000) and Claret (2007))

The different stages depend on the type of alloy and the properties that are desired. For example, alpha alloys do not respond to heat treatment therefore do not have solubilized and aged stage during processing. Furthermore, this type of heat treatment is common in alpha-beta and beta alloys.

2. Materials and Methods

The starting material was a rectangular specimen of Ti CP whose dimensions were 50 mm wide, 30 mm high and 120 mm long, with and without heat treatment (water quenching) (see Figure 4). Metallographic structures of these specimens were as follow: (CNEA IT-MAT-22-06 and 34-06 (2006))

Material without prior heat treatment   Material with water quenching
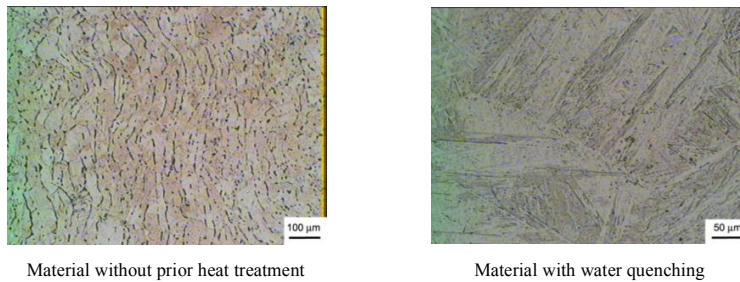
Fig. 4: Comparison between the initial structures.

In order to investigate the microstructural evolution of Ti CP (Ti Gr 4), several hot rolling routes at different temperatures were made and with two different initial conditions. The material was sampled at the beginning of the hot rolling and after each pass. Three different temperatures have been defined, one of them could work in the alpha field. The remaining two temperatures were estimated from resistivity tests and literature, allowed to work in the alpha-beta field, recommended for thermomechanical processing of Ti CP. (Donachie et al. (2000), Collins (2003) and Claret (2007))

The Table 1 shows the different working paths:

Table 1: Temperature and initial state of each hot rolling route.

Hot rolling temperature	Initial state	
	Without heat treatment	Water quenching
840°C	Route 1	Route 4
870°C	Route 2	Route 5
900°C	Route 3	Route 6

A Krupp two-high-mill was used, belonging to Mechanical Testing Laboratory, Department of Materials of the Comisión Nacional de Energía Atómica whose features were:

- Electric motor: 29 Kw.
- Roll diameter: 230 mm.
- Roll speed: 50 rpm.

The heating was performed in an electric furnace, muffle type ("Termoquar ᵀᴹ"), located in the same laboratory. The pieces were introduced into the furnace at the working temperature and held for one hour. This time was enough to homogenize the whole piece and prevent excessive oxidation. (Claret (2007) and Dieter et al. (2003))

It has been considered to apply the greatest amount of deformation per pass reducing the part thickness of 30 mm to 8 mm, except in the first rolling route where it had to make three passes. In the rest of proposed routes could be achieved applying the deformation in two passes, where the first pass was 25% to 35% reduction and with the second pass a total reduction from 60% to 75% was reached.
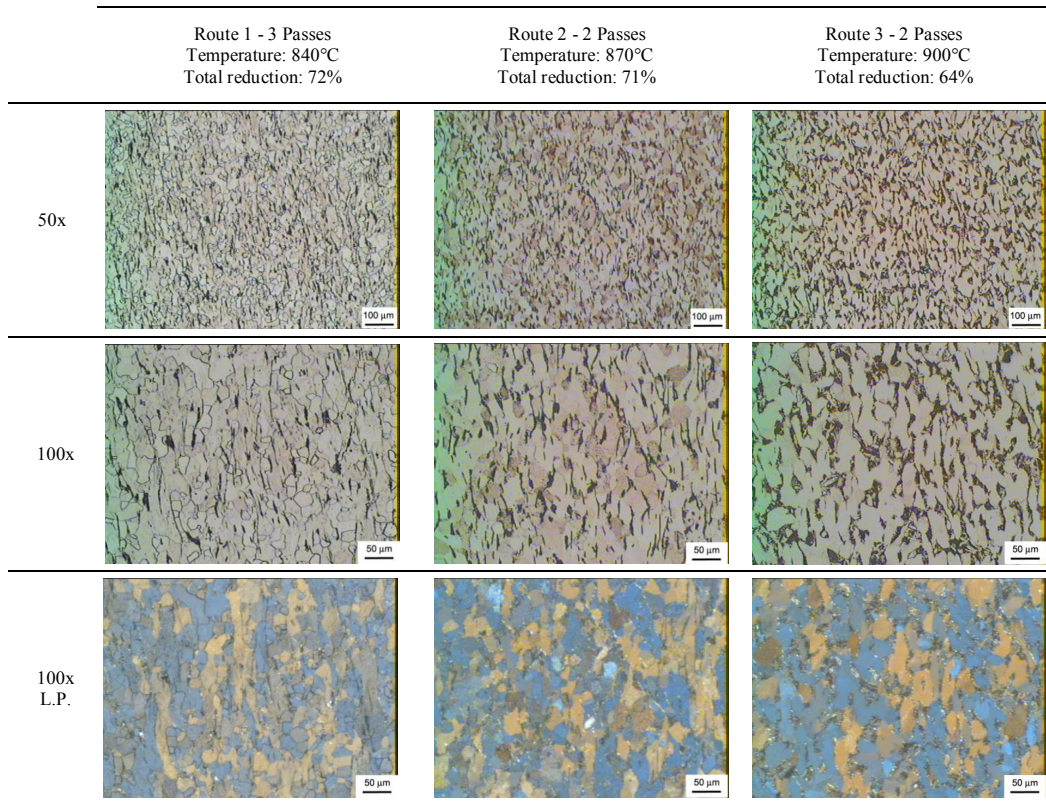
All samples were cut by mechanical sawing, then included and later subjected to a progressive grinding with sandpaper water (#80, #220, #320, #400, #600) and mirror polished with ammonium dichromate ash. The chemical reagent for the attack of the specimen was an aqueous solution of hydrogen peroxide and sulfuric acid.

3. Results and Discusion

Routes 1, 2, and 3 (samples without prior heat treatment)
Table 2 shows the structural evolution of the different routes of lamination of the material without preliminary heat treatment.

Table 2: Microstructural evolution of material without prior heat treatment (Note: P.L. polarized light).

	Route 1 - 3 Passes Temperature: 840℃ Total reduction: 72%	Route 2 - 2 Passes Temperature: 870℃ Total reduction: 71%	Route 3 - 2 Passes Temperature: 900℃ Total reduction: 64%
50x			
100x			
100x L.P.			

On Route 1, it can be seen that the grain morphology alpha becomes more equiaxed and its size decreases. Regarding the morphology of the beta phase, while its length decreases, its thickness is not altered. Consequently, its distribution is still heterogeneous.

On Route 2 a significant increase in grain size can be seen compared to Route 1. The reason for this because on Route 1, it was necessary to make three rolling passes rather than two because of the greater strength of the material, while on Route 2 there was an increase of temperature obtaining a larger grain size by static recrystallization.

It is important to remark that, at first glance, it seems to be that the fraction of beta phase increases by working at this temperature and because of the applied strains compared to the initial structure. If the estimation of the value of the alpha transus temperature by the method of the resistivity is correct, together with the increase in temperature because of the mechanical work, the material would be deforming in the biphasic field.

At these temperatures and longer times, a bimodal structure composed of primary alpha grains and transformed beta is expected to be formed. Due to the fact that the material would be at the bottom of the biphasic field at this temperature, it should take longer to obtain such bimodal microstructure
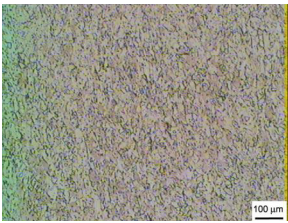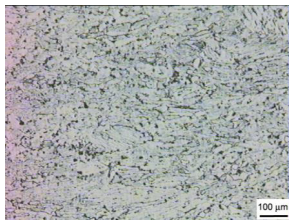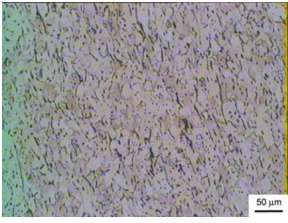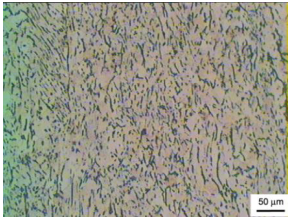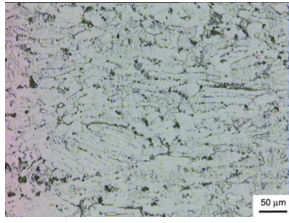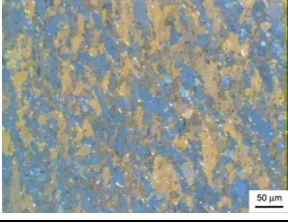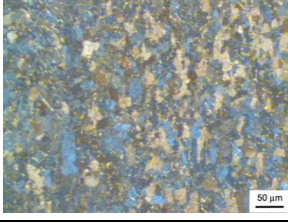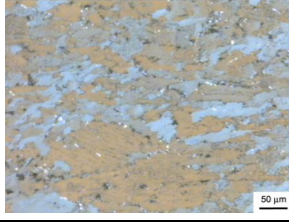
Unlike the previous, on Route 3 the preheating time working at this temperature was enough to generate a well-defined bimodal structure. This is because the working temperature is closer to the beta transus temperature. On the other hand, there was no decrease in grain size between passes.

In all cases it is noticed that both, the primary alpha phase grains and transformed beta phase, are aligned in the rolling direction.

Routes 4, 5 and 6 (samples with prior heat treatment (water quenching))

Table 3 shows the structural evolution of the different routes of hot rolling with prior heat treatment (water quenching) or solubilized.

Table 3: Microstructural evolution of material with prior heat treatment (water quenching) (Note: P.L. polarized light).

On Route 4, an equiaxed microstructure was developed in the alpha phase and the beta phase is homogeneously distributed and predominantly spherical due to a precipitation process during reheating.

On Route 5 similar structures to the ones obtained on Route 4 are reached, but the beta phase is in more elongated shape. The latter is consistent with studies that show that at temperatures below the beta transus temperature, the recrystallization is static in the case of developed strain rates in rolling process. (Dieter et al. (2003)) As in the case of Route 2 a bimodal structure is developed despite having different starting structure.

On Route 6 recrystallization is not observed. At this temperature it is likely to carry out a recovery, dynamic or static, which would reduce the required energy for recrystallization and the development of bimodal structure. This speculation would also explain the fact that on Route 3, also carried out at 900°C, the grain size is not reduced among passes.

The morphology and distribution of the beta phase is mainly affected by the cooling rate from the beta field and by the rolling temperature. This is noticeable by comparing routes 1 and 4, both held at 840°C in the alpha field.

On one side in Route 1, which starts from a laminar structure, the morphology of the beta phase does not seem to be changed by plastic deformation and appears mainly in elongated shape, although its size decreases with the plastic deformation.

On the other hand, Route 4 starts from a martensitic structure and the beta phase is presented with spherical morphology with a more homogeneous distribution. (See Figure 5)

Laminar structure (Route 1) (500 X)   Spherical morphology (Route 4) (500 X)
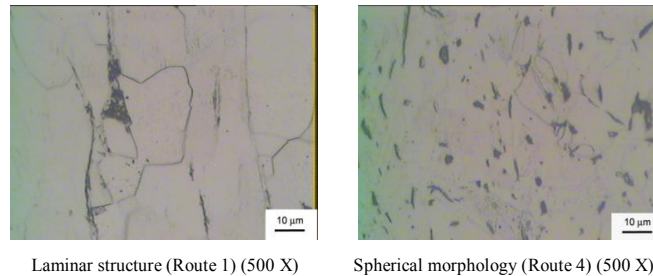
Fig. 5: Comparison between Routes 1 and 4

Regarding the working at temperatures within the range biphasic it can be said that promotes the formation of bimodal structures or growth of the laminar structure prior to working. As result of that, the distribution and morphology of the beta are very different in comparison with the starting microstructure.

The grain size and morphology of the alpha phase also depend on the working temperature and the rate at which the alloy was cooled from the beta field.

At material temperatures below the alpha transus it is observed that there is a grain size refinement. This is more noticeable on Route 1 than in Route 4 because Route 4 starts from a structure without prior deformation. On the other hand, at temperatures in the alpha-beta field it can be noticed that the grain size does not decrease between passes. When operating at temperatures in the alpha field and the starting microstructure is laminar structure, the grain morphology of alpha is affected by how the beta occurs. (see Figure 6)
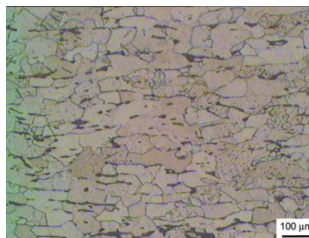

Fig. 6: Second pass Route 1 (50 X)

Instead if it is assumed a finer structure as alpha or acicular martensite, resulting from rapid cooling and working in the field alpha, beta phase is presented in a less continuous and allows the alpha phase morphology to take a more equiaxial. (see Figure 7)
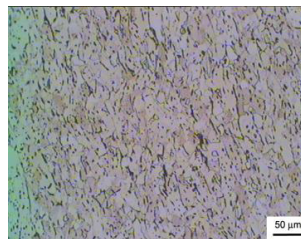

Fig. 7: Second pass Route 4 (100 X)

The microstructure of Figure 7 is similar to the structures of reference regarding the morphology and distribution of the beta phase. (Lutjering et al. (2007))

4. Conclusions

The paper shows that for hot working of titanium commercially pure, considered as an alloy of alpha microstructure, the same procedure applied in alpha-beta alloy should be adopted since the fine distribution of a second phase rich in iron depends on its corrosion resistance. And this distribution is closely related to the deformation mode.

The thermomechanical processing to obtain a microstructure like the proposed one should estimate a minimum deformation by 50%, cross section, in beta field followed by a beta quenching and finally a final deformation estimated by 75 % in alpha field. The mechanical properties are finally adjusted with additional annealing.

It would be of great interest that the present work is complemented by an evaluation of the behavior of these structures when exposed to different corrosive media through a corrosion rate, thereby letting us have a reference value in the determination of tighter parameters of the thermomechanical process. These practices will let us achieve optimum microstructures of a titanium alloy that is commercially pure in the presence of a second phase rich in iron in various corrosive media..

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