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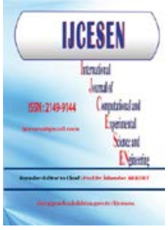

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Research Article

Microstructure and characterization of titanium alloy (Ti–6Al–4V) and pur titanium prepared by PM[#]

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Abstract: The aim of this work is to obtain surfaces with high content of beta enhancing titanium alloy by diffusion process at settled temperatures and times. Two metal alloys are selected, Niobium (Nb) and Molybdenum (Mo). In separate experiments, they are diffused over titanium alloy (Ti–6Al–4V) and over pure titanium (Ti) by using Powder Metallurgy. For this study different experiments were performed, Mo and Nb were diffused in Ti and Ti-64 samples at 1100 and 1200 ºC for 3 and 4 h, respectives. Their microstructure and some mechanical properties such as microhardness were investigated. The experiment succeeds in changing the microstructural properties of the surface of Ti and Ti–6Al–4V samples as characterized by opticla microscopy, SEM and XRD diffraction to confirm the phases obtained, beta and alpha plus beta structures are obtained. In particular, by using diffusion process at 1100 ºC for 3 h. Moreover, the experiment were simulated using DICTRA, these results were compared to the real results of the experiment. DICTRA results were reliable because they were similar to real experiment results.

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1. Introduction

Titanium and titanium alloy have involved more attention than other metallic materials with affection due to its capability, performance and protection [1, 2]. In adding its superior corrosion resistance and exceptional raised temperature performance. One of the most important properties of titanium alloy is his lowerstiffness and rigidity compared to other metallic olloys. Titanium and titanium alloy have been used in structural applications for load-bearing sandwich cores in the aerospace and transportation industries and in many other applications.

However, despite these good properties, titanium is used frequently in many applications due to its high raw material and industrial costs [3-4]. Ti-6Al-4V alloy is the most widely used titanium alloy because of their good machinability, high strength, excellent corrosion resistance, and mechanical properties. Thus, Ti-6Al-4V alloy deals the best performance for a variety of weight reduction applications in aerospace, automotive and biomechanical applications. However, holding to their poor wear resistance, their potential applications in relieving engineering tribological components are limited. Hence, the properties of alloy can be developed using the appropriate surface treatment techniques such as ion implantation and low energy high current pulsated electron-beam [5]. The wear resistance of the Ti-6Al-4V alloy can also be improved by producing composite structure using second phase particles.

Powder metallurgy techniques meaningfully donate to the development of effective operating implants. It is found on the limited densification during sintering of metal powders [3]. The process begins by mixing the metal powders with a space holder, followed by the powder mixture uniaxial or isostatic compaction to form a green sample. The sample is subjected to a heat treatment process to eliminate the space holder. Sintering stage at developed temperatures grows sinter neck formation and growth, resulting into densification of the structure and development of structural integrity [1, 5]. This process based on mechanical alloying of elemental powders tracked by hot association have emerged. In addition, this method is also very effective to prepare a complex alloy in various alloying elements.

However, in this study an attempt has been made to produce fine-grained titanium alloys via a powder technic based on mechanical alloying of powder mixtures followed by Mo/Nb powders. The microstructural of titanium and titanium alloy is exhibitedand discussed. The effect of temperature and time on the microstructure and properties have been also evaluated and the results related to this aspect are presented and discussed.

2. Experimental

In the present study, pure titanium and Ti-6Al–4V alloys were used as initial material. Titanium alloy has a chemical composition of 6% aluminum, 4% vanadium, 0.2% oxygen, and the remainder titanium. Chemical composition of Titanium alloy is shown in table 1.

Table 1. *Chemical composition of Titanium*

elements	Al	V	O	Ti
(wt.%)	6	4	0. 2	Balance

One of the most important properties of titanium is its modulus of elasticity. For the Ti α phase at room temperature, this value is about 115 Gpa, when the temperature increases it decreases linearly to 58 GPa at a temperature close allotropic transformation values [8, 9].

The microstructural characterization of titanium samples were observed by a Leo 1430 VP scanning electron microscopy (SEM) and optical microscopy (Optika B-600). The thicknesse measured by an optical microscopy are averages of at least 12 measurements.

The presence phase analysis was carried out at ambient temperature by Philips X-ray diffraction (XRD) analysis with a Cu Kα (λ= 1.5406 Å) source at 40 kV voltage and 30 mA current ranging from 20 to 90°.

Mechanical properties of the sintered compacts were evaluated by micro hardness measurements at ambient temperature. Vickers micro-hardness was estimated by indentation under load of 4.903 N (HV0.5) and dwelling time of 15 s. The indentation during hardness test was carried out randomly, wherein the distance between the indents was kept at least three times the size of the indent. The representative hardness value of the test specimen was an average of 20 indentations.

3. Results and discussion

XRD analysis was carried out toidentify the phases that compose the alloys. The XRD pattern representative of both the prealloyed Ti–6Al–4V powder and pure titanium blend is shown in Figure 1.

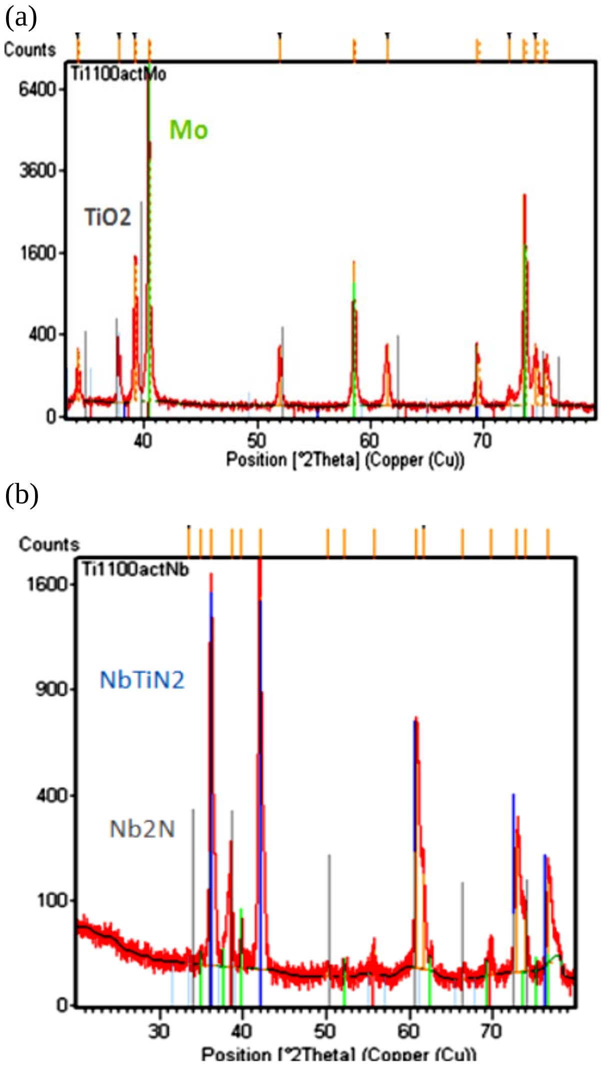

Figure 1. *XRD analysis at 1100°C for 4 h.*
(a) of Ti/Mo (b) of Ti/Nb

The main phase found during the XRD analysis of the powders is the alpha phase, although some beta phase and some aluminium/vanadium phase, indicating that the composition of the prealloyed Ti–6Al–4V powder is not completely homogeneous.

The microstructural evolution of the prealloyed Ti–6Al–4V powder during vacuum sintering over the range 1100-1200 °C. The microstructural analysis of the Ti–6Al–4V prealloyed samples sintered at 1100 °C shows that the sintering of the powder particles was already initiated, in agreement with the results of the dilatometric study were observed to disappear and the residual porosity was mostly irregular in shape. The microstructural analysis also indicates that the main microconstituent is the alpha phase.

Beyond this temperature reaches the value of the beta phase with not only good mechanical properties, but also a good corrosion resistance. The inability to have β phase of pure titanium at room or elevated temperature is not used too much titanium alloys with different elements for stabilizing the β phase. This is where the importance of unalloyed titanium, it can change its properties to suit a particular function and find states coexisting in α and β titanium phase. Titanium alloys are classified by the effect of the titanium alloy, if α phase supports, β, or maintained. This can be seen that α phase stabilizers as Al, N, O, or C phases, which can be seen from the phases diagrams that increasing the nconcentration of these elements in the temperature at which the β phase is greater than 882 °C [10, 11].

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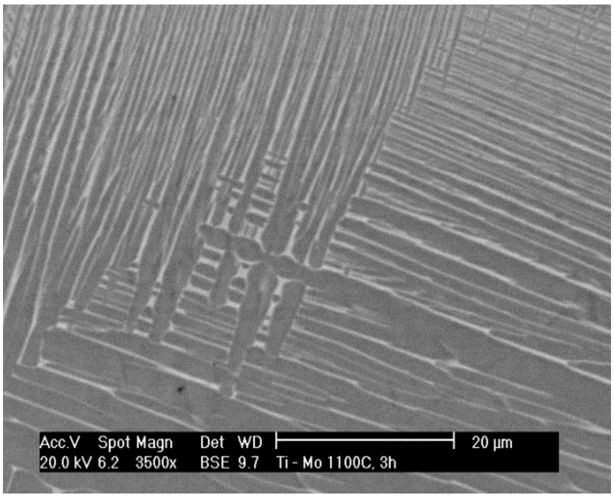

Figure 2. *Microstructural evolution of the prealloyed Ti–6Al–4V/Nb powder sintered under high vacuum at 1200 °C for 4h.*

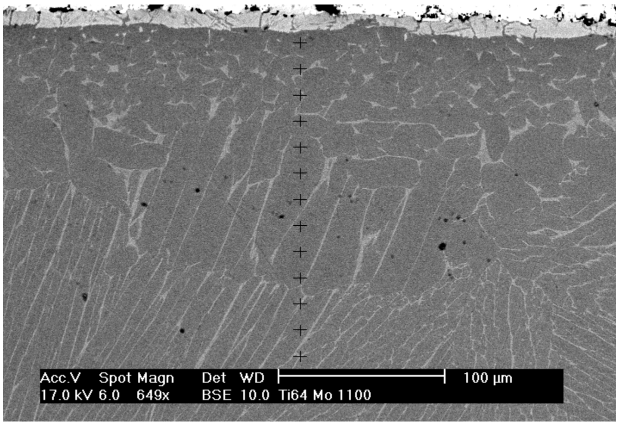

Figure 3. *Microstructural evolution of the prealloyed Ti–6Al–4V/Mo powder sintered under high vacuum at 1100 °C for 4h.*

Titanium alloys used are constituted of more than one phase diagrams ternary and quaternary alloy used instead. The titanium α phase is widely used for parts in contact with corrosive or oxidizing media, while α + β phase of the alloy known for its resistance.

The results of the EDS analysis of the two phases thatconstitute the microstructure of the Ti–6Al–4V alloy resemble those of the compositional analysis, where thevariation in the percentage of the alloying elements is due to the limited area of the zones analysed. EDS analysis of the zone where the diffusionof the alloying elements takes place in the Ti–6Al–4V alloy and pur titanium powderblend specimens during sintering at 1100 °C are similar tothose of the Ti–6Al–4V powder used to obtain the blend becausealuminium diffuses more quickly than titanium, and vanadiumis present only in isolated zones composed of lamellae. The microstructural evolution of the elementallyblended Ti–6Al–4V powder blend during vacuum treatment between 1100 °C and 1200 °C. The necking between the powder particles isinitiated at a sintering temperature of 1100 °C and XRD pattern shows that is representative of thesintered samples, particularly those processed at 1100 °C and 1200 °C.

The initial microstructure of the titanium surface is modified becoming lamellar α + β. Therefore, all the other experiments are simulated in the same manner. Besides in some areas of the part the different directions laminar growth, the meeting of two colonies of α + β formed by diffusion is shown in Figure 2.

The phases detected in the specimens at 1100 °C are titanium alpha and titanium beta which had already been detected in the starting powder. The XRD pattern of the specimens at 1100 °C confirms the results of the microstructural evolution and of the EDS analysis, indicating that the distribution of the alloyingelements is homogeneous because no Al_2V_3 peaks werefound. No significant difference can be observed in the XRD patterns of the specimens sintered at 1200 °C incomparison to those of the samples processed at 1100 °C. In contrast to α titanium properties are due to the orientation of the stress with respect to the workpiece due to the nature of the α structure. Ti alloys with Al as used in this experiment reproduces this effect is because aluminum acts by decreasing the unit cell parameters. The main forms of hard titanium add interstitial elements such as oxygen or nitrogen or the addition of substitution elements such as Al or Z. In the α + β alloys is three types of microstructure: kelp, equiaxed or bimodal containing equiaxed α structure in a matrix of α + β phase.

Figurie 3 shows the cross section of a Ti-6Al-4V sample of in which Mo has spread. At the top of the piece, molybdenum has spread by creating a layer of Ti in β phase, this was the goal of the project. Immediately this an area of phase α + β equiaxial, this is not the original Ti-6Al-4V structure since it has structure laminating. Finally the farthest part area maintains the structure laminating, it shows image of the porous titanium sample, titanium sample processed with space holder displays an interconnected porosity type with few closed pores and a higher pore volume fraction than the sample without space holder.

The properties of titanium with respect to α + β phase, α titanium mainly depends on their composition and then treating mechanism. In contrast to α titanium properties are due to the orientation of the stress with respect to the work piece due to the nature of the α structure. Ti alloys with Al, as used in this experiment reproduces this effect is because the aluminum works by reducing the unit cell parameters.

The main forms of hard titanium add interstitial elements such as oxygen or nitrogen or the addition of substitution elements such as Al and Zr. The α titanium alloys are among the most resistant structural materials than working in a corrosive environment. Especially in an oxidizing environment and weather resistant layer is created, if the room is in reducing environments then this layer is broken if you need to add inhibitors such as palladium. Due to its high performance in many corrosive titanium desalination for ships. As the part is used in heat exchangers, condensers, plants in many cases, in addition to being a corrosive liquid is exposed is at a high temperature that makes it should consider the high temperature corrosion and crevice corrosion. In addition of a little Mo is known to crevice corrosion resistance to corrosion and high temperature increases. However, the molybdenum phase for the α phase can not add more than 1% molybdenum, for if one enters the α + β phase.

In the α + β alloys is three types of microstructure: kelp, equiaxed or bimodal containing and equiaxed α structure in a matrix of α + β phase. For such structures, simply by performing an annealing treatment of the alloy at the end of treatment.

The most important process parameter is the ratio of cooling crystallization process, which will be determine by the characteristics of the lamellar structure and size of α phase, the thickness of layers in the edge or edges β and α zone colonized by the structure α. All these features reduce the cooling rate increases. Although it is also possible to obtain these structures without recrystallization of the material is not a very common form in reality. In the important of the annealing process is no longer the temperature time. A small area colonized by α phase is less effective slip planes causing an increase in the creep strength.

For the formation of these structures further processing is performed at a temperature lower than the temperature of β transits temperature of the α + β sample .In this case, what determines the thickness of the lamellar phase, α is the coolant report from homogenization. These alloys have better mechanical properties than the fully lamellar alloys with similar cooling ratios, except as regards the behavior or propagation of fatigue cracks, in a small area bimodal alpha colonized greater fatigue resistance as shown in Figure 3.

The upper part of the brightest sample of the coating layer is composed of Mo and Cu. In the contact between copper and the porosity sample exists. On the surface of the workpiece is a region where the α and β lamellar titanium coexistence phase, the latter being perpendicular to the direction of the sample surface.

In addition, in some areas of the laminar piece growth was different directions, the meeting of two phases α + β formed colonies presented by diffusion. Pure titanium powder has very high tendency to conglomerate and staff with the vial and balls during mechanical alloying.

The mechanical properties in this case is essentially influenced by the size of titanium grains in α phase over the border size have high creep. These analyzes the shape of α + β alloy is the theory, will later be applied to our special experience. For these small grain structures during the homogenization must have a strong cooling rate in the process.

The main feature of this type of alloy are its mechanical properties and having a high resistance in the tensile test, fatigue and fracture. The main characteristic of beta type alloys is that you can tighten to much higher values than α + β alloys can be worked a little cold. The process of obtaining this type of alloy is α + β equal to the alloy.

To achieve these alloys at temperatures below the temperature of β phase such alloys are used. To create these alloys must be used betagene elements such as Mo, Nb, V, Ni, Cu or Ta.

To improve the properties of these alloys are used to precipitate fine particles of Ti-α in the sample instead of being Ti β only. The ductility of the alloy is greater than β phase. Young's modulus of titanium alloy decreases, this activity is important in biomaterials because of the difference in Young's modulus of the bone.

SEM was obtained to measure the depth of diffusion zone, it was about 60 µm, therefore we can say that the previous calculations correspond to reality. It is due represents the percentage by weight of Mo and Ti with respect to the distance to the sample surface. The depth at which there is relevant percentages Mo about 60 µm.

The addition of activator affects diffusion. In the high surface areas are created and Mo as the distance to the surface decreases the size of these areas increased. One can appreciate that some porosity is because the chlorine has attacked activator part, due to titanium chlorides that form as predicted by thermodynamic calculations.

When the activator is added to the percentage of Mo near the surface of the sample is greater than when it is added but not turn the distance penetrated in the workpiece is lower. Then there one α + β laminar zone where the Nb concentration is decreasing.

Note that the presence of N in the sample, as in previous cases we have to use the activator NaCl. It was attached to the coating in small areas Ti β phase due to the high concentration of Nb DICTRA as predicted in both concentration and diffused away.

The size of the sample surface is increased. Therefore, without a preferred direction has managed to create a phase α + β laminar. Figure 3 shows the electro disposition of titanium with Nb diffusion to 1100 °C for 3 h. By adding N activator experiments in 1100 in contrast no oxide layer, there is a large area with high content of Nb. In this area there Ti in the β phase is the objective of the experiment, at the bottom of this layer is a layer with some of a large N. The analysis of the sample surface shown by XRD the surface is composed of the compounds $NbTiN_2$ and Nb_2N. This result is consistent with the results of the SEM.

Figure 3 shows the cross section of a sample of Ti-64, which has spread Mo. At the top of the piece-disseminated molybdenum creating a layer of Ti in β phase, this was the goal of the project. Immediately this area α + β phase equiaxed, this is not the original structure of Ti-64 since it has laminar structure. Finally the farthest part area holds the laminate structure.

By employing as Ti-64 substrate it has a surface layer rich in Nb, over a layer of oxide with a content of Cu coating. There is a Ti layer with α + β due to the presence of V in the lower part of the oxide layer. The spread of V creates a layer of Ti-α.

The activator in experiments at 1100 °C creates a porous layer due to the attack of Cl Ti as expected due to thermodynamic calculations. activator is more effective in the experiences of Nb and Mo in the gaseous species formed Nb are more likely than those formed of Mo, this experience was calculated and was corroborated. The simulation experiment and approach to the dissemination of Mo and Nb.

Therefore, it has been ratified that DICTRA as a program adapted to simulate the spread if the experiment proper discharge of the defined objective. The diffusion of Mo and Nb in titanium and titanium alloy their original microstructure are changed on the surface becoming α + β phase. DICTRA simulation using the Mo diffusion experiment is not very accurate by the model used, while the model used for calculating Nb itself which corresponds to reality.

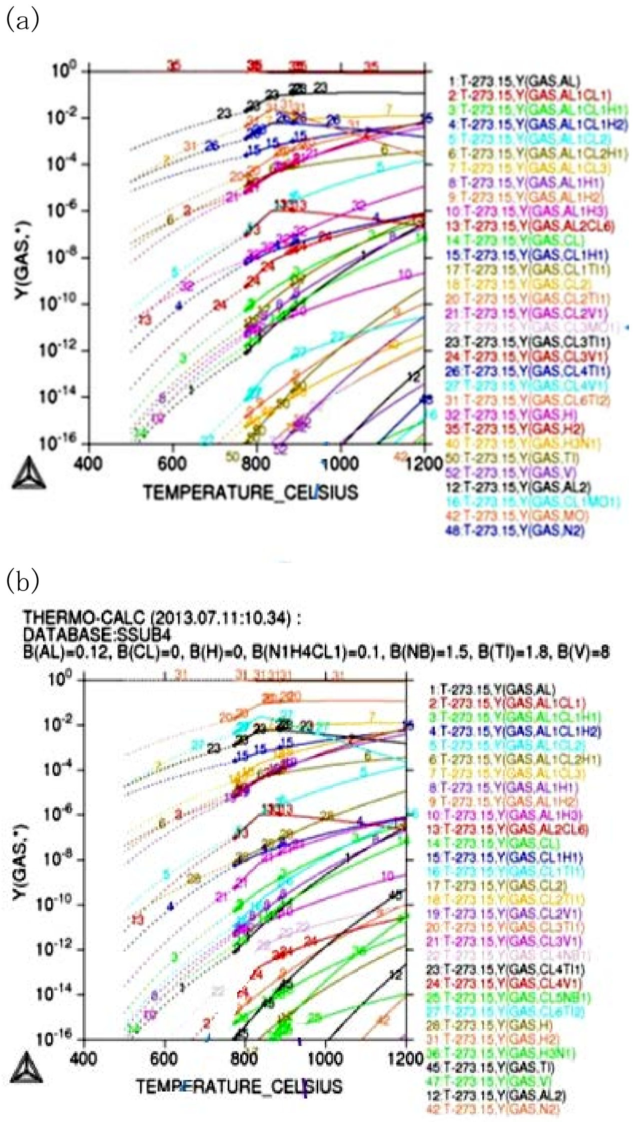

Figure 4. *probable gaseous species in experiments at 1100 °C for 4h. (a) for Mo (b) for Nb.*

Hence, the remaining experiments are simulated in the same manner.

It is achieved create a β phase layer on the surface of the samples. In addition, the microstructure of the sample is changed α + β laminar to α + β equiaxed. The simulation experiment and disseminating coming Mo and Nb as shown in Figure 4.

The activator in experiments to 1100 °C creates a porous layer due to the attack of NaCl.

The activator is effective in experiments in Nb and Mo as the gaseous species, it us formed of Nb more than it is formed of Mo, this resulat was calculated and experiments corroborated it with DICTRA predicted that the Nb would not spread in the experiments at 1100 ° C and dissemination not predicted or using the activator. Instead DICTRA if predicted 30 µm to spread in spreading Mo, but the reality was different because it does not disseminated in any experiment.

Therefore it has been ratified DICTRA as an appropriate program to simulate spread if the experiment is properly defined fulfilling the target.

3. Conclusion

The following conclusions can be obtained from the results of characterisation of the prealloyed Ti–6Al–4V and elementally blended powders and their microstructural evolution:

– The titanium powder metallurgy industry needs morereliable powders that can be usedto obtain final products or that can be employed as masteralloys to add the alloying elements desired.

– By using a prealloyed powder, the interaction of elementalTitanium with elemental aluminium, which generatesintermetallic $TiAl_3$, is avoided.

– The conventional powder metallurgy route of pressing andsintering can be exploited for the fabrication of titaniumproducts starting from irregular prealloyed powder orelementally blended powder using a master alloy.

– Various relative densities, between 83% and 95%, porestructures and grain sizes can be obtained by selecting aproper sintering temperature, but when the alloying elements have to diffuse to homogenise the composition, aminimum temperature of 1200 °C should be selected.

– Higher flexural strength and hardness in comparison tothe values specified for biomedical devices obtained bymeans of conventional metallurgy are obtained.

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