JOURNAL OF OPTOELECTRONICS AND ADVANCED MATERIALS Vol. 17, No. 9-10, September – October 2015, p. 1410 - 1416

Studies on structural changes in titanium alloys by heat treatment

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For the studies it has been chosen Ti6Al4V alloy; evidences of this material have undergone several heat treatment operations (afterglow, hardening, abatement). After obtaining these samples we have studied the obtained structures, the hardness and the shock resistance. The structures were analyzed at magnification between 100 and 1000 times, using both metallurgical aspect and equilibrium and heat treatment diagrams. It has been found that the parameters of the heat treatment have a decisive influence on the structural composition as well as in terms of the form, the size of crystal phases and mutual distribution of phases. It has been determined that at the same time the values of mechanical properties varied in a timely manner; thus the hardness changed from the initial value of about 20 HRC to 42…44 HRC. The resilience tests emphasized that the modification of heat treatment parameters (hardening treatment, cooling medium, abatement time) the resilience values KCU could be adjusted according to the needs. The trials carried out prove that the titanium-based alloy Ti6Al4V is appropriate to a wide range of heat treatment operations: these may have as a purpose the modification in relative distant limits the properties and structural layout. At the same time it can be taken into consideration that relatively low density and good corrosion resistance may find a wider applicability as a prosthesis material.

(Received July 30, 2015; accepted September 9, 2015)

Keywords: Titanium alloys, Heat treatment, Microstructure, Hardness, Resilience

1. Introduction

Due to its outstanding properties, titanium has established itself as a precious metal in many fields; its low density, high mechanical strength, favorable thermic and chemical properties brought it to the attention of both researchers and industry. Furthermore, the existence of two allotropes α (hexagonal compact) and β (body-centered cubic) presents a great practical importance; many of these characteristics are enhanced by the development of alloys. The main alloying element is aluminum, but there are used for the same purposes Mo, V, Mn, Zr, Cu, Ta, etc. [1,4]

The structural composition of the titanium alloy can be monophasic α, monophasic β or biphasic α + β. In binary alloys, but especially in the polinare ones there are created complex structures which present many intermetallic compounds. The alloying elements also stabilize or increase the existence of a phase or another. The aluminum stabilizes the α phase and the β-stabilizing elements are V, Mo, Cr.

The α phase is tougher, has a higher mechanical strength and the β phase increases the plasticity and dissolves some impurities which would increase the fragility of the alloy. The most widely used titanium alloys are biphasic and characterized by good deformability at heat, weldability, mechanical processability.

The existence of allotropic forms provides real possibilities of modifying some mechanical properties by heat treatments. Two-phase alloys have multiple transformations in solid state based on both the α↔β transformation and the formation of soluble phases; therefore the applying of hardening and annealing or hardening and aging heat treatments becomes possible [5,6,7,8].

- The hardening can be done in the β-phase or the biphase α + β, the β → M martensitic transformation occurring. In the case of titanium alloys, several types of martensite can be obtained by hardening from the solid solution β.

Fig. 1 illustrates the transformations during the hardening for diagrams with or without eutectoid transformation.

Under the equilibrium diagram from the above figure there are represented the phase diagrams at the hardening temperature Tc and respectively at ambient temperature after hardening, Tamb.

Depending on the type of titanium alloy, as shown, there can be obtained after hardening several types of martensitic structures, namely: [1,2,3]

- α '- solid solution oversaturated of betagene elements; has a distorted hexagonal lattice. From the structural point of view has the appearance of needle and has a hardness which increases the saturation concentration of the components;
- α″- solid solution with rhombic lattice. Its hardness is lower than the one of the α ' phase;
- Sometimes at high cooling speeds the β phase preserves at ambient temperature β: β → βs or β → β depending on the concentration;

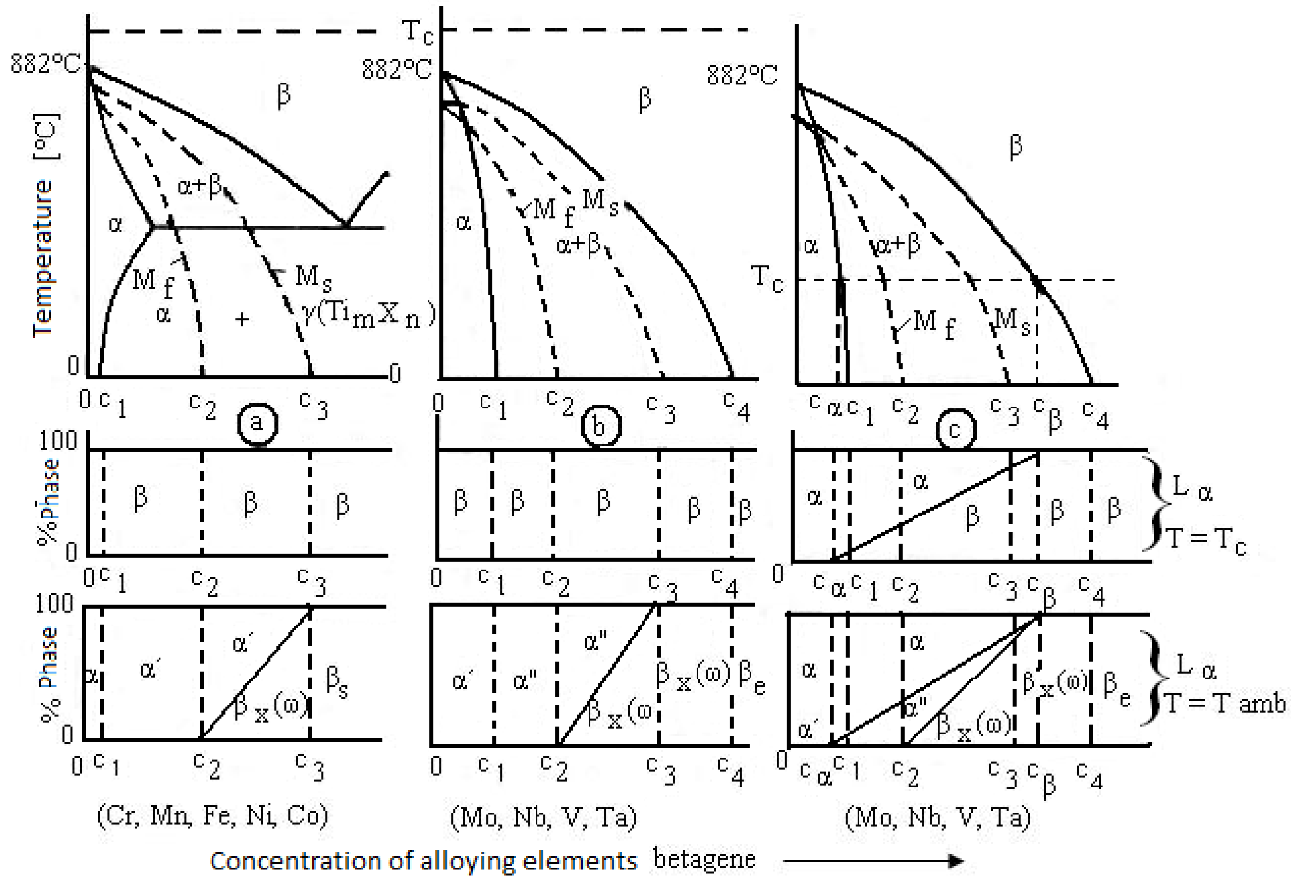

Fig. 1 Hardening of titanium aluminum alloys for the following cases: a) equilibrium diagram with eutectic transformation and hardening of the β phase; b) equilibrium diagram without eutectic transformation and hardening of the β phase; c) diagram without eutectic transformation and hardening of the α + β - phase (intercritical hardening) [3]

- β_s - oversaturated solid solution (unstable over time); Therefore, in the body-centered cubic lattice appear areas coherently related to it, but with a hexagonal crystalline lattice. This newly formed phase is denoted by βx or ω. The βs → βx (ω) partial transformation weakens the alloy (natural aging stage I);

In the case of biphasic alloys (α + β) subjected to intercritical hardening there are obtained structures such as:

α + α ', α + α', α + βs, α + βx (ω) and others.

-Annealing and artificial aging

During the heating of the hardened α -type alloys in their structure occur decomposition processes such as:

α '→ α + TimXn

The heating temperature significantly influences the kinetics and extent of transformation, as follows:

• At low temperatures 200 ... 300 ° C, newly formed α particles are fine and uniformly distributed - the hardness is less influenced. The higher the temperature is, the more the α particles expand by coalescence, and the more the hardness decreases slightly to about 600 ° C. During the heating of the alloys without eutectic transformation (ω type) the heating may produce changes such as:

βx → β + ω
α '→ βx + ω

These transformations are similar to the second stage of aging and lead to a hardness increase, respectively to a toughness decrease.

The hardening also produces the following transformation:

βs + βx + ω → ω

However, if the heating temperature increases to more than 450 ... 500 ° C the following conversion takes place:

βs + ω → α + βe,

thus reaching the third stage of aging,along with a hardness decrease and a resilience increase.

In conclusion, the alloys with eutectoid are subjected to hardening and annealing heat treatments (with a hardness decrease during the annealing) and the alloys without euectoid are subjected to hardening and artificial aging heat treatments; in the second case the hardening occurs during the aging (stages I and II) and respectively soaking in the third stage of artificial aging.

2. Experimental research

For the experimental tests we used an alloy from the Ti-Al system with applications both in industry and implants, namely Ti6Al4V, whose standardized and effective chemical composition is shown in Table 1

Table 2 presents some resistance characteristics supplied by producers (alloy in the state of delivery)

Table1 Chemical composition

Alloy type	Indication type	[%]Chemical composition									
		Al	V	Fe	C	N	O	H	residual elements		Ti
									Each	total	
Ti6Al4V	afterAS9100	5,5-6,75	3,5-4,5	≤0,4	≤0,08	≤0,05	≤0,2	≤0,015	≤0,1	≤0,4	rest
	measured	6,23	4,10	0,19	0,02	0,02	0,19	0,003	<0,1	<0,4	rest

Table 2. The resistance characteristics

Alloy type	Indication type	Tensile resistance R [MPa]	$R_{0,2}$ flow resistance [MPa]	δ [%] Elongation at break	ψ [%] Constriction at break	[HRC] Hardness
Ti6Al4V	AS9100 ISO 14001	>895	>828	10	25	-
	experimental	1030	953	20	54	33

The alloy in the state of delivery (annealed) falls in the type α + β. Samples of this material were subjected to heat treatment operations under different conditions of temperature and time to emphasize changes in the structure and of the mechanical properties.

Table 3 presents heat treatments experimentally performed and the results for hardness and resilience

Table 3. The heat treatments and the results

Material	Hardening				Annealing				
	Temp [°C]	Cooling environment	[HRC] hardness	KCU [J/cm^2] resilience	Temp [°C]	[min] duration	Cooling environ ment	[HRC]	Rezilienţa KCU [J/cm^2]
Ti6Al4V	1020	Ventilated N_2	53,46	37,17	-	-	-	-	-
					500	30	N_2	58,13	46,33
					500	60	N_2	54,99	48,12
					850	60	N_2	52,43	49,25
	950	water	54,90	32,41	-	-	-	-	-
	850	water	56,56	36,58	-	-	-	-	-
					500	30	aer	53,80	44,25
					500	60	aer	54,63	40,42
					500	120	aer	56,00	41,66
	Delivery state		42,56	55,30	-	-	-	-	-

Observation. The annealing at 1020° C was effectuated in a vacuum oven, as well as the corresponding annealings. In other embodiments the heating took place in laboratory ovens with air atmosphere.

The following Figs. 2-14 show the micrographs of structures obtained after the heat treatments effectuated according to Table 3. For highlighting the metallographic structure used a reagent consisting of potassium hydroxide, KOH - 10% (12 cm^3), H2O2 peroxide - 30% (15cm3) and water 78 cm^3 .

Fig. 2. Ti6Al4V Titanium alloy, delivery state, 500:1

Fig. 3. Ti6Al4V Titanium alloy, delivery state, 1000:1

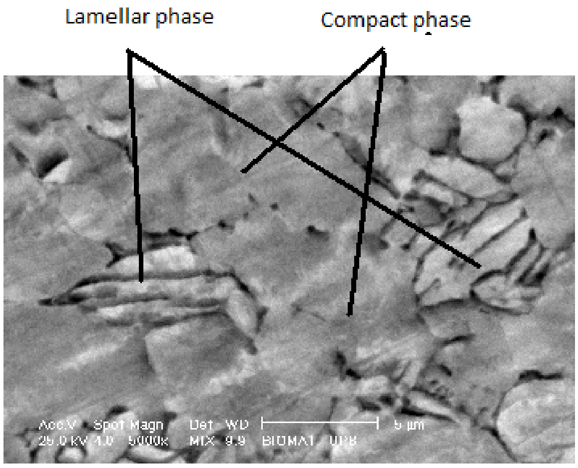

Fig. 4. Microscopy ESEM for Ti6Al4V in delivery state

Fig. 5. Ti6Al4V Titanium alloy, vacuum hardening at 1020°C, 90 minutes maintenance, cooling in ventilated nitrogen 1000: 1

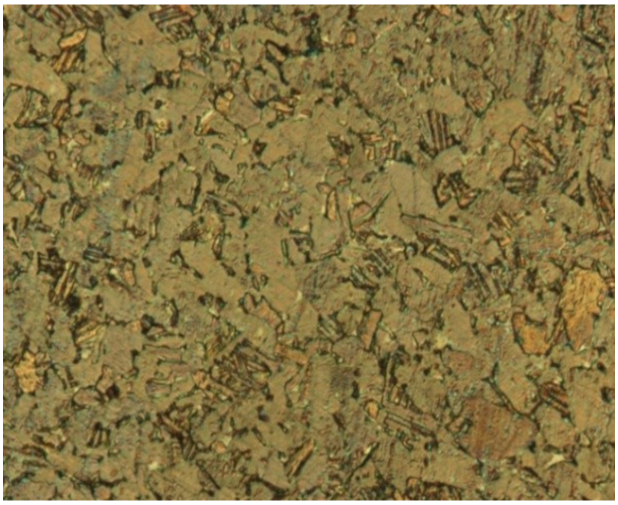

Fig. 6. Ti6Al4V Titanium alloy, vacuum hardened at 1020°C and annealed at 850°C for 60 minutes, 1000:1

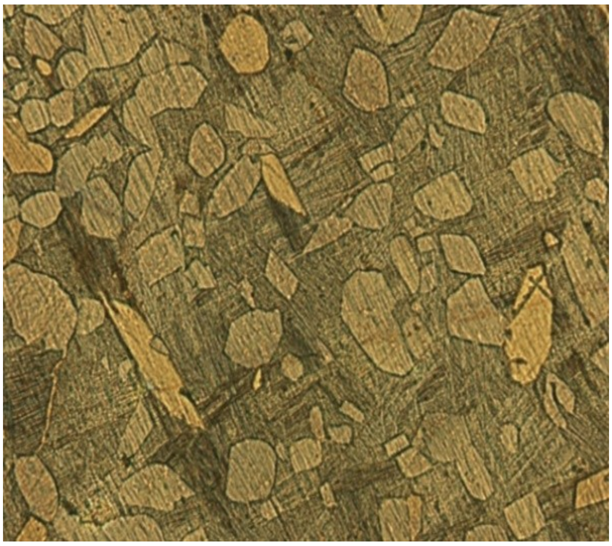

Fig. 7. Ti6Al4V Titanium alloy, vacuum hardened at 1020°C and annealed at 500°C for 30 minutes, 1000:1

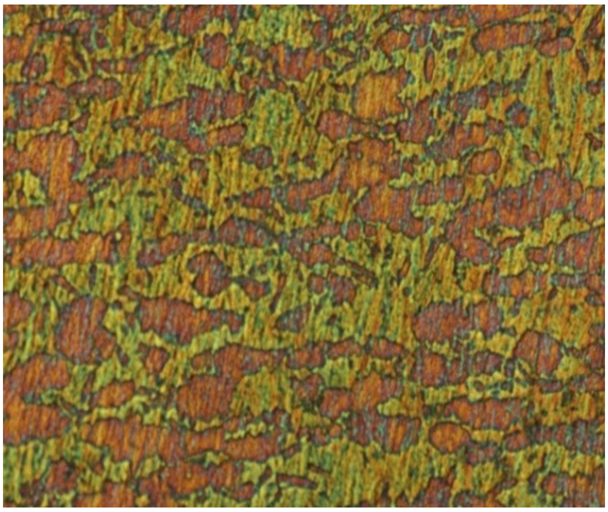

Fig. 8. Ti6Al4V Titanium alloy, hardened in water starting from 950°C, 1000:1

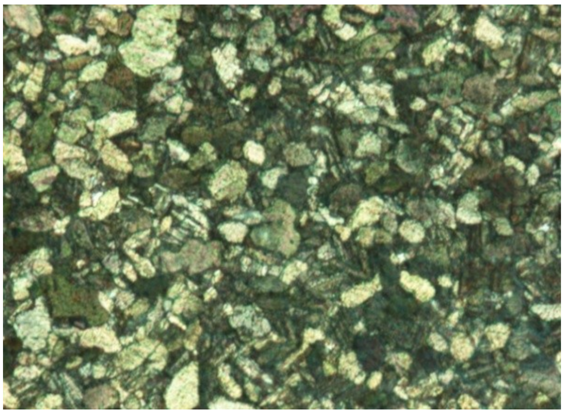

Fig. 9. Ti6Al4V Titanium alloy, hardened in water starting from 850°C, 1000:1

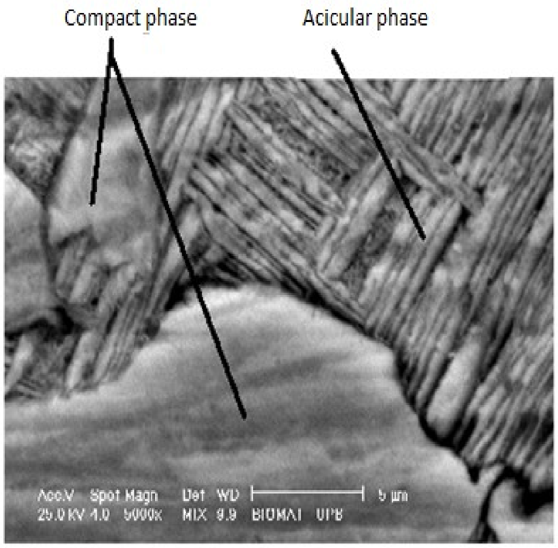

Fig. 10. Microscopy ESEM for Ti6Al4V hardened in water starting from 850°C

Fig. 11. Ti6Al4V Titanium alloy, hardened in water starting from 850°C and annealed for 30 minutes at 500°C, 1000:1

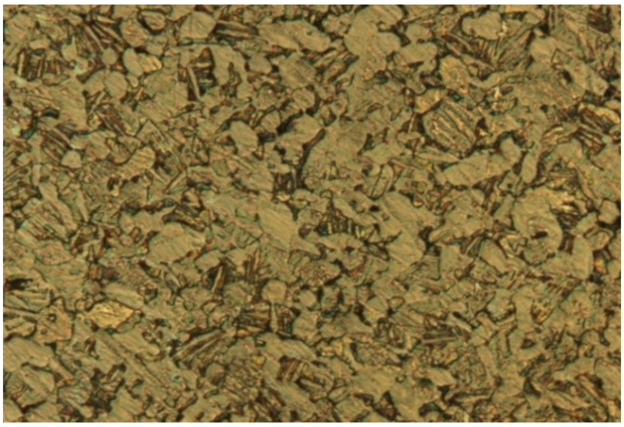

Fig. 12. Ti6Al4V Titanium alloy, hardened in water starting from 850°C and annealed for 60 minutes at 500°C, 1000:1

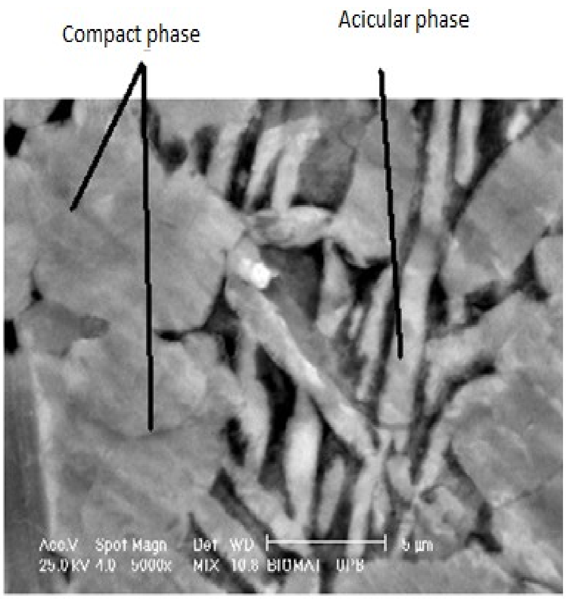

Fig. 13. Microscopy ESEM for Ti6Al4V hardened in water starting from 850ºC and annealed for 60 minutes at 500°C

Fig. 14. Ti6Al4V Titanium alloy, hardened in water starting from 850ºC and annealed for 120 minutes at 500°C, 1000:1

- the heatings applied after the hardening serve as artificial aging serve with different effects depending on the temperature.

Thus, at a heating temperature of about 500 ° C, the changes in the structure fall within the second stage of aging. The hardness is slightly increasing among with the maintenance duration. The structure is composed of a mixture of α + βs + ω + TimXn. The presence of α and βs phases in the structure maintain a relatively high resilience.

At a heating temperature of 850 ° C and the transition to the third stage of transformation, the structure becomes α+ βe + TimXn, structure confirmed by the hardness and resilience values.

From the presented micrographs, correlated with the hardness and resilience values there can be identified the following structures due to the suffered heat treatments.

3. Conclusion

- In the delivery condition (annealed) the structure consists of a mixture of α + β, α being the predominant phase[5-10];

- The heating at 1020 ° C, the maintaining and the cooling in ventilatednitrogen (equivalent with thecooling in oil) produce a change in size and shape of the crystals; in the structure there are identified the following constituents: α '+ βx + ω. The presence of the ω phase influenced the resilience which decreased by about 40% along with a moderate increase of the hardness;

- the heating for the hardening in the intercritical field and the cooling in air leads to a structurally aspect with more shaped crystals of larger size (size partially stored from the initial state); the α 'and α " phases are the structural constituents;

From the above we can draw the following conclusions:

- The Ti6Al4V Titanium alloy , which has a biphasic equilibrium structure allows the application of heat treatments with significant structural changes;

- The Ti6Al4V alloy is sensitive to the variation of thermic and temporal parameters of the heat treatment;

- The structural changes and the ones of the mechanical characteristics also occur after the heatings which are subsequent to the hardening; the transformations are specific to those of an artificial aging. The temperatures bounding the transformation stages are within 450 ... 550 ° C;

- The heat treatments applied to α + β alloys must aim the achieving of greater resiliences depending on a certain level of the other mechanical characteristics.

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