Development and Characterization of Nanostructured Ti6Al4V Alloy

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Nanostructured Ti6Al4V alloy was successfully formed through mechanical alloying of a stoichiometric mixture of Ti, Al and V powders. Phase evolutions of the milled powders were investigated by XRD. The Ti(Al,V) solid solution formed after 10 h milling. Prolongation of the milling process up to 20 h lead to a structure consisting of the β-Ti phase in the α-Ti(Al) matrix. The nanostructured Ti6Al4V alloy had a grain size and hardness of ~20 nm and ~600 Hv respectively. Thermal stability of the alloyed powder was good as it's hardness increased to ~950 Hv after 5 hr heat treatment at 1100 °C.

Keywords: Hardness, Heat Treatment, Nanostructure, Solid Solution, Ti6Al4V Alloy.

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1. INTRODUCTION

Titanium alloys primarily stand out due to two major properties: high specific strength and excellent corrosion resistance. This also explains their preferential use in the aerospace, the chemical industry and medical engineering sector [1]. Usually titanium alloys are classified as α, α + β and β alloys. The α + β alloys are the most widely used alloy group [1]. Among the α + β alloys, Ti-6Al-4V is by far the most popular titanium alloy. The aerospace industry is the largest user of Ti-6Al-4V alloy due to the good balance of its properties [1]. Despite all the mentioned advantages, the Ti6Al4V alloy still suffers from poor tribological properties, poor oxidation behavior and low hardness.

The properties of titanium alloys are essentially determined by two factors: the chemical composition and the microstructure [1-3]. The type of phases present, grain size and grain shape, morphology and distribution of the fine microstructure determine the properties and therefore the application of titanium alloys [3]. Reig et al. [4] analysed the effect of Ti6Al4V microsphere size on the mechanical properties of porous Ti6Al4V specimens developed by sintering. Based on this research, optimum mechanical properties were obtained when sintering the smallest size microspheres. Titanium alloys with smaller grain size unexpectedly exhibit more stable thermal behaviour upon heating [5]. According to Mahboubi et al. [6], higher microhardness was obtained for the as-milled nanostructured Ti6Al4V powder in comparison to the raw Ti6Al4V alloy. Thus, production of nanostructured titanium alloys could possibly benefit by yielding improved thermal stability at high working temperatures, good erosion behavior, good wear resistance and good fracture toughness.

Casting routes for the production of titanium and its alloys, with high levels of vulnerability to oxidation, have always been much troublesome [1]. Solid state synthesis of titanium alloys is thus of practical importance. Mechanical milling (MM) is a cost effective solid state route for the production of nanosized powders [7, 8].

In the present study solid state synthesis of nanostructured Ti6Al4V alloy during mechanical alloying of elemental powders and the formation mechanism were investigated.

2. EXPERIMENTAL PROCEDURE

Ball milling of titanium, aluminium and vanadium elemental powders was conducted under protective atmosphere of Ar (99.999 % pure) using a planetary (Retsch PM200 type) ball milling machine. The grinding media is made of stainless steel. Milling parameters are given in Table 1.

Table 1 – The milling parameters used for the process.

Rotation speed of vial(rpm)	500
Vial material	Hardened Cr steel
Vial capacity(ml)	120
Ball material	Hardened C steel
Diameter of balls(mm)	20
Number of balls	5
Ball to powder weight ratio	10:1
Total powder mass(g)	17

Sampling was done from milled materials at selected time intervals to study the phase evolution using Philips Xpert MPD x-ray diffraction (XRD) machine fitted with Cu kα radiation ($\lambda = 1.54$ Å), and 0.02 step size scanned from 20 to 90° (2θ). Changes of the grain size and strain of the samples were revealed by use of the Williamson-Hall formula (Equation 1) [9, 10]:

$$\beta\cos\theta = 0.9\lambda/D + \varepsilon\sin\theta \qquad (1)$$

where β is the diffraction peak width at half maximum intensity, λ the wave length of the radiation used (1.54 Å), D the grain size, θ the Bragg diffraction angle and ε is the internal lattice strain. This strain can be estimated from the linear slope of βcosθ versus sinθ, and the grain size can be estimated from the intersect of this line at sinθ = 0.

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3. RESULT AND DISCUSSION

X-ray diffraction patterns of Ti-6wt %Al-4wt %V powder milled for 0, 5, 10, 15 and 20 hr are shown in Fig. 1.
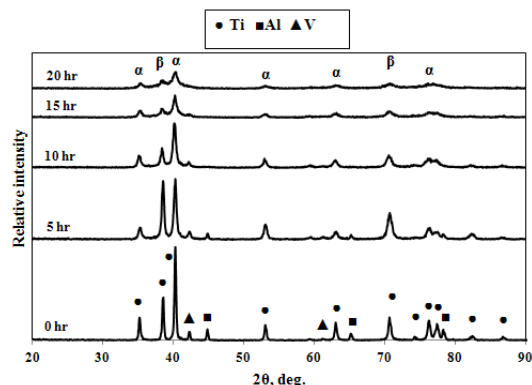

Fig. 1 – XRD patterns of Ti-6wt%Al-4wt%V powder at different milling times

By increasing the milling time, the continuous decrease in the intensities of diffraction peaks occurred. After 10 hr, the diffraction peaks of both Al and V disappeared in XRD pattern. These could be due to gradual diffusion of Al and V atoms in to the Ti atomic structure, since Al and V have enough solid state solubility in Ti in contrast to zero solubility of Ti in Al and V [11, 12]. Previous investigations have also called for Al atoms' diffusion in Ti lattice structure as the cause for Al diffraction peaks' disappearance after 4 hr of ball milling [13]. Trends of Ti peak shift are especially depicted for Ti(101) peak before and after 10 hr of milling (Fig. 2). This shift must be caused by solution of one element in Ti. Before 10 hr, the shift in Ti peaks towards lower angles occurs due to the diffusion of Al (r_{Al} = 1.82 Å) and V (r_V = 1.92 Å) atoms in to Ti (r_{Ti} = 2 Å) interstitial spaces, and a metastable Ti(Al,V) solid solution is formed in this way (Fig. 2.a). Beyond 10 h, the Ti(Al,V) diffraction peaks shift to higher angles due to increase of V content in Ti(Al,V) as well as simultaneous homogenous distribution of V atoms in the solid solution structure (Fig. 2.b). By the end of this shift, α-Ti(002) and α-Ti(103) diffraction peaks are replaced with β-Ti(110) and β-Ti(211) peaks, subsequently and the stabilization of β-Ti phase in the primary α-Ti matrix is thus guaranteed. The lattice parameter of the new β-Ti phase is identical to the literature values and corresponds to the bcc structure with a = 3.23 Å. Peak broadening can be indicative of small crystallite size and increased lattice strains [14]. However, stress gradients and/or chemical heterogeneities can also cause peak broadening [14].

In order to assure the effect of Al and V atoms' dissolution on Ti peak shift, pure Titanium's (101) peak position at different milling times was also investigated and no shift occurred (Fig. 3). No Fe contamination was detected after EDAX analysis. Solid state solubility of Al and V in Ti could thus be the only cause for the shift of Ti peaks in this research. Thus, after 20 hr milling, the solid state in-situ synthesis of nanostructured Ti6Al4V alloy with grain size and lattice strain of about 20 nm and 0.08 % respectively was obtained.
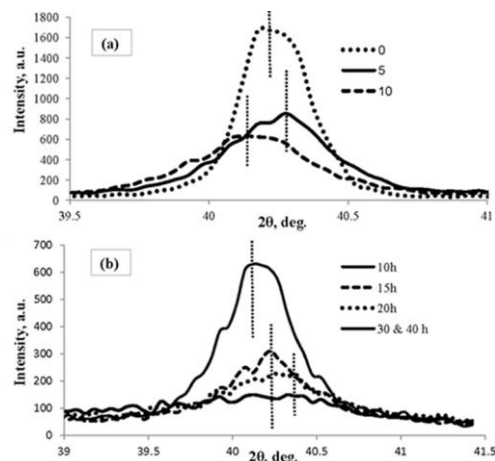

Fig. 2 – Ti(101) peak shift towards: a. lower angles for milling times less than 10 hr, b. higher angles for milling times more than 10 hr
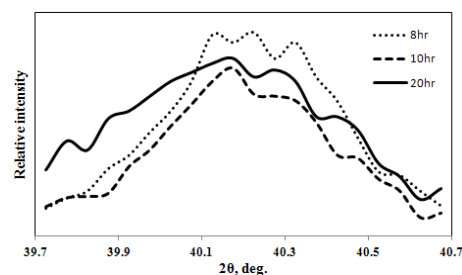

Fig. 3 – stable position of pure Ti(101) peak before and after 10 h of ball milling

The nanostructured alloy had a hardness of ∼ 600 Hv which increased to ∼ 950 Hv through 5 hr heat treatment at 1100 °C. During the quench from solution temperature, both the decomposition of the transformed β phase or the precipitation of aluminium rich α₂ in the primary alpha phase could cause such an increase in hardness [15]. Thus, the nanostructured Ti6Al4V alloy has good thermal stability as well as high hardness. Continuation of milling for 110 hr yielded an amorphous structure (Fig. 4).
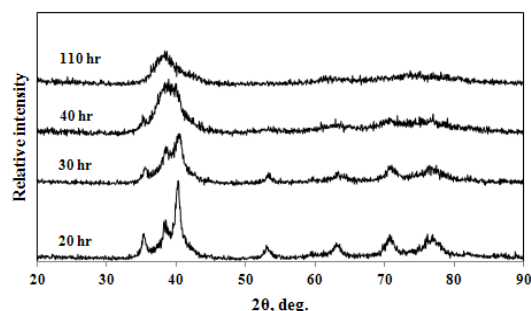

Fig. 4 – The Ti-6wt %Al-4wt %V powder diffraction patterns obtained through different milling times after the Ti₆Al₄V alloy formation

After 110 hr milling, the stable position of the peak of the amorphous phase indicates that no secondary amorphous phase has been formed (Fig. 5).
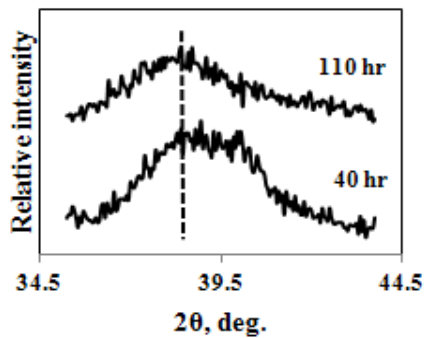
Fig. 5 – Stable peak position of the amorphous phase after 110 hr milling

4. CONCLUSION

The nanostructured Ti6Al4V alloy with grain size of about 20 nm was successfully produced through 20 h of mechanical alloying of elemental powders. The alloy's response to 5 hr heat treatment at 1100 ˚C was an increase in hardness from ~ 600 Hv to ~ 950 Hv. This good thermal stability of the alloy could be assigned to decomposition of the β phase or precipitation of the aluminium rich α_2 phase in the primary α matrix.

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