

# DUAL PHASE $\alpha + \beta$ FORMED IN Ti–6Al–4V TITANIUM ALLOY AND ITS MECHANICAL CHARACTERIZATION

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**Abstract:** In this study, the formation of dual phase  $\alpha + \beta$  in Ti–6Al–4V titanium alloy has been described. The microstructure of  $\alpha + \beta$  dual phase was achieved via a heat treatment. Scanning electron microscopy and X–ray diffraction amounts were achieved to reveal the microstructural evolution and to confirm the phase constitution during the decomposition. The detailed microstructure of  $\alpha + \beta$  phase was observed using transmission electron microscopy. The microstructural features included irregular shaped variants, dense and fine twinning with width about 25  $\mu\text{m}$ . The microstructure of  $\alpha + \beta$  dual phase inbred the characteristics of the microstructure of  $\alpha$  phase. Moreover, only a specific variant combination of the decomposed  $\alpha$  and  $\beta$  phases was observed. Crystallographic orientation of the decomposed  $\alpha + \beta$  phase was similar to that of the initial  $\beta$  phase. These results indicated the existence of a strict variant selection rule between  $\alpha$  and  $\beta$  phases.

**Keywords:** Microstructure, Ti–6Al–4V titanium alloy, mechanical characterization, X–ray diffraction

## INTRODUCTION

The formation of microstructures from the high temperature  $\beta$  domain is studied for various titanium alloys. Phase transformation diagrams have been established for various titanium alloys ( $\alpha + \beta$  or metastable  $\beta$ ) by electrical resistivity and by synchrotron XRD. For titanium alloys of the  $\alpha + \beta$  or metastable  $\beta$ , depending on the transformation temperatures and the driving force of transformation, various germination and growth mechanisms are involved. At low driving forces of transformation, the germination of the phase is heterogeneous and occurs at grain boundaries followed by wetting along the joint. Lateral growth occurs followed by the formation of  $\alpha$  lamella colonies with the same crystallographic orientation as the grain boundary from which they originated. The growth kinetics are strongly controlled by the partition of the solutes between parent phases. The change in the mean composition of the parent phase is also illustrated by the changes in chemical composition characterized by synchrotron XRD during studies under isothermal transformation condition. When the driving force increases, the germination of  $\alpha$  grains, with spatial arrangements evolving from a few parallel platelets to individual platelets characteristic of the different  $\alpha$  variants in the same  $\beta$  grain. The characteristic sizes of colonies such as intra granular platelets are a function of the transformation temperature; their thickness decreases when the driving force of transformation increases and their number increases [1–2]. For lower temperatures (<500 °C) metastable phases are formed  $\alpha'$  and  $\alpha''$ .

The characterization by XRD synchrotron radiation leading to the lattice parameters phases shows the low partition of the alloying elements. At the lowest temperatures studied,  $\alpha''$  phase has the same lattice parameters as a martensitic phase formed under stress ( $\beta$  metastable alloys). The XRD characterizations confirmed that the transformation sequence is the formation of  $\alpha''$  (then when the temperature increases (as the diffusion of the solutes is more favourable)

the  $\alpha''$  phase evolves towards  $\alpha$ . The microstructures of these alloys can also be formed during tempering, from the metastable  $\beta$  phase. It is clearly the influence of the heating rate on the microstructures after aging. In fact, the precipitation sequence is a function of this heating rate, with very slow heating rates forming the  $\alpha''$  then  $\alpha$ , or  $\alpha''$  then  $\alpha'$ . These metastable phases do not precipitate for very fast speeds [3–5]. The  $\alpha''$  martensitic phase is formed via either the cooling of the parent  $\beta$  phase below the martensitic transformation start temperature ( $M_s$ ) or the application of stress greater than that required for the production of martensitic.

In other hand, titanium (Ti) alloys are considered as the next generation biomedical alloys due to their good biocompatibility. However, these alloys need to improve some their mechanical properties. At for example, high yield strength to safeguard the load–bearing orthopaedic implants against sudden impact. The transformation from the  $\beta$  to  $\alpha''$  phase via heat treatment is accompanied by atomic diffusion, arises via two mechanisms. The first mechanism, hereafter mentioned to as mechanism 1, includes the aging induced decomposition of the  $\beta$  phase [6–7]. This results in the formation of the  $\alpha''$  phase via either the martensitic transformation during aging or the subsequent cooling after aging. The formation of the  $\alpha''$  phase via this mechanism is accompanied by the formation of the  $\beta$  phase with a high content of  $\beta$  stabilizers. The second mechanism, hereafter referred to as mechanism 2, involves the disappearance of the local barriers for  $\alpha''$  formation. The disappearance of the barriers is induced by the short–range atomic rearrangement during aging.

The microstructural formed  $\alpha''$  phase has attracted significant attention. In the literature, it is reported that the crystal structure of isothermally formed  $\alpha''$  continuously changes from  $\beta$  (bcc) to  $\alpha$  (hcp) during heating [8] as shown if Fig. 1. The allotropic transformation  $\beta \rightarrow \alpha$  ( $cc \leftrightarrow hc$ ) is a martensitic type transformation. In titanium alloys, this transformation

occurs by a shear mechanism with athermal germination, possibly followed, in the case of slow cooling (around 2°C/min), thermally activated growth. From the crystallographic point of view, the transformation of the  $\beta$  phase to the  $\alpha$  phase can be described by the displacement of atoms in a plane followed by a rearrangement of them perpendicular to the plane: this mechanism is characteristic of reactions without diffusion say displacives. The concentration of beta genic elements is sufficient for the  $\beta$  phase, or metastable at room temperature. This alloy class offers the advantage of power generate a large number of microstructures on condition of mastering the mechanisms fundamentals related to the decomposition of the  $\beta$ -metastable phase.

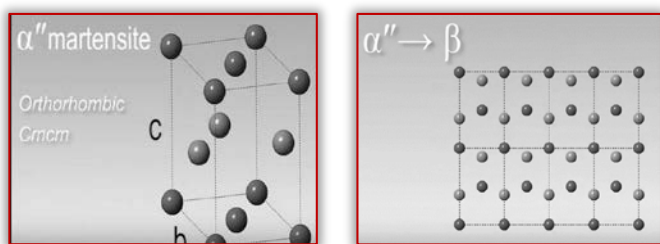


Figure 1. Allotropic transformation  $\beta \rightarrow \alpha$  ( $cc \leftrightarrow hc$ )

The microstructure and morphology of the  $\alpha + \beta$  dual phases are strongly dependent on the mechanism of formation and the crystallographic conditions between the  $\alpha$  and  $\beta$  phases. The microstructure of the  $\alpha + \beta$  dual phases that are formed via the precipitation of the  $\alpha$  phase from the supersaturated  $\beta$  phase has been observed in various studies [9, 10]. However, there are limited reports on the microstructure of the  $\alpha + \beta$  phases that are formed via the decomposition of the  $\alpha''$  phase, the  $\alpha''$  phase is formed via martensitic transformation or isothermal aging.

In this study, we try to confirm that good mechanical properties could be achieved by formation of equiaxed fine-grained  $\alpha$  phase embedded in  $\beta$ -matrix. A good combination of high strength and low Young's modulus is necessary for the design and fabrication of biomedical titanium alloys. The response of the mechanical behavior to the microstructural evolution in the alloy was investigated.

The objective of the present study was to explicate the microstructure of the  $\alpha + \beta$  phases that were formed via decomposition (i.e.,  $\beta \rightarrow \alpha'' \rightarrow \alpha + \beta$ ). The crystal structure of the Ti-6Al-4V alloy was investigated using Scanning Electron Microscopy (SEM) and X-ray diffraction (XRD). The microstructures were observing using scanning electron microscopy (SEM). The  $\alpha + \beta$  phases exhibited a unique microstructure comprising fine lamellae, and the existence of a strict variant selection rule between the decomposed  $\alpha$  and  $\beta$  phases was observed. The microstructure of the  $\alpha + \beta$  phases that were formed via the decomposition was subjected to crystallographic analysis and discussed based on the results of the systematic microstructural observations.

## MATERIALS AND METHODS

This study mainly focused on Ti-6Al-4V titanium alloy. In this alloy, the  $\beta$  phase is metastable at room temperature, and

heat treatments suitable make it possible to generate a large number of microstructures on condition to master the fundamental mechanisms linked to the decomposition of the  $\beta$  phase. In order to study the micro-mechanisms responsible for the mechanical properties of this alloy as well as their microstructure. We used different techniques. The titanium Ti-6Al-4V alloy has the theoretical nominal mass composition of 5% aluminum, 4% vanadium, 0.35% Iron, 0.16 oxygen, 0.03 nitrogen and 90.46 % titanium. However, there is a slight dispersion in the composition of the alloy due to the presence of traces of impurities and the exact composition of the alloy is presented in the Table 1.

Table 1. Chemical composition of titanium alloy Ti-6Al-4V

Element	aluminum	vanadium	iron	oxygen	nitrogen	titanium
Weight w.t.%	5	4	0.35	0.16	0.03	90.46

The specimens are deformed post mortem, during in situ tests under SEM. The samples used during the in situ deformation were taken from the head of the deformation. The samples that were used in the manufacture of thin sections observable under a microscope. An electrochemical polishing is then carried out using an electrolytic thinner.

The phase constitution was determined using XRD at room temperature using Cu K $\alpha$  radiation with Si powder as the reference material. The step size and scanning speed of XRD measurement in this study were 0.0084° and 30 s/step, respectively. The microstructure was observed at room temperature using SEM (TUSCAN) in conjunction with energy-dispersive X-ray spectroscopy (EDS).

The microstructure of the alloy is composed of a centered cubic  $\beta$  matrix, in which both precipitate  $\alpha$  nodules or platelets but also the thin  $\alpha$  phase lamellae. Solution treatments in  $\alpha / \beta$  domain lead to bimodal structures made up of primary  $\alpha$  grain in a  $\beta$ - matrix transformed. The temperature is kept at a lower temperature from 1200 °C, the pre-existing  $\alpha$  platelets or nodules grow larger. On the other hand, the very fine ones as coverslips are put back into solution. It is during the subsequent quenching that precipitate new thin lamellae of  $\alpha$  phases, the nodules or platelets of  $\alpha$  phase remaining unchanged during this cooling. Income raising the temperature and keeping it at a much lower temperature  $\beta$  then make it possible to grow the lamellae of phase that precipitated during the quenching after dissolving.

## RESULTS AND DISCUSSION

The phase constitution of the sample treated Ti-6Al-4V titanium alloy was investigated and presented in Figure 2. The  $\alpha$  and  $\beta$  phases were detected via the XRD in the sample that was aged. The XRD measurements detected the presence of a low-intensity peak of the  $\alpha$  phase.

The  $\alpha$  phase remained because the resolution treatment time was not enough to obtain the single  $\beta$  phase. However, most of the  $\alpha$  phase was resolved, and

the resolution treatment induced a reverse transformation to the  $\beta$  phase.

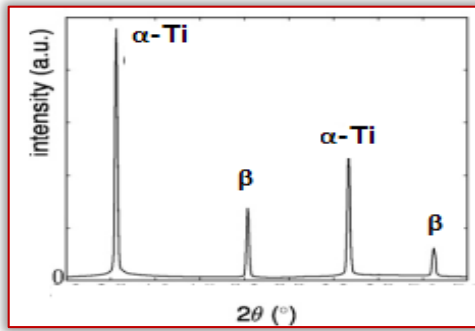


Figure 2. XRD profiles obtained at room temperature

The  $\beta$  phase in the sample directly transformed to the  $\alpha''$  phase via aging at 600°C. Furthermore, the  $\alpha''$  single phase was obtained before the decomposition. Thus indicating that macroscopic phase separation was not necessary for the formation of the  $\alpha''$  phase in the titanium alloy. This result was similar to that in the previous studies in the literature [11–13]. Each peak of the  $\alpha''$  phase shifted with the increase in the aging time. The crystal structure of the  $\alpha''$  phase approached that of the  $\alpha$  phase, and the peak shift corresponded to the changes in the crystal structure of the  $\alpha''$  phase. A heating obtain the  $\alpha + \beta$  dual phase via the decomposition of the  $\alpha$  phase was determined.

Titanium alloy has a relatively heterogeneous microstructure made up of colonies of  $\alpha$  phase, this phase has precipitated in the centred cubic  $\beta$  matrix. Thus, in the microstructure of this alloy, the  $\alpha$  phase precipitates in colonies. The  $\alpha$  phase precipitates in the form of platelets and the  $\alpha$  phase in the form of fine lamellae as shown in Figure 3. The microstructure of the decomposed  $\alpha + \beta$  phases was observed. The sample for SEM is shown in Figure 3. Thus, the sample comprising the decomposed  $\alpha + \beta$  phases was resolution treated.

The micrograph presented shows the presence around the  $\alpha$  nodules an area measuring between 20 and 25  $\mu\text{m}$  thick, made up of  $\beta$  matrix and in which no  $\alpha$  lamella phase precipitates. The existence of this zone can be attributed to the difference in stoichiometry between the  $\alpha$  and  $\beta$  phases of the alloy. During the precipitation of nodule, the alphasgenic elements are absorbed by the hexagonal phase nodule, and therefore pumped from the  $\beta$  matrix.

The precipitation of the  $\alpha$  lamellae in this depletion zone during the heat treatment of income becomes difficult, even because of the greater scarcity of alphasgenic elements in the immediate vicinity of the nodule. However, it could not be demonstrated directly with the experimental means. In particular, the EDX was not conclusive because it presented measurement uncertainty well beyond the expected local variations in composition. Relationships of

orientation observed are equivalent to those determined above for the titanium alloy.

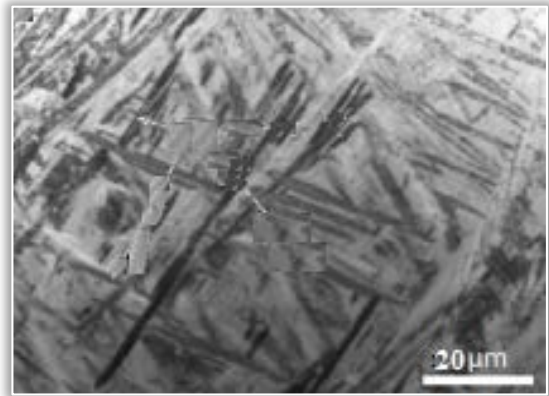


Figure 3. Lamellar microstructure of sample x 1000

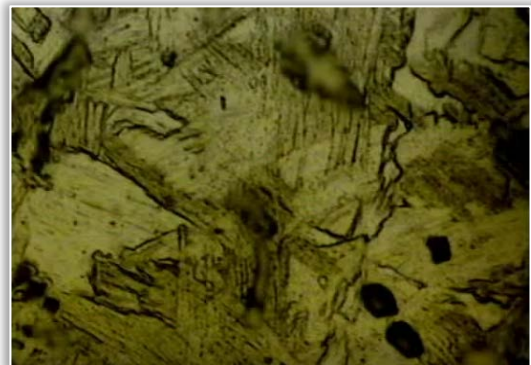
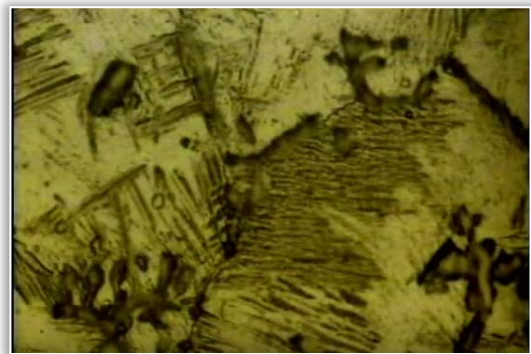


Figure 4. Results of the SEM that were obtained from the same sample and region

## CONCLUSIONS

The microstructure of the  $\alpha + \beta$  phases was evaluated in this study. The obtained results were summarized as follows.

- The microstructure of the  $\alpha + \beta$  phases inherited the characteristics of the microstructure, it was achieved via a heat treatment.
- Scanning electron microscopy and X-ray diffraction amounts were achieved to reveal the microstructural evolution and to confirm the phase constitution during the decomposition.
- The detailed microstructure of  $\alpha + \beta$  phase was observed using transmission electron microscopy and he microstructural features included irregular shaped variants, dense and fine twinning with width about 25  $\mu\text{m}$ .
- Only a specific variant combination of the decomposed  $\alpha$

and  $\beta$  phases. The crystallographic orientation of the decomposed  $\beta$  phase was similar to that of the initial  $\beta$  phase. These results confirmed the existence of a strict variant selection rule between the  $\alpha$  and  $\beta$  phases, and this rule originated from the  $\alpha''$  variant that was formed before the decomposition. The  $\alpha''$  variant determined the parallel plane and parallel direction between the  $\alpha$  and  $\beta$  phases.

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