

Effect of milling speed on the formation of Ti-6Al-4V via mechanical alloying

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Abstract. Ti-6Al-4V alloys are well known for good corrosion resistance, excellent surface oxide biocompatibility and high specific strength. This paper investigates the influence of milling parameters on the formation of Ti-6Al-4V alloy processed using mechanical alloying. A mixture of CP Ti and master alloy (60Al-40V) powder were milled using a high energy ball mill (HEBM) in order to produce a mechanically alloyed Ti-6Al-4V alloy powder. Milling was carried out at a constant milling time of 5 hours at varying speeds of 800, 900, 1000 and 1100 rpm in order to investigate the influence of speed on the formation of Ti-6Al-4V alloy. Scanning electron microscopy with energy dispersive analysis (EDX) and X-ray diffraction (XRD) were performed on the mechanically alloyed powder to investigate the chemical homogeneity and the formation of alpha (α) and beta (β) phase during the mechanical alloying of CP Ti and 60Al-40V.

1. Introduction

Titanium and its alloys have been widely used for aerospace and biomedical applications due to its low modulus of elasticity, excellent corrosion resistance, and high strength to weight ratio, good biocompatibility and attractive mechanical properties [1–6]. The most commonly used Ti alloy is the Ti-6Al-4V alloy which contains aluminium and vanadium as major alloying elements. They fall in the two phase ($\alpha + \beta$) region where aluminium acts as an alpha (α) stabilizer and vanadium as a beta (β) stabilizer [1,3,6]. The formation of α and β phase leads to the attainment of a variety of microstructures and property combinations in the alloy.

Ti-6Al-4V alloy has been usually produced using the conventional melting and casting process routes, followed by thermomechanical processes such as forging and extrusion. These process routes are associated with problems such as high costs, high energy consumption and excessive loss of material [3–7]. Recently, it has been shown that the abovementioned problems can be overcome by using powder metallurgy and solid state production techniques [3]. Powder metallurgy methods such as mechanical alloying have become a viable and promising route for cost effective fabrication of Ti alloys. The mechanical alloying process involves the transfer of material by repeated deformation, cold welding, fracturing of blended powder particles in a high energy ball mill to produce an alloy through highly energetic ball collisions [3,8]. This process is capable of producing nanosized structures, metallic alloys (high temperature resistant, high entropy), composites (metals-polymers-oxides), nano-crystallines (alloys & composites), and high temperature alloys [3,8]. Therefore the current study aims to investigate the influence of milling parameters on the formation of the Ti-6Al-4V alloy powder produced via mechanical alloying.

2. Experimental Procedure

2.1. Starting materials

The starting materials were CP Ti powder (99.5 wt. %) and the master alloy containing (60 wt. % Al - 40 wt. % Ti) according to suppliers. The titanium powder was ASTM Ti Grade 2 quality with particles of 100 mesh (-150 μm) particle size range. The master alloy powder had a particle size of -45 μm .

2.2. Method

CP Ti and master alloy (60Al-40V) powders were mixed in the proportions of 90% CP Ti and 10% master alloy. The mixture was milled using the Simoloyer high energy ball mill (HEBM), using a high strength stainless steel jar in order to produce a mechanically alloyed Ti-6Al-4V alloy powder. The mill charge was composed of 100g powder blend and 2000 g 5 mm diameter 100Cr6 stainless steel milling balls giving a ball to powder ratio of 20:1. Milling was carried out at speeds of 800, 900, 1000 and 1100 rpm with constant milling duration of 5 hours in order to investigate the effect of milling speeds on the formation of the Ti-6Al-4V alloy. Scanning electron microscopy (SEM) with energy dispersive analysis (EDS) was used to study the morphology and chemical analysis using a JEOL® JSM-6510 instrument with a tungsten electron gun. X-Ray powder diffraction analysis was carried out in a PAN analytical X-Pert X-Ray diffractometer with Cu K α radiation ($\lambda = 1.5421 \text{ \AA}$). Several powder samples were analysed over the 2θ range of 5 – 90°, with a scanning step size of $2\theta = 0.026^\circ$, and a scan rate of 58 seconds/step. The resulting diffraction patterns were analysed using X'Pert-HighScore® analysis software package.

3. Results and Discussion

Figure 1 shows micrographs of CP Ti and 60Al40V master alloy powder in the as- received condition after mixing (Figure 1). Individual powders had fine and coarse particles.

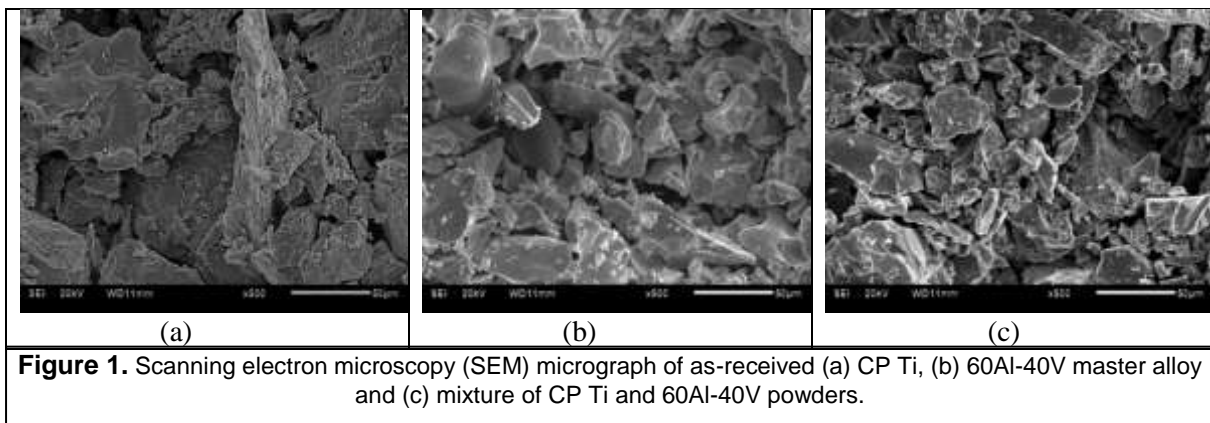


Figure 2 shows micrographs of the mechanical alloyed powder at different speeds of 800, 900 and 1000 and 1100 rpm using a constant milling duration of 5 hours. It is worth noting in figure 2 that with varying speeds there were no significant changes taking place in the powder so this might mean that the change of speed had no effect on the formation of the alloy. The Ti-6Al-4V alloy powder was already formed at a speed of 800 rpm with a milling duration of 5 hours and this was confirmed by the EDS results in Figure 3 and in Table 1. EDS analysis was done on a few powder particles of different milling speeds in order to confirm the formation of Ti-6Al-4V alloy. Table 1 shows the EDS results of the powder particles at different speeds. It can be seen that with all the different milling speeds, there was no significant change in the chemical composition of the powder and Ti-6Al-4V alloy had formed.

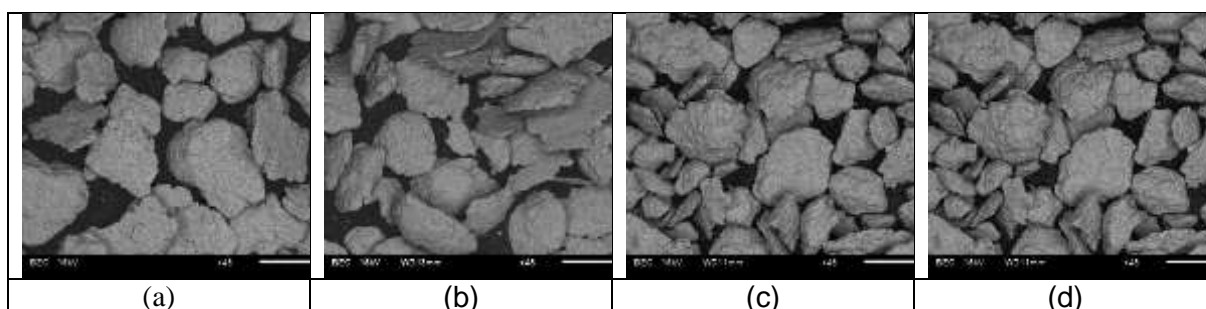


Figure 2. SEM micrographs after milling Ti and master alloy at a constant ball to powder ratio (20:1), milling duration (5 hours) and different speeds of (a) 600rpm, (b) 800rpm, (c) 1000 rpm and (d) 1100rpm.

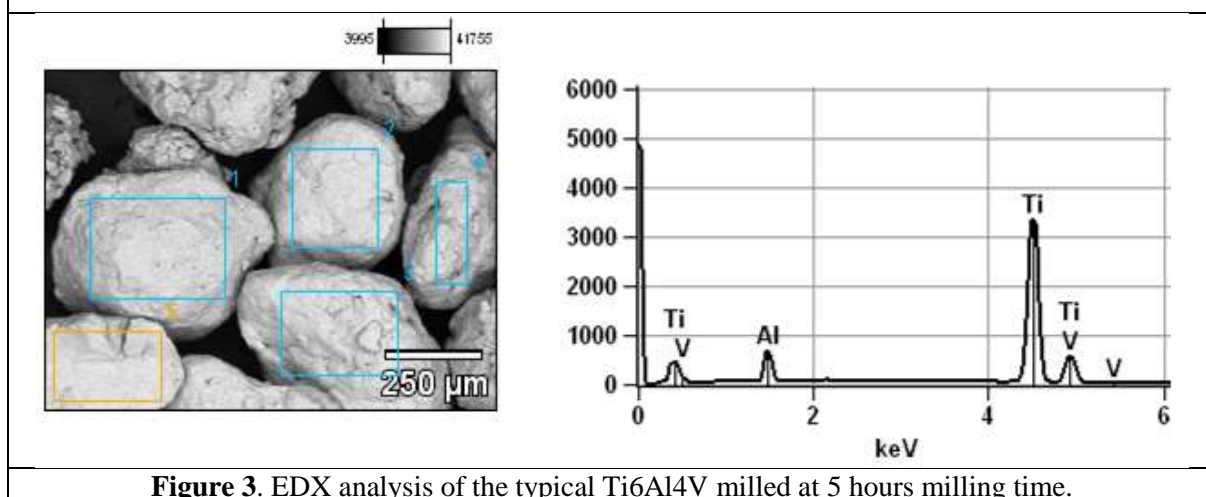


Figure 3. EDX analysis of the typical Ti6Al4V milled at 5 hours milling time.

Table 1. EDS analysis results in wt.% of the as-mixed and mechanically alloyed powders of Ti-6Al-4V for 5 hours at varying speeds of 800, 900, 1000, 1100 rpm.

Elements	As-mixed	800 rpm	900 rpm	1000 rpm	1100 rpm
Ti (wt. %)	90	90.84	89.4	91.25	89.03
Al (wt. %)	6	5.73	6.70	5.47	6.32
V (wt. %)	4	3.43	3.90	3.28	4.65

The X-Ray diffraction (XRD) analysis of the milled CP Ti and 60Al-40V master alloy is shown in Figure 4. XRD patterns of Ti-6Al-4V before mechanical alloying, standard Ti-6Al-4V alloy and after mechanical alloying for 5 hours using different milling speeds are revealed. Mechanically alloyed samples' XRD results were compared with standard Ti6Al4V XRD to confirm the formation of the alloy produced using mechanical alloying. It is worth noting that after 5 hours of milling, Ti peaks had broadened whereas Al and V peaks had disappeared with all different milling speeds.

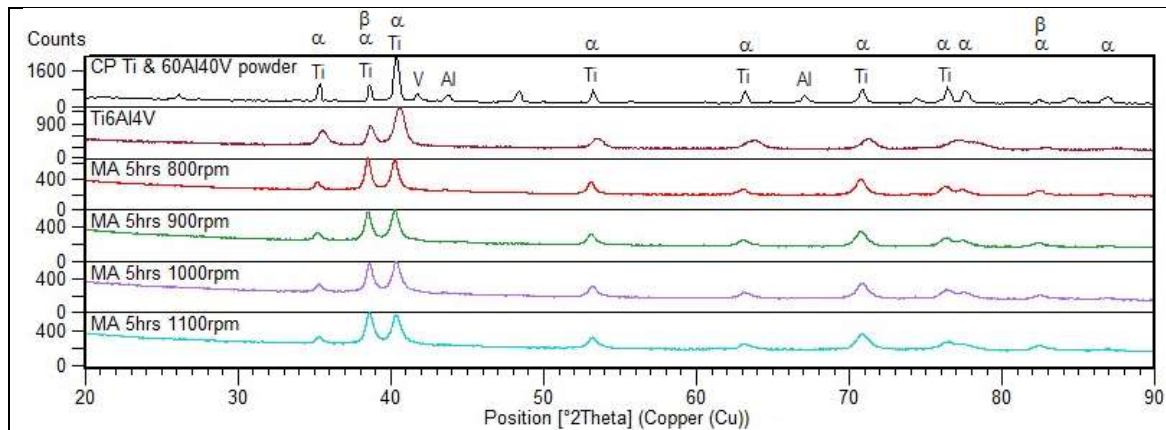


Figure 4. XRD results of the (a) CP Ti and 60Al40V powder, (b) standard Ti6Al4V alloy and mechanically alloyed CP Ti and master alloy for 5 hours at different speeds of (c) 800 rpm, (d) 900 rpm, (e) 1000 rpm and (f) 1100 rpm.

4. Discussion

Milling CP Ti and 60Al-40V master led to the formation of Al rich phase and V rich phase as revealed by the EDX analysis (Figure 3 and Table 1). This is a result of the chemical segregation caused by the high energy milling process. The repeated cold welding and fracturing of particles and increased density of crystalline defects and dislocations leads to the diffusion of Al and V which also led to the broadening of the mechanically alloyed XRD peaks [3]. Aluminium and vanadium dissolve in the Ti and forms a homogeneous Ti (Al, V) solid solution or phase depending on Al or V content.

The Al and V rich phases are revealed by the XRD analysis (Figure 3) to be α -phase and β -phase respectively. Increasing the rotational speed proportionally increases the kinetic energy input in the ball milling, which was expected to affect the formation of α and β -phase. However, varying milling speeds within the range of 800 to 1100 rpm, evidently is not affecting the formation of α and β -phase.

5. Conclusions

A mixture of CP Ti and 60Al-40V powder were milled using a high energy ball mill at speed range of 800 to 1100 rpm and at constant time of 5 hours. The following conclusions were drawn:

- The high energy milling process may lead to the formation of α -phase (hcp) and β -phase (bcc) which are stabilized aluminium and vanadium respectively.
- The formation of Ti6Al4V is not affected by the milling speed within the rotation range of 800 to 1100 rpm
- Ti-6Al-4V alloy can be produced using high energy ball milling process.

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Reviewers' Comments

The layout of the Abstract needs to be revised	Done
Caption of Figure 2, Figure 3 and Table 1 need minor revisions	It was revised accordingly
Few things need be corrected. Please go through the guidelines again.	Corrected according to the guidelines
Image (figure 2b) is blurry.	The image was changed
It would have been a little interesting to include micrographs of the as-received materials before mixing	Micrographs of the individual powders have been inserted
Please check the grammatical errors.	Checked
What was the aim of mixing the CP Ti and master alloy before milling? What mixer was used?	No mixing equipment was used. The powders were mixed to make 90% CpTi and 10% 60Al40V.
Basic guidelines were not followed.	This was rectified.
However, need to point out the Ti and master alloy powders in Figure 1	Micrographs of individual powders have been used
Also, the choice of milling speed was not motivated. Milling speed affects the kinetic energy impacted on the powder particles - but it is not clear that kinetic energy from the different speeds used is statistically differentiated. Milling also leads to a broadening of the XRD peaks: the authors have not explained why it happened for their powders.	The broadening of XRD peaks due to milling has been addressed that its due to repeated cold welding and etc
Explain whether PCA was used during milling. Provide particle size distribution, yield, oxygen content, and iron contamination of the milled powders.	Some processing techniques might not be revealed in this paper. The main focus of the paper is to investigate the effect of milling speed on the formation of Ti64. No iron contamination was found on the powders.