"HENRI COANDA"
AIR FORCE ACADEMY
ROMANIA

"GENERAL M.R. STEFANIK"
ARMED FORCES ACADEMY
SLOVAK REPUBLIC

INTERNATIONAL CONFERENCE of SCIENTIFIC PAPER
AFASES 2014
Brasov, 22-24 May 2014

ESEM AND X-RAY EMISSION SPECTRA OF TITANIUM ALLOY IN DIFFERENT STRUCTURAL STATUS

Camelia PITULICE, Adriana ZARA, Nicoleta TORODOC, George VASILE

TRANSILVANIA UNIVERSITY OF BRASOV

Abstract : *Upon Ti6Al4V titanium alloy there were effectuated different heat treatment operations. The results obtained were tested by numerous processes such as optical microscopy, mechanical and technological attempts, and also ESEM - Enviromental Scanning Electron Microscopy investigation and X-ray analysis , respectively. The latter are the objectiv of this paper. Trough these observations there has been highlighted a series of aspects regarding the composition and internal structure of the alloy in different states obtained by heat treatment . There has been also establish the proportion between the structural phases. These analyzes come to complete the data necessary for an accurate assessment of the influence of thermal treatment concerning use characteristics. Thus, it can be chosen the structural state according to practical necessites. There are presented in this paper, some of this investigations.*
Keywords: *titanium alloy, metallographic structure, hardening and tempering, ESEM- Enviromental Scanning Electron Microscopy , X-ray spectra.*

1. INTRODUCTION

Titanium alloys, they benefit from numerous advantages concerning the properties, as compared with other metallic materials, such as low density (about 4.5 g/cm^3), high refractoriness, corrosion resistance, good weldability, mechanical properties appreciable. Meanwhile, the two allotropes Ti α compact hexagonal lattice and Ti β with volume centered cubic lattice, offers them application availability of many heat treatment operations. [1, 3]. Among the addition elements of titanium, the most common are aluminum, vanadium, molybdenum, chromium. The titanium alloys, with properties that can appear in a wide range of value found accordingly and varied utility such as in aeronautics, marine, chemical energy, chemical industry, in medical prostheses, etc. [2] . For the present research it has been chosen Ti6Al4V titanium alloy whose chemical composition is given in Table 1.

Table 1. Chemical composition of the alloy Ti6Al4V

Sort of alloy	Chemical composition, [%]							
	Al	V	Fe	C	N	O	H	Ti

Ti6Al4V	6,23	4,14	0,20	0,02	0,02	0,19	0,003	rest

2. APPLIED THERMAL TREATMENTS

From the above composition of semi-finished, there were made attemps specimens required. These were subjected to heat treatments given in Table 2.

Table 2. Thermal treatments applied titanium alloy Ti6Al4V

Nr. crt.	Applied Treatments	Temp. [°C]	Time [min]	Cooling Medium	Hardness [HRC]	Resilience [KCU]
1	Delivery status	-	-	-	42,56	55,30
2	Hardening	850	60	apă	53,46	36,58
3	Tempering (aging)	500	60	apă	54,63	40,42

The heating temperature was set to 850 ° C, found in the stability domain of the α phase. Maintanance duration it was determined so that to be realized both, the temperature uniformity on section and also the development time needed to make structural changes. Considering the increase in hardness after heating at 500 ° C subsequent hardening, it appears that this operation is actually an artificial aging. Successively the samples treated, they had structures such as:

$$\text{inițial } \alpha+\beta \xrightarrow{\textit{hardening from } 850°C} \alpha+\alpha''+\beta \xrightarrow{\textit{aging at } 500°C} \alpha+Ti_mX_n+\beta \text{ [2]}$$

in which: - α" - has a martensitic structure with a rhombic chrystalline lattice;

- Ti_mX_n - an intermetallic phase which precipitates during cooling and maintaining from 500 ° C.

3. THE RESULTS OF ANALYSIS BY ESEM AND SPECTRAL X

Enviromental Scanning Electron Microscope - ESEM - is a method for investigating of surfaces studied micro and nano scale. On the analyzed area it is sent an electron beam that scans an certain area; it will generate more signals, which received and processed generates information regarding on the chemical composition of the samples and structure. Investigations revealed the following:

- For sample in delivery status. Figure 1 shows the micrograph of the existence of two main constituents, one with an aspect compact α and another with a cvasilamelar aspect, a mixture of α + β.

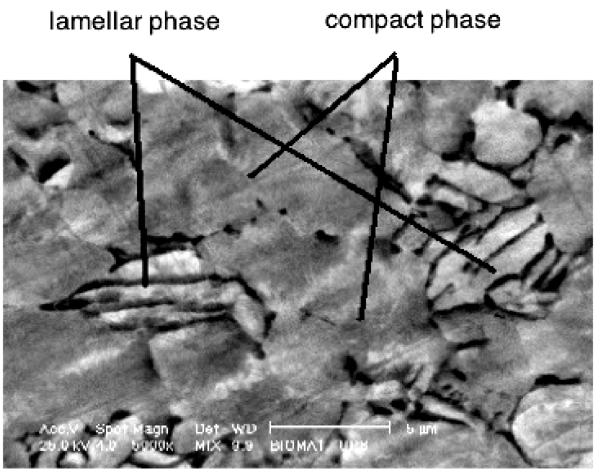
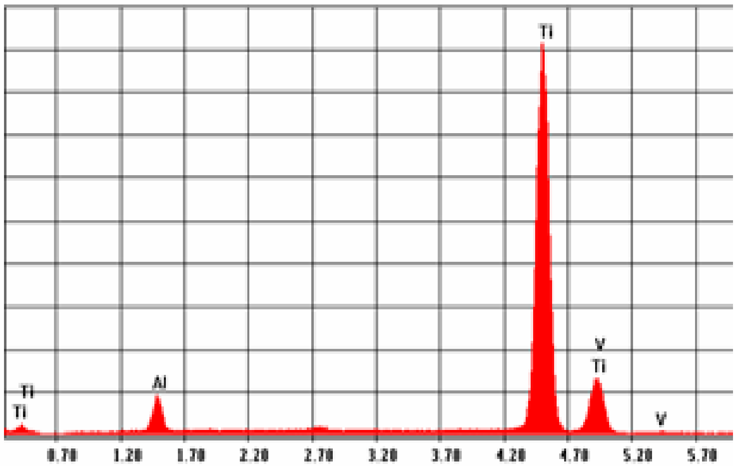


Figure 1. Micrograph alloy Ti6Al4V in delivery status obtained by ESEM .

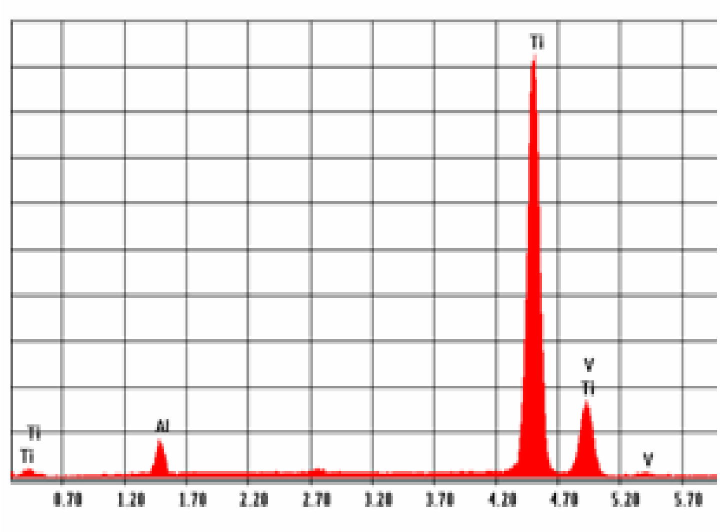
The emission spectrum of compact phase of solid solution α, identifies an content of titanium and aluminum. In the lamellar phase was identified among titanium and aluminum also vanadium in addition rate of 3.25%. The X-ray emission spectra for these two constituents and the chemical composition are shown in Figures 2 and 3.



Element	Percent by weight Wt [%]
Al	7.91
Ti	92.09
V	0
Total	100

Figure 2. The X-ray emission spectrum of the compact phase. Ti6Al4V alloy. Delivery status.

.



Element	Percent by weight Wt [%]
Al	6.92
Ti	89.82
V	3.25
Total	100

Figure 3. Ray emission spectrum in the lamellar phase.

According to program ImageJ analysis resulted a 62% proportion of compact phase and 38% lamellar phase. - for the sample water hardening from 850° C. For this case also there have been effectuated ESEM analysis and X-ray emission spectrum. Metallographic structure (fig. 4) reveals o radical change compared with the previous , the two constituents being more accurate delimited. The compact phase of solid solution reduced quantitatively; the constituent on mechanic mixture type gained an acicular aspect with a higher share.

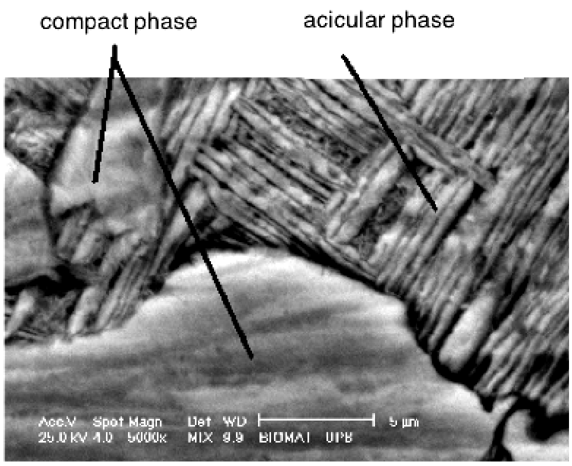
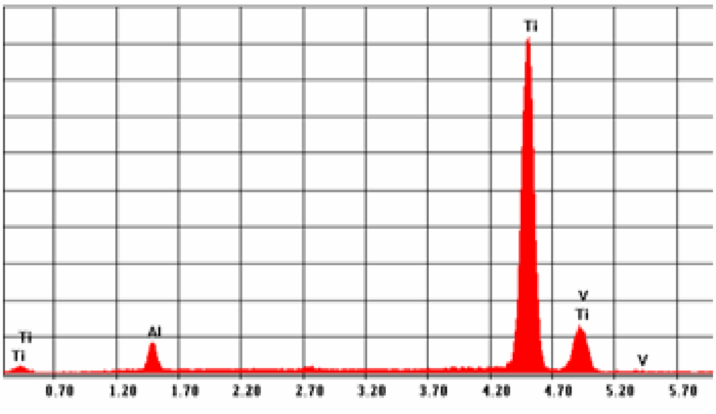


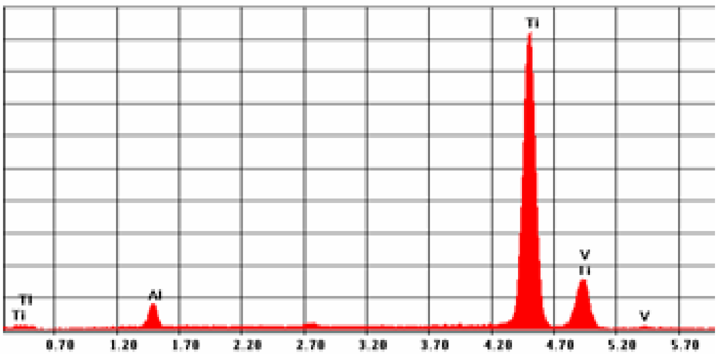
Figure 4. ESEM micrograph for Ti6Al4V alloy, hardened in water at 850 ° C.

This is composed of a mixture of phases α "+ β + Ti_mX_n. Changing the quantitatively report between constituents, as well as finely dispersed structure and qualitatively different from the original mixture phases (acicular phase) explains the differences measured of some mechanical properties.



Element	Percent by weight Wt [%]
Al	7.37
Ti	92.63
V	0.00
Total	100

Figure 5. X-ray emission spectrum in the compact phase. Ti6Al4V alloy. Hardening in water from 850 ° C.



Element	Percent by weight Wt [%]
Al	6.94
Ti	90.97
V	2.09
Total	100

Figure 6. X-ray emission spectrum in the acicular phase. Ti6Al4V alloy. Hardening in water from 850 ° C.

The quantitatively report of constituents in this state is 38,6% compact phase and 61,4% acicular phase : - for sample hardening from 850° C and aging at 500° C. After hardening from 850°C effectuated with a higher speed in water, on some samples it has been applied a one hour reheating at 500° C. As was shown this heating , by its effects is an aging ; more specifically this is an artificial aging, which place the alloy in second stadium of this process. During the heating occurs an complete process of precipitation of a secondary phaseTi_mX_n .

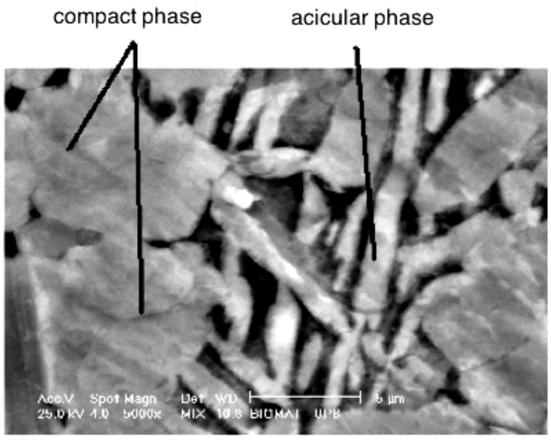
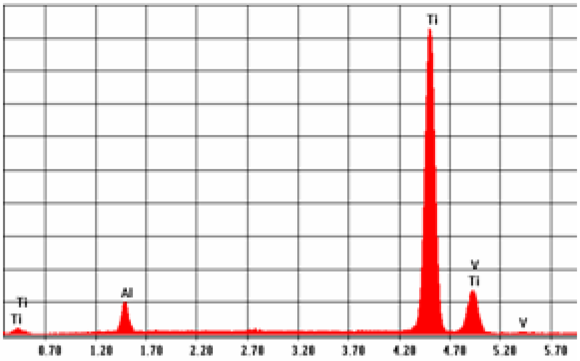


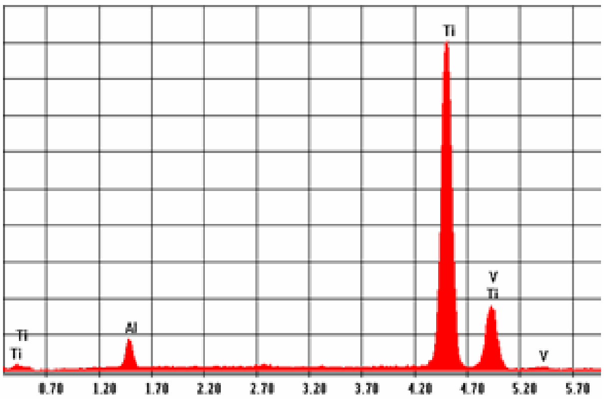
Figure 7. ESEM image of Ti6Al4V alloy, water hardened from 850 ° C and aged at 500 ° C.

Emission spectrum of the compact phase found the presence of titanium and aluminum components (see Figure 8). Emission spectrum of the mechanical mixture with acicular aspect indicates also the presence in a proportion of 4.81% of vanadium (Figure 9).



Element	Percent by weight Wt [%]
AlK	8.36
TiK	91.64
V K	0.00
Total	100

Figure 8. Emission spectrum in the compact phase. Ti6Al4V alloy. Tempering and aging at 850 ° C to 500 ° C



Element	Percent by weight Wt [%]
AlK	7.06
TiK	88.12
V K	4.81
Total	100

Figure 9. Emission spectrum in the RX acicular phase . Ti6Al4V alloy. tempering and aging at 850 ° C to 500 ° C.

In this case, the quantitatively report of constituents is 65.5% compact phase and 34.5% acicular phase .

4. CONCLUSIONS

Thermal treatments aim to change some physical-mechanical characteristics in order to obtain the necessary properties for further processing or use. These properties are determined by the structural composition of alloy and the quantitatively report of the phases.

Experimental attempts of the present work were able to produce significant changes in the structural aspect of Ti6Al4V alloy, supported also by mechanical properties (density, resilience).

Through investigations effectuated by electron microscopy and X-ray it was revealed internal structure of the alloy in three different status, refering to the size and shape of the grains, the nature of the phases of the structure, their reciprocally arrangement also quantitatively report of phases.

All this is fully consistent both with the sequence of thermal operations but also with performed properties. Also, it is concluded that in the case of the present alloys and thermal parameters used, the heating subsequently hardening could be enclose in the artificial aging. The temperature of 500 ° C places the alloy in stage II of the aging process.

5. REFERENCES

1. Collings, EW, Boyer, R., Welsch, G., *Materials properties handbook. Titanium Alloys,* ASM International, pp. 3-170, 483-609, (1994).aaaaaaaaaaaaaaaaaaaaa aaa
2. Dumitrescu, C., Serban, R., *Physical metallurgy and heat treatment*, Ed. Fair Partners, Bucharest, vol. II, (2001).
3. Fatu, S., Tudoran, P., *Study of metallic materials. Thermal treatments. Alloy steels. Alloys. New Materials*, Vol. II, Ed. Libris, Braşov, pp. 74-75, 80-82, 99-111, (1997).
4. Pitulice, C., - *Studies and research on biocompatible materials used in prosthetics*, Thesis - Transilvania University of Brasov, (2013).