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# EFFECT OF SILVER CONTENT ON THE WEAR AND MECHANICAL PROPERTIES OF POWDER METALLURGICAL Ti-5Al-2.5Fe-xAg ALLOYS

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#### Abstract

In the current research, the effect of Ag on the mechanical properties of Ti5Al2.5Fe alloy was investigated. The Ti5Al2.5Fe alloy, with different amounts of Ag ranged from 1 to 5 wt. % was prepared by mechanical mixing and then fabricated by hot pressing at 950 °C for 15 min under 50 MPa. Three holding steps were applied to the powder compacts to restrain the liquid phases inside graphite die before reaching the maximum sintering temperature. The sintered samples were subjected to hardness, bending and wear tests to study the effect Ag on the mechanical properties of Ti5Al2.5Fe alloy. The microstructural characterization was carried out by means of optical and scanning electron microscope. The results showed that Ag played a differential role on the mechanical properties supported by microstructural constituents. The bending strength and hardness of the produced samples increased with the addition of Ag, the hardness of the alloys then tended to decrease with increasing Ag content but still remained above the hardness of Ti5Al2.5Fe alloy. Wear test also showed similar trends with hardness test results. Finally, the optimum Ag content for the Ti5Al2.5Fe alloy was determined as 1 wt.%. XRD analysis showed that undissolved Ag content was the main reason for the decrease in the mechanical properties.

Keywords: Ti5Al2.5Fe alloy; Silver Content; Microstructure; Wear

## 1. Introduction

Low density and high mechanical properties make Ti allovs one of the most preferred materials in military and civil applications [1]. Beside these advantages, high corrosion resistance and biocompatibility properties are other ideal properties to create orthopedic prostheses, orthodontics and joint replacements with Ti alloys [2]. However, there are some restrictive reasons in the production of Ti alloys such as high melting point and high chemical reactivity to oxygen and nitrogen at high temperatures. The applied possible solutions are to change the production technique or decrease melting temperature by alloying with other elements [3]. Ti alloys are generally produced by casting, forging, and metallurgical techniques. powder Powder metallurgical production techniques of producing Ti alloys provide advantages such as faster and near net shape production cycle [4].

Recently, many studies have been carried out on titanium alloys containing silver. The addition of Ag on the titanium alloys has an intensive effect on both

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mechanical and antibacterial properties. Ti-Ag alloys exhibit better mechanical properties than pure Ti [5]. Wen et al., studied Ti-Nb-Ag alloys, produced by powder metallurgical techniques and observed that Ag precipitation around  $\beta$  phase significantly affects the fracture characteristics of the alloy [6]. Xiao et al., reported that Ag addition in Ti-5Al-4Mo-4V alloy increased the relative density and compression properties [7]. Takahashi et al., investigated mechanical properties of 22.5, 25, 27.5 and 30 wt.% Ag addition in Ti and they announced that a great enhancement in hardness and tensile strength occurred because of precipitation of Ti<sub>2</sub>Ag and TiAg intermetallics [8].

CP-Ti, known as one of the most important biomedical material, exhibit poor wear properties [9]. Therefore, many researchers suggested that alloying of Ti with copper and silver can increase tribological properties, while antibacterial properties still remain [10-13]. In our previous study [14] different content of Cu addition in Ti-5Al-2.5Fe was investigated and the results showed that Cu addition affects mechanical properties in a small range whilst still showing good



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antibacterial properties and cytotoxicity. In this study, the effect of Ag addition in Ti-5Al-2.5Fe alloy was studied. Hot pressing was used to consolidate initial powders. This technique, as a fast sintering route of powder metallurgy, provides some advantages such as better mechanical properties and easy control the intermetallic formation in a shorter time at lower temperatures [15]. The sintered samples were examined and the results discussed in detail.

## 2. Materials and Experimental Works

The scanning electron microscopy images (SEM) of the commercially pure titanium, aluminum, iron, and silver powders used in the current study are shown in Fig. 1. The elemental powders were poured into the Ø 85 mm x 70 mm cylindrical stainless steel jar to yield a nominal composition of Ti-5Al-2.5FexAg alloys and mechanically mixed by high-energy ball milling for 10 h with a rotation speed of 400 rpm under 10<sup>-2</sup> mbar vacuum atmosphere. Prepared powder mixtures were placed inside cylindrical graphite dies with a diameter of 20 mm and sintered at 950 °C for 30 min under 50 MPa using a uniaxial hot press (DIEX Corp.). Hot pressing cycle is given in Fig. 2. 10<sup>-4</sup> mbar vacuum atmosphere was used during all the sintering process to prevent the oxidation of sintered samples.

As can be seen from the sintering cycle, three different holding times at different temperatures (550, 650, and 750 °C) were used to prevent the escape of liquid aluminum from the graphite die during pressure assisted sintering and to ensure a homogeneous elemental diffusion [16-18]. The final dimensions of the produced compacts were 20 mm in diameter and 4



Figure 1. SEM images of powders (a) Ti, (b) Al, (c) Fe, (d) Ag

mm in height. Following sintering, the density of the sintered samples was measured using Archimedes' method. Vickers hardness test was performed using a Future-Tech Vickers hardness tester under 10 kg loads for 10 s loading duration. Three-point bending tests were applied with 0.5 mm/min cross-head speed.

All samples were metallographically prepared using 600, 1000, and 2000 SiC papers, polished with 9, 6, 3, and 1 µm diamond solution, and then etched with Kroll's reagent for 10 s. The microstructure of the etched samples was examined using an optical microscope. Phase analysis was carried out by XRD using a Rigaku diffractometer. The diffraction patterns were measured with 40 kV acceleration voltage, 20 mA current and Cu K $\alpha$  radiation (= 1.544 Å) in the scanning angle 2  $\theta$  range from 10° to 80°. The step size was 0.05° 2 $\theta$  with a scan rate of 2°/min. The detected peaks were indexed according to the inorganic crystal structure database (ICSD) and the International Centre for Diffraction Data (ICDD) Powder Diffraction File (PDF-2) databases.



Figure 2. Sintering cycle during hot pressing

To investigate the effect of the Ag content on the wear resistance of the Ti5Al2.5Fe alloy, wear tests were performed at room temperature under dry sliding conditions. Dry sliding wear tests were conducted using a ball-on-disk setup (Nanovea MT/60/NI) according to the ASTM G99 standard against 52100 steel ball bearings (6 mm in diameter) as a counterbody in the tests. The sliding speed of 0.13 m s<sup>-1</sup> and distance of 500 m were kept constant during all the sliding tests. The applied load used in the wear tests was 25 N.

The equation below was used to obtain the wear rate of the specimens:

$$W = M \rho^{-1} D^{-1} \qquad ...(1)$$



where W is the wear rate (mm<sup>3</sup> m<sup>-1</sup>), M denotes mass loss (g), and  $\rho$  (g.mm<sup>-3</sup>) and D (m) are the density and sliding distance, respectively [19]. SEM type JEOL 6060 was used to investigate the worn and fracture surfaces of the sintered samples.

#### 3. Results and Discussion

Fig. 3 shows the optical microscope images of the Ti-5Al-2,5Fe alloy with 1, 3, and 5 Ag (wt. %) addition. Typical basket weave ( $\alpha$ + $\beta$ ) structure consisted of lamellar  $\alpha$  phase with a fine  $\beta$  lath in all samples after production process. As it could be seen by the Figure 3(a), coarse plate-like  $\alpha$  phase dominated in Ti-5Al-2,5Fe alloy. Meanwhile, 1 wt.% Ag addition to alloy changed a bit structure to finer  $\alpha$  plates. With the increasing of Ag amount to 3 and 5 wt.%, coarse  $\alpha$  plates became more finer and thinner; however, the  $\beta$  phase amount in the structure increased because of  $\beta$ -stabilizing effect of the Ag element (Figure 3(c-d)). Similarly, Xiao et al., found that increasing Ag addition decreased thickness of  $\alpha$  phase at boundaries of primary  $\beta$  grains [7].



**Figure 3.** Optical images of Ti-5Al-2.5Fe alloys with (a) 0, (b) 1, (c) 3, and (d) 5 wt.% of Ag

Fig. 4 shows the XRD patterns of sintered Ti-5Al-2.5Fe with different contents of Ag. Reference sample Ti-5Al-2.5Fe consisted of main α-Ti (ICSD-98-005-2522) and β-Ti (ICSD-98-016-8322) phases together with intermetallic phases Ti<sub>3</sub>Al (ICSD-98-005-8188) and Ti<sub>2</sub>Al<sub>5</sub> (ICSD-98-010-6253). Jia et al. [20] also reported the presence of the detected phases such as  $\alpha$  phase, Ti<sub>3</sub>Al, and a small amount of transformed  $\beta$  phase in the Ti-5Al-2.5Fe alloy sintered at 1250 °C. With addition of 1 wt.% Ag, pure Ag (ICSD-98-005-6269) and intermetallic Ag<sub>3</sub>Al (ICDD-00-028-0033) were detected and Ti<sub>2</sub>Al<sub>5</sub> disappeared in 22 and 24° angles but formed again in 62° angle. Results showed that the addition of Ag increased  $\beta$ phase in the alloy and unresolved pure Ag was detected. Observation of pure Ag phase indicates that reaction between Ag and other elements was not completed. Increasing Ag addition to 3 and 5 wt.%, did not change the types of phases but they appeared in new angles. Xiao et al. [7], examined Ti-5Al-4Mo-4V alloy with addition of different content of Ag. The XRD results showed that the addition of Ag increased  $\beta$  structure in the alloy and it affected mechanical properties. Moreover, Daoush et al. [21], confirmed that increasing content of  $\beta$  phase can be attributed to the lower hardness results. In addition, the presented microstructure in Fig. 3 shows that the Ag addition to Ti-5Al-2.5Fe alloy increase the amount  $\beta$  phase, which is in agreement with the results of the XRD analysis.



Figure 4. XRD patterns of Ti–5Al-2.5Fe alloys with different Ag content

Fig. 5 shows the density measurement results as a function of Ag content added to Ti-5Al-2.5Fe alloy. The density of silver is relatively higher than titanium and its alloys have. Therefore, the final density values of the produced compacts increased gradually with increasing Ag content. This result is similar to Xiao et al. [7] study as they added 0, 2, 5, and 10 wt.% Ag to Ti-5Al-4Mo-4V alloy. They found that Archimedes' density increased with the addition of increasing content of Ag. Kikuchi et al. [22], showed that addition of increasing amounts of Ag (5, 10, 20, and 30 wt.%) increased the final densities of the material. In this study, the densities of the produced compacts have a good correlation with theoretical densities of the desired compositions. In addition to the final densities of the produced alloys, there is a small decrease that has been observed in the relative densities, this may be due to the change of the crystal structure of the titanium alloy from HCP to BCC and may be due to the formation different intermetallics within the final compact after sintering. Silver is also a beta stabilizing element in titanium alloys. HCP alpha ( $\alpha$ ) titanium has densely packed atoms compared to BCC titanium atoms [23].





Figure 5. Density of Ti–5Al-2.5Fe alloys as function of Ag addition

Fig. 6 shows that the addition of Ag to the titanium alloy caused an intensive difference in hardness values. While the hardness value in the main alloy was 250 HV, the addition of 1 wt.% Ag increased the hardness value to 322 HV. However the addition of more Ag content (3 wt.%) did not change the hardness. With 5 wt.% Ag addition, a small reduction in hardness was observed but still remained higher than main alloy equal to 293 HV. Chen et al. [24] alloyed Ti with 1, 3, and 5 wt.% Ag and sintered the samples under 15-30 MPa pressure in 900 °C with a hot press. They also observed that the addition of increased amount of Ag decreased the hardness of the final material. Szaraniec et al. [25], studied powder metallurgical production of Ti alloys with varying silver content (3.5; 5; 10; and 20 at.%). Their results showed small reductions in hardness observed between 3.5 and 5 at.% Ag addition.

Fig. 7 shows the bending tests results. The results show that the addition of Ag increased bending strength and displacement. This result can be correlated to the hardness results [26]. In the reference sample, bending result showed the lowest (1190 MPa) bending strength and Ti-5Al-2.5Fe-5Ag showed the



Figure 6. Hardness measurement of Ti-5Al-2.5Fe-xAg

highest bending strength and displacement (1750 MPa and 1.3 mm). Similarly to XRD results, that could be the reason for the increase of  $\beta$  phases in the structure [27]. It could also explain the results obtained for hardness, which was continuously reduced with Ag addition, whereas bending strength and elongation have improved.



Figure 7. Bending strength–deflection profiles of Ti–5Al-2.5Fe and Ti–5Al-2.5Fe-xAg alloys

SEM images of fracture surfaces after bending test can be seen in Fig. 8. Fracture surface of the Ti5Al2.5Fe alloy as a reference sample consisted of brittle and ductile fracture modes. Bright areas showing brittle fracture and dimples showing ductile behavior can be clearly seen in Fig. 8a. It was found that the main fracture mechanism for the reference samples is a transgranular fracture. Similar mixture of fracture modes was reported in literature for Ti-based alloys [28, 29]. Brittle behavior of the reference



Figure 8. SEM images of fractured surfaces after bending test of (a) Ti-5Al-2.5Fe, (b) Ti-5Al-2.5Fe-1Ag, (c) Ti-5Al-2.5Fe-3Ag, (d) Ti-5Al-2.5Fe-5Ag alloys



sample was also supported by the bending test results, as shown in Fig. 7, showing lower displacement compared to the Ti5Al2.5Fe-xAg alloys. The addition of silver to the reference alloy changed the fracture mode from mainly brittle behavior to the combination of brittle and ductile fracture (Fig. 8b-d). Planar faces and small dimples are seen on the Fig. 8b while elongated dimples can be seen on Fig. 8c-d. Fig. 8b, shows that 1 wt.% Ag addition caused more homogenous distribution of equiaxed dimples between cleavage facets, compared to reference sample. Increasing the amount of Ag addition to 3 wt.% enhanced cleavage facets and homogenous distribution in the structure. The sample with 5 wt.% Ag addition exhibited more rough dimples and less cleavage facets. According to Shi et al. [30], the main difference between fracture characteristic is related to plasticity.

Fig. 9 shows the wear rates of produced samples. Ti-5Al-2.5Fe showed the lowest wear rate among the sintered samples. With the addition of 1 and 3 wt.% Ag, a small increase in wear rate was observed. However, the addition of more Ag content (5 wt.%) resulted in a reduction in the wear rate. It is important to note that; highest wear rate was observed in Ti-5Al-2.5Fe-3Ag alloy which showed the most unresolved Ag peak, according to XRD results. As is mentioned before, hardness results of 1 and 3 wt.% Ag added samples were the same, but there was a big difference in wear rate between the two samples. This is in agreement with the research of Lee et al. [31], who confirmed that there was not a direct relationship between hardness and tribological properties. Similar results were also reported about the hardness/wear relationship [13, 32]. Such phenomenon can be explained by the fact that wear process is a complex combination of multiple factors while the surface hardness presents only one of those factors.

SEM images of worn surfaces of the produced alloys are shown in Fig. 10. Typical adhesive and



Figure 9. Variation of wear rate according to Ag addition in Ti-5Al-2.5Fe alloy

abrasive wear mechanisms were dominant in all samples. In Fig. 10a, abrasive wear indicating grooves and adhesion, which caused material transfer, were observed in the sliding direction. Sampiao et al. [33] suggested that the main reason for the groove interruption could be due to the formation of abrasive particles caused by three-body abrasion. Based on that study, the newly created abrasive particles roll on the surface and scratch it in a limited zone. Fig. 10b consists of long abrasive grooves between ridges, material transfer caused debris, and plowing zones. Lee et al. [34], showed that the plowing mechanism consisted of pushing material to the side of the grooves during sliding. Fig. 10c shows noticeable amount of wear debris accumulation, which are most wear loss according wear rate results given in Fig. 9. High wear rate zones and deep abrasive grooves were observed from Fig 10d, which shows that both the abrasive and adhesive mechanisms worked instantaneously. Moreover, Yang et al. [35], found materials transfer in a very small load condition such as 5N during investigation the wear properties of a Ti-6Al-4V alloy.

## 4. Conclusions

1. The sintered Ti-5Al-2.5Fe-xAg alloys exhibited a typical basket weave  $(\alpha+\beta)$  structure in all the samples after production process.

2. Addition of 1 wt.% Ag increased hardness but the addition of more Ag caused a small decrease in hardness results.

3. A significant improvement in bending strength was obtained with Ag addition.



Figure 10. SEM images of worn surfaces of (a) Ti-5Al-2.5Fe, (b) Ti-5Al-2.5Fe-1Ag, (c) Ti-5Al-2.5Fe-3Ag and (d) Ti-5Al-2.5Fe-5Ag



4. Fractography images showed rough surfaces, indicating cleavage and ductile cracked regions.

5. The elongated dimples structure of the facture behavior of the alloys increased with increasing silver content in the alloy.

6. Wear rate increased with 1 and 3 wt.% Ag addition, however, 5 wt.% Ag addition showed a slight decrease. This result is associated with the occurrence of unresolved Ag in the structure and these results were supported by the XRD analysis.

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## UTICAJ SADRŽAJA SREBRA NA HABANJE I MEHANIČKE OSOBINE KOD Ti-5Al-2.5Fe-xAg LEGURA DOBIJENIH METALURGIJOM PRAHA

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## Apstrakt

U ovom radu je ispitivan uticaj srebra na mehaničke osobine Ti5Al2.5Fe legure. Legura Ti5Al2.5Fe sa različitim sadržajima srebra, od 1 do 5 wt. %, dobijena je mehaničkim mešanjem, a zatim je podvrgnuta vrućem presovanju na temperaturi od 950 oC, u periodu od 15 minuta i pod pritiskom od 50 MPa. Praškasto telo dobijeno kompresijom je podvrgnuto tri puta postupku fiksiranja da bi se potisnula tečna faza unutar grafitnog kalupa pre nego što se postigne maksimalna temperatura sinterovanja. Sinterovani uzorci su podvrgnuti testovima za ispitivanje čvrstoće, savijanja i habanja da bi se ispitao uticaj srebra na mehaničke osobine Ti5Al2.5Fe legure. Optičkim i skenirajućim elektronskim mikroskopom je urađena mikrostrukturna karakterizacija. Rezultati su pokazali da Ag ima diferencijalnu ulogu kada su u pitanju mehaničke osobine podržane mikrostrukturnim komponentama. Čvrstoća na savijanje i tvrdoća dobijenih uzoraka se povećala kada se dodalo srebro, a zatim se tvrdoća legure smanjila prilikom povećanja sadržaja Ag, ali je i dalje bila veća od tvrdoće Ti5Al2.5Fe legure. Test habanja je takođe pokazao slične rezultate kao i test tvrdoće. Na kraju se došlo do zaključka da optimalni sadržaj Ag za Ti5Al2.5Fe leguru iznosi 1 wt.%. XRD analiza je pokazala da je nerastvoreni sadržaj Ag glavni uzrok smanjenja mehaničkih osobina.

Ključne reči: Ti5Al2.5Fe legura; Sadržaj srebra; Mikrostruktura; Habanje

