International Journal of Modern Manufacturing Technologies
ISSN 2067–3604, Vol. VII, No. 2 / 2015

PHYSICAL AND STRUCTURAL CHARACTERIZATION OF TI-BASED ALLOY

Bălţatu Mădălina Simona[1], Vizureanu Petrică[1], Istrate Bogdan[2]

[1]"Gheorghe Asachi" Technical University of Iasi, Department of Technologies and Equipment for Materials Processing, 41 "D. Mangeron" Street, 700050, Iasi, Romania
[2]"Gheorghe Asachi" Technical University of Iasi, Department of Mechanical Engineering, Mecatronics and Robotics, 43 "D. Mangeron" Street, 700050, Iasi, Romania

Corresponding author: Petrică Vizureanu, peviz2002@yahoo.com

Abstract: Metallic biomaterials like cobalt based alloys, stainless steel and titanium based alloys are the most used in medical applications, as orthopedic implants, dental materials and cardiovascular devices. Pure titanium and titanium alloys are widely used as implant materials in the medical and dental applications because of their superior biocompatibility, corrosion resistance and specific strength compared with other metallic implant materials. The paper present a study of a Ti implant screw used in dental application. Microstructure and phase analysis of Ti dental screw was investigated by using optical and scanning electron microscopy, X-ray diffraction (XRD). The importance of knowing the behavior of Ti-based alloys at high temperatures provides information on the ways that can be improved mechanical properties, physical and technological. Therefore it was studied thermal expansion changes for Ti implant screw, subjected heating to 1200ºC by using dilatometric analysis.
Key words: Ti- based alloys, biomaterials, SEM, XRD, microstructure dilatometric analysis.

1. INTRODUCTION

Ti-based alloys is used in many fields due to a unique combination of physical, chemical and mechanical properties. The domains are aerospace industry (figure 1a), chemical industry, shipbuilding, energy industry, medicine (figure 1b), food, transport (figure 1c), [1, 2].

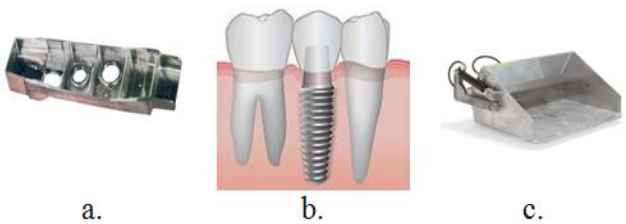


Fig. 1. Ti-based alloys applications [3-5]: a) aerospace industry, b) medicine, c) transport

Application of Ti-based alloys in dentistry are in continuous expansion. Ti-based alloys has been recently introduced, but is more used instead Co-based alloys, because have good properties for biomedical applications like high biocompatibility, resistance to corrosion and low density, [2, 6].

Titanium and titanium alloys are clinically well-suited as biomaterials and play significant roles in the longevity of the prostheses and implants, [7].

Titanium is an allotropic element which has two crystallographic forms. At the low temperature Ti has a hexagonal close-packed (hcp) which is referred to as „alpha“ phase. At the temperatures 883ºC this structures transforms to a body-centered cubic (bcc), called „beta“ phase, [8].

Ti-based alloy with includ other elements contribute to the transformation phase. Alloying elements generally can be classified as alpha or beta stabilizers. Al, O, N, Ga are consider alpha stabilizers, stabilize apha phase at the high temperatures. Mo, V, W and Ta are beta stabilizers, stabilize beta phase at the low temperatures [8, 9].

At mechanical processing, for alloys based on Ti can occur variations in size of the high temperature to generate phenomena of thermal expansion partially irreversible, processing on the affected areas. Therefore, dilatometer analysis was used to determine the length changes influenced by temperature increase for Ti-based alloys [10-14]. It was study the dilatometric analysis to see the influence of the Ti-based alloys heating up to 1200ºC.

2. MATERIALS AND METHODS

The experimental material used in this study was one Ti implant dental screw as a sample. The purpose of this research was to study of microstructures, phase and dilatometer analysis of a Ti-based alloy.

The chemical composition of the surface of the Ti sample is shown in Fig. 3. SEM images and EDX – chemical composition analysis are shown by using a FEI Quanta 200 3D dual beam SEM (figure 2a), [15, 16].

Determination of the compositional phases was performed by qualitative analysis by X-ray diffraction, made on the diffractometer X'Pert PRO MPD PANalytical X-ray (figure 2b) with the following parameters: continous scan, 2θ - (10º-90º), Step size: 0.0131303, Time per step: 61, 20, Scan speed: 0.05471, 45KV and 40mA using a copper anode X-ray tube.

a) b)

Fig. 2. Structural analysis equipments:
a) SEM QUANTA 200 3D DUAL BEAM
b) X'Pert Pro MPD Diffractometer

The sample was grinding and polishing then attacked metallographic aimed to highlighting structural constituents. Metallographic structure of Ti was shown by the attack surface with a chemical solution having the following composition: 10ml HF, 5ml HNO_3, 85 ml H_2O immersed in 5-30s.

The devise used in microstructural analysis for Ti alloy was metallographic microscope Leica 5000DMI. Scanning electron microscope used was FEI Quanta 200 3D SEM dual beam, [7, 17].

After studying the microstructure, it was studied the dilatometer analysis for sample of Ti.

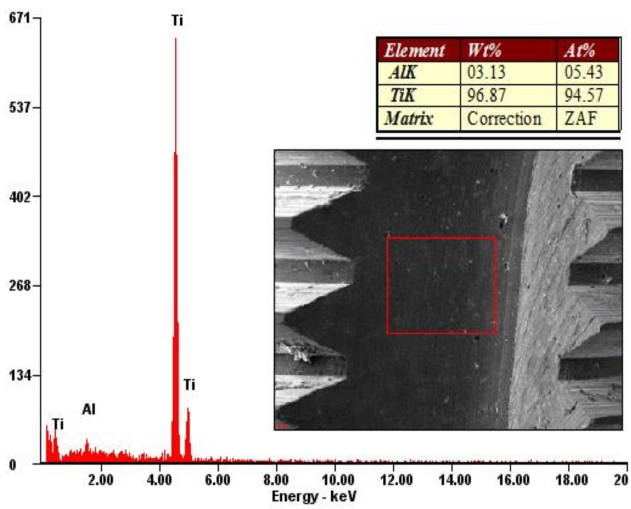


Fig. 3. EDX spectrum and chemical composition of Ti sample

At mechanical processing, for alloys based on Ti can occur variations in size of the high temperature to generate partially irreversible phenomena of thermal expansion processing on the affected areas.

Under these conditions we studied the behavior of a Ti sample, subjected to heat, for their application (medical applications) and high temperatures that can be reached during their processing (preparation, melting and casting into molds specific medical application), [13, 18, 19].

Dilatometer analysis is used to determine the coefficient of thermal expansion and transformation points in solid materials.

It is based on expansion-temperature curves occurrence of irregularities at temperatures of transformation from the normal appearance of these curves, deviations caused by dimensional changes of the parts, [20, 21].

The heating rate of the test sample was 10°C/min and cooling rate of the furnace is determined by the flow rate of water, $5m^3/h$. Dilatometer analysis was performed with a differential Dilatometer LINSEIS type, model L75H-1400 (figure 4), [7].

Fig. 4. Dilatometer LINSEIS type, model L75H-1400

Heating and cooling of Ti sample was performed in a horizontal cylinder furnace with electric resistance. The sample temperature during analysis can be measured and recorded with a thermocouple placed near the sample.

Alpha phase transformation in beta Ti in Ti-Al binary system occurs at 882ºC, according with the binary diagram Ti-Al.

At the dilatometer analysis occurs αTi influencing the structure causing a contraction at 858ºC.

3. RESULTS AND DISCUSSIONS

3.1 Phase and microstructure

X-ray structural analysis is the method to determine the direction and intensity of the diffracted radiation and deduce the order of these atoms, [13].

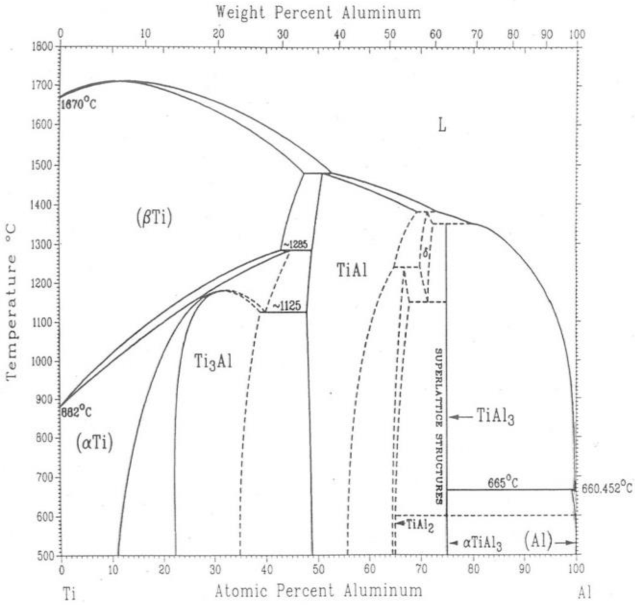
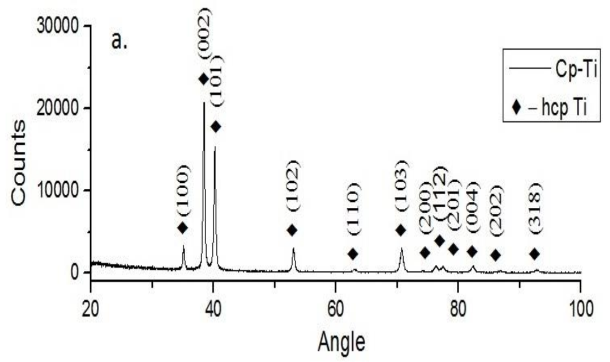
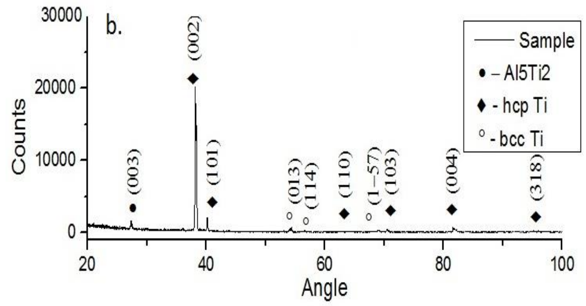


Fig. 5. Ti-Al phase diagram, [9]

The XRD results present a comparison between Cp-Ti and Ti-Al sample. Phase analysis, Miller parameters and lattice parameters are shown in figure 6 and table 1. Cp-Ti has a single hcp phase and Ti-Al sample present three phases: the main hcp-Ti phase, the bcc-Ti phase and Al5Ti2 compound with the following parameters: a – 3.9053Å, b- 3.9053 Å, c- 29.1963Å.



a.



b.

Fig. 6. XRD patterns of sample and Miller parameters: a) Cp-Ti, b) Ti3Al

In figure 6 and table 1are presented XRD pattern with typically peaks of Cp-Ti and Ti based alloy.

The predominant alloy phase of cp-Ti and Ti-based alloy is hcp Ti at the angle 2θ=38.4546.

In sample of cp-Ti was found hcp Ti phase with the major peak at the angle 2θ=38.4546.

In the sample Ti-Al was found the hcp Ti main peak with 2θ = 38.4415, bcc Ti main peak with 2θ = 54.3685 and compound Al5Ti2 main peak with 2θ = 27.4908

Table 1. Compound parameters

Compound	Cp-Ti	Sample		
	Ti	Ti	Ti	Al5Ti2
Space Group	P63/mmc	P63/mmc	Im-3m	P4/mmm
Crystal system	Hexagonal	Hexagonal	Cubic	Tetragonal
a(Å)	2.95	2.95	3.32	3.90
b(Å)	2.95	2.95	3.32	3.90
c(Å)	4.68	4.69	3.32	29.19
α(°)	90	90	90	90
β(°)	90	90	90	90
γ(°)	120	120	90	90
Cell volume (10^6 pm^3)	35.30	35.33	36.59	445.28

The percent amount of phases is highlighted in figure 7.

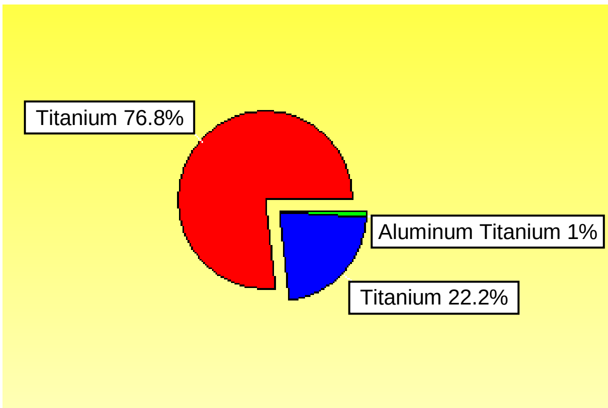


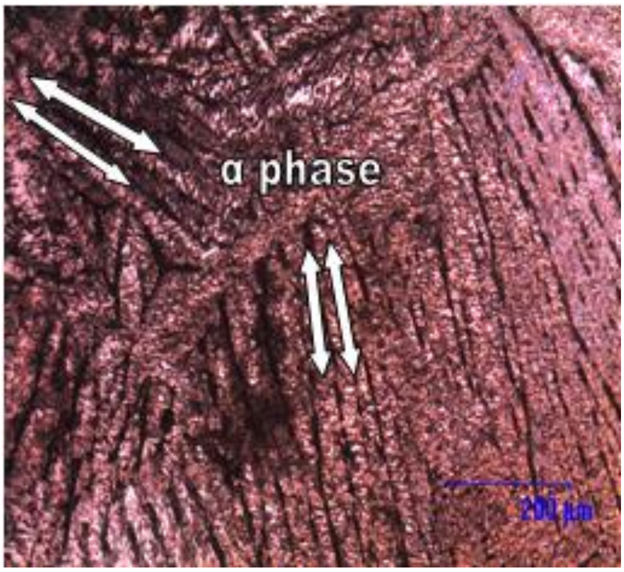
Fig. 7. Quantitative phase analysis of Ti3Al

It was found that the Ti hexagonal structure is in proportion to about 77%, Ti cubic approximately 22% and Al5Ti2 compound at a rate of 1%. It highlights a slight difference from EDAX analysis, which is a front surface beside to XRD analysis which is an analysis in volume.

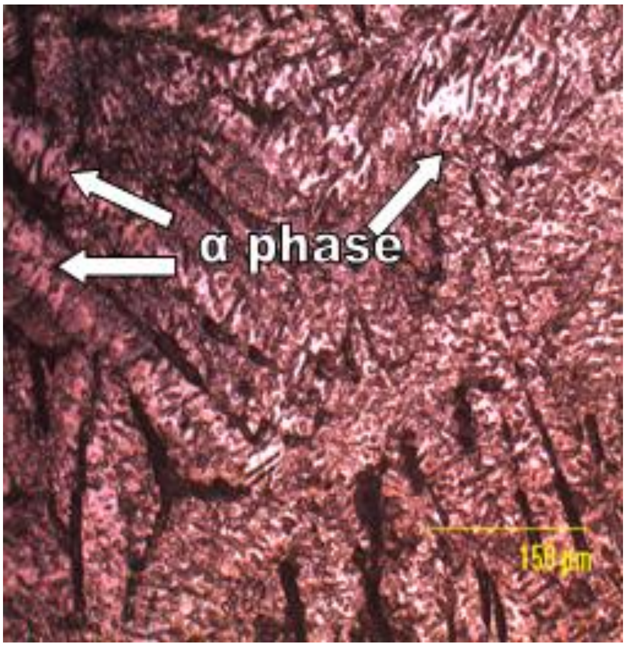
A characteristic crystalline structure of Ti-based alloys is shown in figure 8 with dendritic structure due to high speeds with α type grains.

In accordance with the scientific literature, [8], in Fig. 8, are shown by optical microscopy dendritic appearance and rough form for specific grains αTi of phase at different magnitudine.

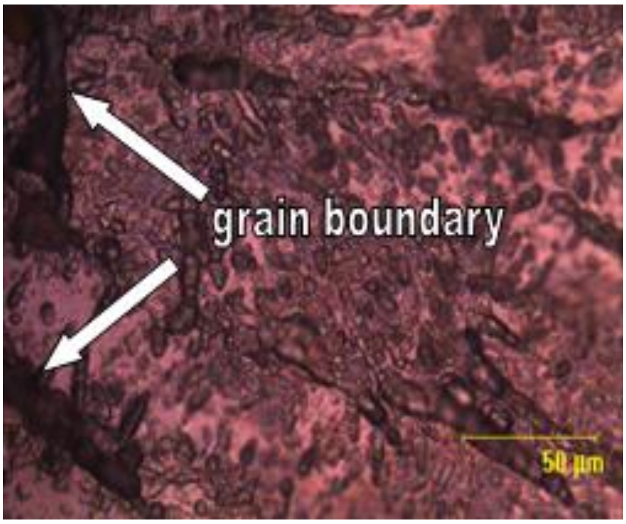
In figure 8a, b, the arrows show us α phase. The grain boundary of specific αTi phase is highlighted in figure 8c.



a.



b.

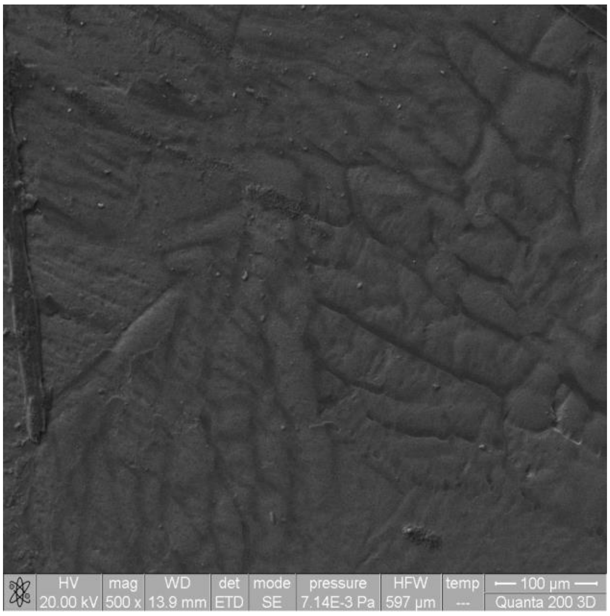


c.

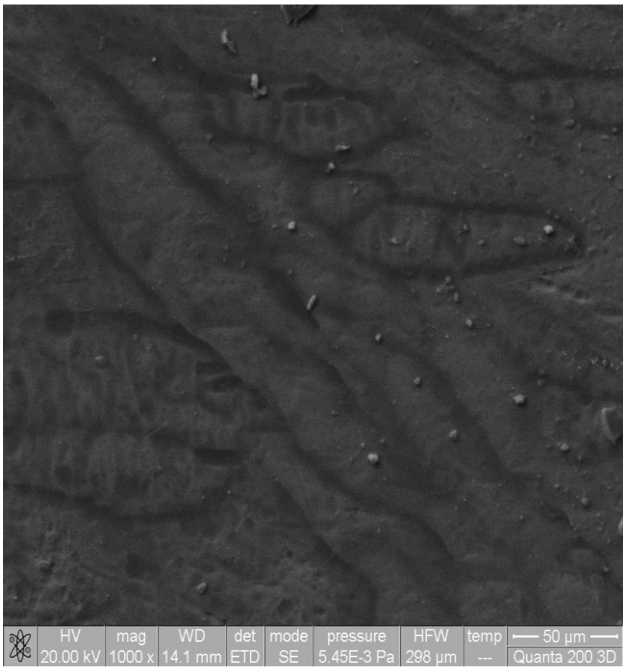
Fig. 8. Microstructure of Ti sample: a) 100X, b) 200X, c) 500X

The electronic microscopy analysis in figure 9, confirm the optical misctroscopy analysis with elongated equiaxed grains willing dendritic due to cooling solidification at high speeds.

For unalloyed Ti, α phase is stable at temperatures exceeding 880ºC, above this value α phase transformation occurs in β phase.



a.



b.

Fig. 9. SEM image of Ti sample: a) 500X, b)1000X

Titanium alloying with other elements (Mo, V, W, Ta, Al, O, N, Ga) contribute to the transformation phase, aluminium stabilizes α phase at high temperatures, [1, 8].

3.1 Dilatometric analysis

Dilatometer analysis is to determine the behavior of metallic materials under heating. The sample of Ti

had a linear dilation at 858ºC until then presented a linear shrinkage. The maximum thermal expansion reached by the sample at 642ºC temperature is by 15.1μm.
Braked linear contraction is caused by alpha Ti phase transformation of hexagonal close-packed in beta Ti body-centered cubic structure in the temperature of 870 ºC. Alpha phase transformation in beta Ti in Ti-Al binary system occurs at 882ºC, according with the binary diagram Ti-Al.

Also can consider this braked linear shrinkage can occur under the action of internal factors (part thickness, internal stress) and external factors (walls form, coolers), [8, 17].
Phase αTi over 858ºC temperature causes a decrease in the length of the sample during heating. The sample behavior of Ti alloy may be observed in Fig. 5. Like is shown, the sample present a contraction after temperatures of 858ºC and an elongation by -44.4μm at 1198.7ºC.

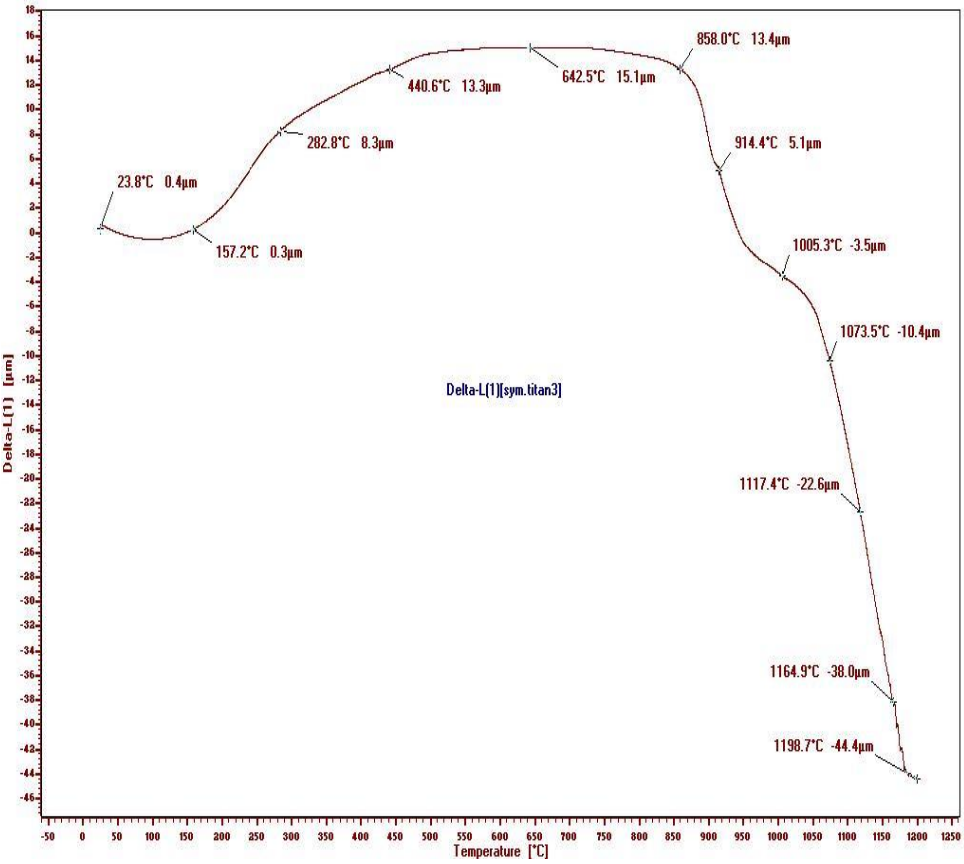


Fig. 10. Elongation variation with temperature for Ti sample

In Table 2 is present the elongation variation by heating temperature for sample of Ti.

Table 2. Elongation values function by temperature for Ti sample

Temperature [ºC]	Thermal expansion [μm]
23.8	0.4
282.8	8.3
642.5	15.1
858.0	13.4
914.4	5.1
1073.5	-10.4
1117.4	-22.6
1164.9	-38.0
1198.7	-44.4

Value of α thermal expansion coeffient for the Ti-based alloys is important to know from the follow considerations:

-The coefficient value must be low for maintain constant the tolerances of implant;
-The coefficient of metallic-implant must be correlated with the biological materials with which it comes into contact, [13].

4. CONCLUSIONS

The importance of knowing the behavior of Ti-based alloys at high temperatures provides information on the ways that can be improved mechanical properties, physical and technological.
The purpose of paper was study the microstructure, phase and dilatometer analysis for a Ti sample. Microstructure of Ti sample was studied by optical and electronic microscopy at different magnifications. The appearance of the sample

studied is dendritic with coarse grain shape specific α phase.
The XRD results present a comparison between Cp-Ti and Ti-Al sample. Cp-Ti has a single hcp phase and Ti-Al sample present three phases: the main hcp-Ti phase, the bcc-Ti phase and Al5Ti2 compound.
It was used dilatometer analysis to determine the behavior of metallic materials under heating of the temperature rise for a Ti sample.
The sample of Ti had a linear dilation at 858ºC until then presented a linear shrinkage braked caused by alpha Ti phase transformation of body-centered cubic in beta Ti body-centered cubic structure.

4. REFERENCES

1. Uram-Tuculescu, S., Bratu, E., Lakatos, S., (2001). *Titanul in stomatologie*, Signata, Timisoara, pp. 19-52.
2. Lutjering, G., Williams, J.C., (2003). *Titanium*, Springer-Verlag, Berlin, pp. 289.
3. Available from http://www.ttonline.ro/, Accessed: 13/05/2015.
4. Available from http://www.dentistonline.ro/, Accessed: 12/05/2015.
5. Avaible from http://www.mowdirect.co.uk/, Accessed: 10/05/2015.
6. Li, Y., Yang, C., Zhao, H., Qu, S., Li, X., Li, Y., (2014). *New Developments of Ti-Based Alloys for Biomedical Applications*, Materials, pp. 1709-1800.
7. M. Niinomi, (2002). *Recent metallic materials for biomedical applications*, Metallurgical and Materials, Metallurgical and Materials Transaction A, Vol. 33, pp. 477-486.
8. ***, ASM Handbook, Metallography and Microstructure, Vol. 9, pp. 2157-2208.
9. ***, ASM Handbook, Alloy Phase Diagrams, Vol. 3, pp. 254.
10. Minciuna, M.G., Vizureanu, P., Achitei, D.C., Cimpoesu, N., (2014*). Study of dimensional changes by dilatometric analysis for some CoCrMo alloys*, International October Conference on Mining and Metallurgy, Bor, pp. 433-435.
11. ***Instruction Manual, Horizontal Dilatometer Platinum Series*, Leinseis, www.linseis.com.
12. Yamane, T., Ueda, J., (1996). *Mechanical properties of commercial pure titanium at high temperature*, The Japan Institute of Metals and Materials, No. 7, pp. 91-95.
13. Minciună, M.G., Vizureanu, P., Achiței, D.C., Ghiban, N., Sandu, A.V., Forna, N.C., (2014). *Structural caracterization of same CoCrMo alloys white medical applications*, Revista de Chimie 65(3).
14. Zhao, W., Zhang, W., Guo, J., Wang, B., Guo J., Lu K., (2006). *Microstructure evolution and tensile properties of pure Ti subjected to rapidly heating and quenching*, Journal of Materials Science & Tehnology, Vol. 22, pp. 190–194.
15. Available from http://www.panalytical.com/, Accessed: 10/05/2015.
16. Available from Information on http://www.fei.com/, Accessed: 10/05/2015.
17. Ciobanu, G., Carja, G., Ciobanu, O., Sandu, I., Sandu, A., (2009). *SEM and EDX studies of bioactive hydroxyapatite coatings on titanium implants*, Micron, 40(1), pp. 143-146.
18. Chenf, K., Chiu, K.H., (2005). *Stamping formability of pure titanium sheets*, Journal of Materials Processing Technology, Vol. 170, pp. 181-186.
19. Elias, C.N., Lima, J.H.C., Valiev, R., Meyers, M.A., (2008). *Biomedical applications of titanium and its alloys*, Biological Materials Science, pp. 46-49.
20. Ohwue, T., Shindo, T., Hayashi, T., (2002). *Square shell deep drawability of commercially pure titanium sheet*, Nippon Steel Technical Report, Vol. 85, pp. 125-128.
21. Pop, S.I., Dudescu, M., Bratu, D.C., Pop, R.V., Petrisor, M., Păcurar, M., (2013). *Influence of the chemical composition on the mechanical properties of orthodontic archwires*, Revista de Chimie 64(7), pp. 771-775.

Received: June 10, 2015 / Accepted: December 10, 2015 / Paper available online: December 20, 2015