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The Influence of Annealing And Chemical Composition Variation on The Microstructure And Mechanical Properties of Biomedical Titanium Alloys

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Abstract: *There are a number of advantages of using titanium alloys for biomedical applications. The purpose of this investigation was to examine the effects of alloying and heat treatment on the microstructure and hardness of biomedical Ti-Cr-Co alloys. Samples were prepared by melting pure elements in a laboratory arc furnace under an argon atmosphere. They were metallographically prepared and analyzed with the X-ray diffraction method. Microstructures of experimental alloys were taken with digital camera and analyzed with computer program Image Tool. Hardness was measured using the Vickers indentation method. Heat treatment was carried out at 950°C over 3 hours with quenching in water. The microstructure and hardness were analyzed after the heat treatment. The results obtained show heat treatment effects the volume ratio of phases in alloys with 10 at.% of cobalt, but alloys with 5 at.% of this alloying element were retained as single-phase. Also, hardness of investigated alloys depends on content of alloying elements (chromium and cobalt), but the effect of heat treatment on the hardness of this alloys was negligible. The results shows that the microstructure and hardness are dependent on the addition of alloying elements as well as heat treatment of Ti-Cr-Co alloys.*

Keywords*: Ti-Cr-Co alloys, biomedical alloys, heat treatment, microstructure, hardness*

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I. Introduction

In the past decade, replacement of diseased teeth, bone or joints by artificial materials is gaining increasing usage in orthopedic surgery in order to relieve pain and recover maximal possible functionality. To achieve this goal, specialized implant materials are required. Titanium and its alloys are extensively used as biomedical implant materials because their good mechanical properties are accompanied by an excellent biocompatibility and corrosion resistance. The most widely used Ti alloy for medical implants is Ti-6Al-4V. Since vanadium and aluminum are known to be cytotoxic elements, alternative Ti-based alloys with other alloying elements are currently the subject of intensive study as new biomaterials. In this work, chromium and cobalt were used as stabilizers of β-phase, which has favorably properties for use in a medical application. In addition, chromium reduces the melting temperature of titanium and cobalt strengthens the alloys. The mechanical properties of titanium can be also enhanced through the addition of these elements [1-6].

The purpose of this investigation was to establish the influence of chromium and cobalt on titanium as well as heat treatment effects on the microstructure and hardness of Ti-Cr-Co alloys with potential as medical implant materials.

II. Materials and methods

The chemical compositions (in atomic percent) of investigated ternary Ti-Cr-Co alloys were as follows: $Ti_{85}Cr_{10}Co_5$, $Ti_{80}Cr_{10}Co_{10}$, $Ti_{70}Cr_{25}Co_5$ and $Ti_{65}Cr_{25}Co_{10}$. They were prepared by melting pure elements in an argon atmosphere using a nonconsumable tungsten electrode arc. The ingots were re-melted three times to improve their chemical homogeneity. Consequently, the cylindrical samples with 8 mm in diameter were prepared by copper mold casting. Those samples were sectioned using a Buehler Isomet low-speed diamond saw to attain several samples for different examinations. The small samples were mounted in an epoxy resin and metallographically prepared by grinding and polishing. Specimens for light microscope observation were etched with solution: 2.5 cm^3 $HF_{conc.}$, 2.5 cm^3 $HNO_{3\ conc.}$, 70 cm^3 H_2O at room temperature.

X-ray diffraction (XRD) for phase analysis was conducted by using a diffractometer (Philips PW3710) operating at 40 kV and 40 mA. Ni-filtered Cu K_α radiation was used for this study, wherein the phases were identified by matching each characteristic peak with the JCPDS files [7].

The microstructures of prepared specimens were observed by light microscope (Leitz, Ortholux) at the magnification of 280 x. Microphotographs were taken with a digital camera, an Olympus DP11. They were used for quantitative analysis which was carried out by the computer program Image Tool UTHSCSA.

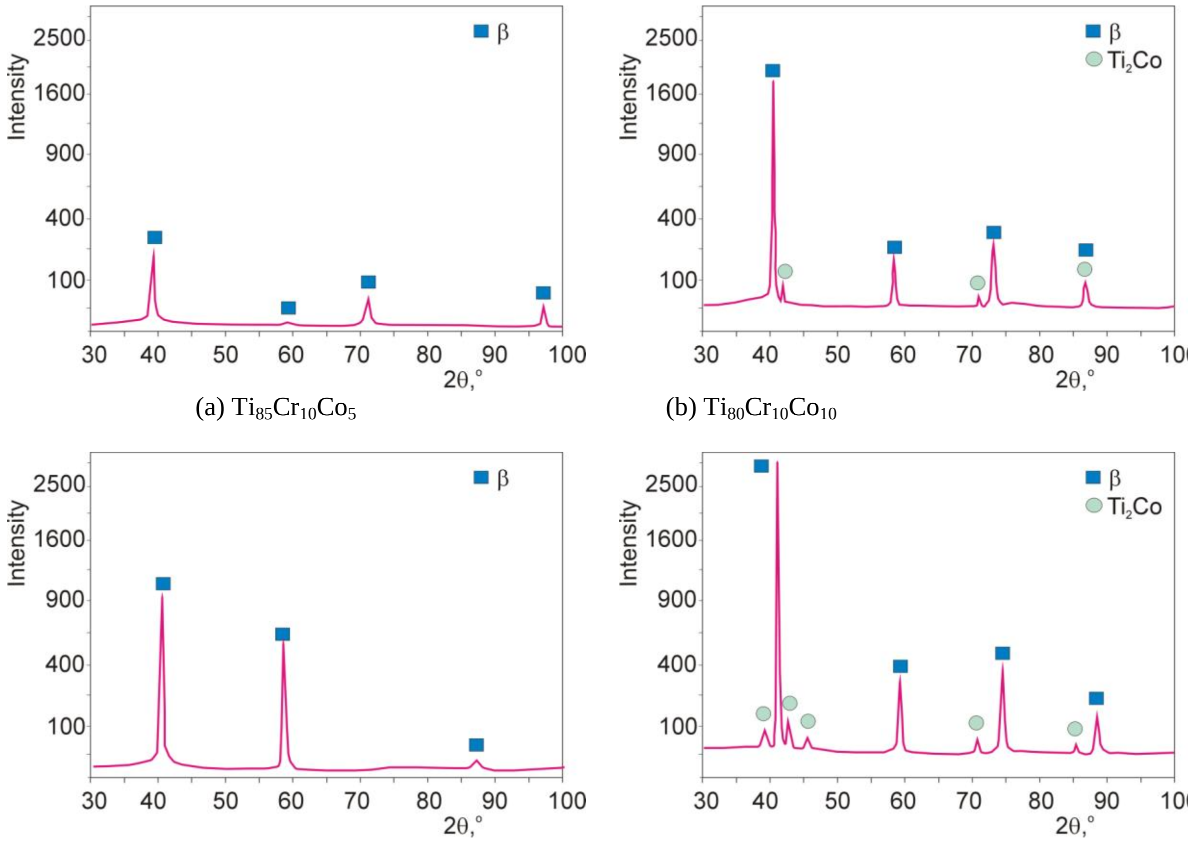
Hardness measurements were performed by the Vickers indentation method on the equipment Leica with load 19.60 N and holding time of 10 s.

The heat treatment of experimental alloys was carried out with the aim of improving the microstructure and mechanical properties. The samples of all as cast alloys were encapsulated in quartz in vacuum and heat treated at 950°C for 3 hours followed by quenching in water. The microstructure and hardness of alloys after heat treatment were analyzed again with methods described above.

III. Results and discussion

This paper shows that the microstructure and hardness of Ti-Cr-Co alloys is dependant on the addition of alloying elements as well as the heat treatment process used.

X-ray diffraction patterns of alloys with 10 at. % of cobalt (Figs. 1b, 1d), i.e. $Ti_{80}Cr_{10}Co_{10}$ and $Ti_{65}Cr_{25}Co_{10}$, show two phases identified as β phase, which is dominant, and the intermetallic compound Ti_2Co. XRD patterns of alloys with 5 at. % of cobalt (Figs. 1a, 1c) show diffraction maximums of β phase only. It follows that with reduction of cobalt content from 10 to 5 at. % the intermetallic compound Ti_2Co formation is avoided. In this way, the microstructure of alloys with lower cobalt content includes just the β phase. At the same time, chromium content in these alloys is higher than 10 at. % which is enough for retaining the cubic body-centered crystal structure of β phase [2].



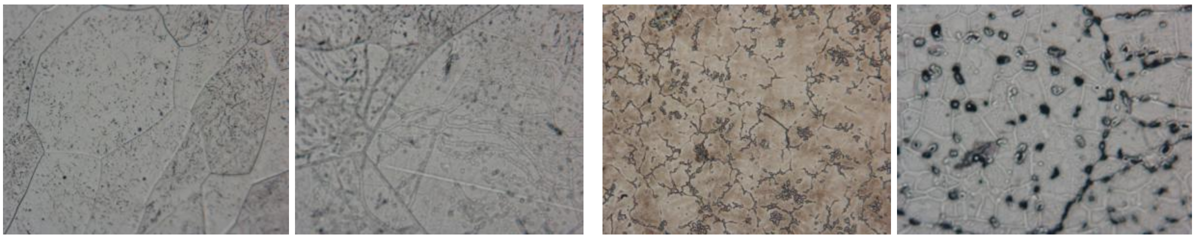
(a) $Ti_{85}Cr_{10}Co_5$ (b) $Ti_{80}Cr_{10}Co_{10}$

(c) $Ti_{70}Cr_{25}Co_5$ (d) $Ti_{65}Cr_{25}Co_{10}$

Figure 1. XRD patterns of as cast experimental alloys

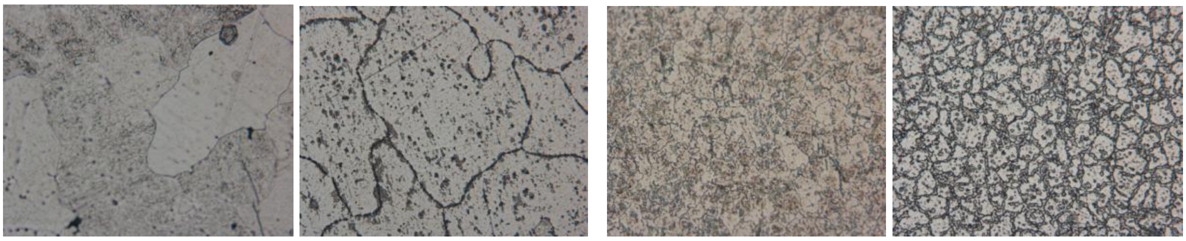
Figure 2 shows light micrographs of as cast experimental alloys taken with digital camera. The microstructure of alloys $Ti_{85}Cr_{10}Co_5$ and $Ti_{70}Cr_{25}Co_5$ (Fig. 2a, 2e) is composed of one phase, which is according to the XRD analysis results identified as the β phase. This phase exhibits very coarse grains, similar to other Ti-Cr biomedical alloys [8]. As cast alloys $Ti_{80}Cr_{10}Co_{10}$ and $Ti_{65}Cr_{25}Co_{10}$ have a three-phase microstructure, which is composed of a metallic matrix, a second phase on the grain boundaries and the third phase inside the grains

(Fig. 2c, 2g). The metallic matrix is β phase, because it's the dominant phase on the XRD patterns and shows the strongest diffraction maximums. Likewise, second phase is identified as the intermetallic compound Ti_2Co, which precipitates on the grain boundaries. Since the XRD patterns for these two alloys show only maximums for two phases the third phase was in such small amounts which could not be detected by the XRD method. According to the corresponding diagrams [8] it was identified as the α phase.



(a) as cast **(b) heat treated** **(c) as cast** **(d) heat treated**

$Ti_{85}Cr_{10}Co_5$ $Ti_{80}Cr_{10}Co_{10}$



(e) as cast **(f) heat treated** **(g) as cast** **(h) heat treated**

$Ti_{70}Cr_{25}Co_5$ $Ti_{65}Cr_{25}Co_{10}$

Figure 2. Light micrographs of as cast and heat treated alloys (280x)

After annealing heat treatment the alloys $Ti_{80}Cr_{10}Co_{10}$ and $Ti_{65}Cr_{25}Co_{10}$ show a two-phase microstructure which consists of the β-matrix and an intermetallic compound, Ti_2Co, at the grain boundaries of β phase (Figs. 2d, 2h). A microstructure comparison of heat treated and the as cast condition of these two alloys implies that the third α phase is no longer present. In the alloys $Ti_{85}Cr_{10}Co_5$ and $Ti_{70}Cr_{25}Co_5$, which were single-phase in their as cast condition, a very small amount of intermetallic compound Ti_2Co was precipitated at the grain boundaries of β-matrix after heat treatment. In addition, it resulted with significantly enlargement of the β phase grains.

A quantitative analysis (Table 1) has revealed that among heterogeneous alloys, i.e. alloys with 10 at. % of cobalt, will have a higher β phase portion those alloys with higher titanium content and smaller chromium content.

Table 1. The chemical compositions and quantitative metallography results for as cast and heat treated experimental alloys

Alloy composition, at.%	As cast				Heat treated		
	% β	% Ti_2Co	% α	Average area of grains, μm^2	% β	% Ti_2Co	Average area of grains, μm^2
$Ti_{85}Cr_{10}Co_5$	100,00	-	-	1117,12	100,00	-	-
$Ti_{80}Cr_{10}Co_{10}$	66,39	30,62	2,99	130,17	92,94	7,06	72,30
$Ti_{70}Cr_{25}Co_5$	100,00	-	-	3517,87	100,00	-	-
$Ti_{65}Cr_{25}Co_{10}$	56,73	36,79	6,48	71,54	53,29	46,71	144,26

The volume portions of the intermetallic compound and α phase increase with decreasing titanium and increasing chromium content. The alloy $Ti_{80}Cr_{10}Co_{10}$, with greater portion of β phase, has coarser grains. The data in Table 1 shows that single-phase β alloys have very coarse average area of grains.

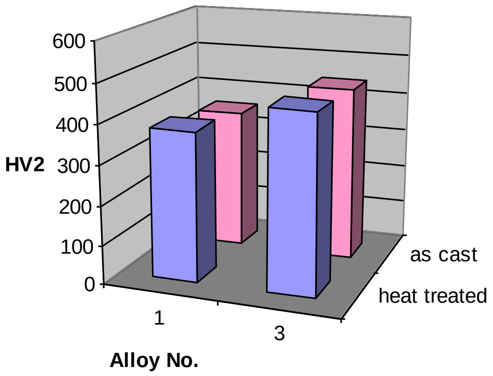
The results of quantitative metallography of heat treated alloys (Table 1) show that intermetallic compound portion decreased in alloy $Ti_{80}Cr_{10}Co_{10}$ for 23,56% and increased in alloy $Ti_{65}Cr_{25}Co_{10}$ for 14,92% in comparison with the as cast condition. The same is valid for the average area of grains, which could not be determined for the other two alloys. The reason for this lies in the fact that the grains are too big and therefore excluded from the micrographs.

The Vickers hardness measurements (Table 2) show the strong effect of chemical composition and microstructure on hardness values. Among the as cast alloys with 10 at. % of cobalt, hardness values increase with increasing chromium content (from 10 to 25 at. % of Cr), which is also observe by Majumdar et al [2]. It could be explained with the following hypotheses: during the Ti-Cr solid solution formation, around the chromium atom in crystal structure of titanium voids were formed, because the diameter of chromium atom is smaller than that of titanium. Consequently, the chromium content increase causes the enlargement of voids whereupon alloys hardness increases. However, hardness upgrowth of this alloys with 10 at. % of cobalt primary is associated with the presence of intermetallic compound Ti_2Co. Therefore, hardness of these three-phase alloys increases with the increase of the intermetallic compound portion (Table 1). Likewise, among the single-phase alloys, hardness increases with increasing chromium content. Measured values (359 and 444 HV2) are similar to that of the other β-type biomedical alloys [9].

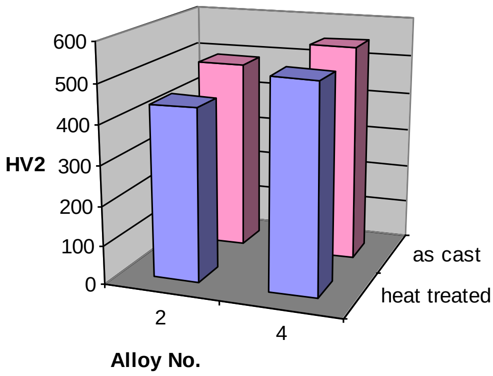
Table 2. Vickers hardness of as cast experimental alloys

Alloy No.	Alloy composition, at.%	HV2	
		As cast alloys	Heat treated alloys
1	$Ti_{85}Cr_{10}Co_5$	359	381
2	$Ti_{80}Cr_{10}Co_{10}$	489	439
3	$Ti_{70}Cr_{25}Co_5$	444	455
4	$Ti_{65}Cr_{25}Co_{10}$	550	527

Vickers hardness values of heat treated alloys (Table 2) are very close to those of as cast alloys (Fig 4).



(a) Alloys with 5 at.% Co



(b) Alloys with 10 at.% Co

Figure 4. Vickers hardness comparison of as cast and heat treated alloys

After heat treatment $Ti_{80}Cr_{10}Co_{10}$ and $Ti_{65}Cr_{25}Co_{10}$ show a similar tendency to slightly decrease in hardness when compared to the as cast condition. Thereat volume portion of intermetallic compound was changed differently. It follows that the grain size of alloys after heat treatment has a more important affect on this mechanical property. Hardness decrement could be related to the fact that the α phase is no longer seen in the microstructure of these alloys. Namely, it is known that the α phase exhibits the higher hardness values than the β phase [10]. Alloys $Ti_{85}Cr_{10}Co_5$ and $Ti_{70}Cr_{25}Co_5$, after heat treatment, possessed a higher hardness value in comparison with the as cast condition. That could be explained by the precipitation of negligible amount of intermetallic compound Ti_2Co, mainly at the grain boundaries of the β phase.

IV. Conclusions

The microstructure and hardness of alloys $Ti_{85}Cr_{10}Co_5$, $Ti_{80}Cr_{10}Co_{10}$, $Ti_{70}Cr_{25}Co_5$ and $Ti_{65}Cr_{25}Co_{10}$ in as cast condition and after heat treatment were investigated. The following results were obtained:

1. Microstructure of as cast alloys with 10 at. % of cobalt consists of a β phase, the intermetallic compound Ti_2Co and a α phase, while in that of alloys with 5 at.% of Co only the β phase is present.
2. The β phase portion in alloys with 10 at. % of Co decreases with decreasing titanium and increasing chromium content. Simultaneously, the portion of intermetallic compound increases.
3. Among alloys with 10 at. % of Co heat treatment did not result in homogenous single β phase alloys, although α phase was transformed into the β phase. Namely, the intermetallic compound Ti_2Co was retained.
4. Vickers hardness of as cast alloys with 10 at. % of Co increases with increasing chromium and Ti_2Co content. Hardness of single β-phase alloys also increases with increasing chromium content.
5. Vickers hardness of alloys with 5 at. % of Co after heat treatment was little higher in relation to the as cast condition, while that of alloys with 10 at. % of Co was decreased.

References

[1]. A. Cremasco, P.N. Andrade, R.J. Contieri, E.S.N. Lopes, C.R.M. Afonso, R. Caram, Correlations between aging heat treatment, ω phase precipitation and mechanical properties of a cast Ti-Nb alloy, Materials & Design, 32 (2011) 2387-2390

[2]. W-F. Ho, T-Y. Chiang, S-C. Wu, H-C. Hsu, Mechanical properties and deformation behavior of cast binary Ti-Cr alloys, Journal of Alloys and Compounds 468 (2009) 533-538

[3]. E. Eisenbarth, D. Velten, M. Müller, R. Thull, J. Breme, Biocompatibility of β-stabilizing elements of titanium alloys, Biomaterials 25 (2004) 5705-5713

[4]. W-F. Ho, C-H. Pan, S-C. Wu, H-C. Hsu, Mechanical properties and deformation behavior of Ti-5Cr-xFe alloys, Journal of Alloys and Compounds 472 (2009) 546-550

[5]. W-F. Ho, S-C. Wu, H-H. Chang, H-C. Hsu, Structure and mechanical properties of Ti-5Cr based alloy with Mo addition, Materials Science and Engineering C 30 (2010) 904-909

[6]. G. He, J. Eckert, Q.L. Dai, M.L. Sui, W.Loser, M. Hagiwara, E. Ma, Nanostructured Ti-based multi-component alloys with potential for biomedical applications, Biomaterials 24 (2003) 5115-5120

[7]. Powder Diffraction File Search Manual, JCPDS International Centre for Diffraction Data, Swarthmore, 1982.

[8]. Alloys Phase Diagrams, ASM Handbook, Vol.3, ASM International, 2002.

[9]. H-C. Hsu, S-C. Wu, T-Y. Chiang, W-F. Ho, Structure and grindability of dental Ti-Cr alloys, Journal of Alloys and Compounds 476 (2009) 817-825

[10]. I. Weiss, S.L. Semiatin, Thermomechanical processing of beta titanium, Materials Science and Engineering A 243 (1998) 46-65

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