Preparation and Characterization of Ti-10Mo Alloy by Mechanical Alloying

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Abstract In the present study, Ti-10Mo alloy was fabricated by mechanical alloying and subsequently consolidated by powder metallurgy techniques. The structural evolution of the powder blends with various milling durations, and the sintered alloys with different holding times, were characterized by x-ray diffraction, scanning electron microscopy, and light microscopy. The results revealed that the size of powder particles decreased with increasing milling time. In addition, only the sintered alloy using powders milled for 5 h exhibited both α -phase and β -phase. When the milling time increased to greater than 40 h, TiN phase gradually formed, which suppressed the α -phase to β -phase transformation. Furthermore, the effects of the sinter holding time on the Vickers hardness and the corrosion behavior of Ti-10Mo alloys was also investigated. The results showed that the Vickers hardness increased with increasing sinter holding time; after sintering the alloy for 5 h, the samples exhibited acceptable corrosion resistance.

Keywords Ti-10Mo alloy · Mechanical alloying · Vickers hardness · Corrosion behavior

Introduction

Titanium and its alloys are widely used in biomedical applications, because of their low specific weight, low elastic modulus, high specific strength, excellent biocompatibility, and high corrosion resistance [1, 2]. Pure

titanium (CP Ti), Ti-6Al-4V alloys, TiNi alloys, and Ti-6Al-7Nb alloys have been successfully employed in clinical applications [3]. Unfortunately, previous studies [4, 5] have reported that the release of aluminum and particularly vanadium ions from the alloy might cause health problems, such as neurologic disorders, osteomalacia, and senile dementia. Other research studies have called attention to the possibility of Ni-hypersensitivity and toxicity [6, 7]. Therefore, it is of particular interest to develop non-toxic element Ti-based alloys, such as Ti-Nb [8–10], Ti-Hf [11], Ti-Ta [12], Ti-Mo [13–15], and Ti-Zr [16].

In recent years, Ti-Mo alloys have received particular attention for use as biomaterials. The advantage of Mo is that this element has strong β -stabilizing properties in titanium, in comparison with Ta or Nb. It has been reported that crystal structures of Ti-Mo alloys are sensitive to the Mo concentration and a minimum content of 10 wt% is needed to stabilize β phase in Ti-Mo alloys at room temperature [17]. Ti-10Mo alloy can exhibit the dual phase $\alpha + \beta$ microstructure, which offers a combination of excellent ductility and strength. Furthermore, Ti-10Mo alloys have been found to possess superior corrosion resistance compared with Ti-Ni alloys [18], and Mo is a favorable non-toxic alloying element in Ti alloys for biomedical applications.

Conventional titanium alloys are made through melting followed by casting and subsequent thermo-mechanical treatments [19, 20]. However, it is very difficult to achieve compositional homogeneity in this approach. Mechanical alloying is a potentially attractive technique for alloy preparation as it can exhibit superior homogeneity compared with conventional melting routes [21, 22]. In the present study, Ti-10Mo alloy powder was prepared by mechanical alloying and consolidated by powder metallurgy techniques. The structural evolution of the powder

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blends milled for various durations was characterized by x-ray diffraction (XRD) and scanning electron microscopy. Furthermore, light microscopy was conducted to characterize the corresponding sintered alloys. The Vickers hardness and the corrosion behavior of the sintered Ti-10Mo alloys were investigated as function of holding time.

Materials and Methods

Elemental metal powders of Ti (purity > 99.9%, particle size < 45 μm) and Mo (purity > 99.99%, particle size < 10 μm) were blended at Ti: Mo weight ratio of 9:1. The powders were blended in a planetary ball milling system with zirconia containers and balls for 120 h, with a ball-to-powder weight ratio equal to 20:1 and rotation rate of 300 rpm. The ball milling was carried out in a high-purity argon atmosphere. During milling, powder samples were removed at 5, 20, 40, 60, and 120 h, allowing for characterization of the powder morphology and assessment of the mechanical alloying process. In general, temperature would rise during the milling process, depending on the milling type, milling speed, and milling operation. To reduce heating, the mill was operated cyclically, with 60 min of milling followed by 30 min of idle time. After milling for a total of 5 h, the powder mixture was cold compressed under 500 MPa for 1 min to form a green compact with a diameter of 10 mm and a length of approximately 10 mm. The green compacts were sintered at 1200 °C in a vacuum furnace for 2, 5, and 10 h followed by cooling in the furnace. The designation of the sintering alloy samples is given in Table 1.

The changes in particle morphology during milling were examined using a scanning electron microscope (SEM). Powder samples with milled for various times were characterized by XRD using Cu radiation. The samples were scanned from 30° to 70° with a scan speed of 4°/min. Light microscopy was conducted to observe the microstructure of the sintered alloy samples. The sintered alloy samples were wet ground with waterproof silicon carbide papers to 2000 grit, mirror polished by standard metallographic

techniques, and then etched with a solution of 5% HF, 10% HNO_3 , and 85% H_2O (by volume). Vickers hardness of the sintered alloys was measured at room temperature. Average hardness values were obtained from ten indents at a load of 100 g and a dwell time of 5 s. Electrochemical experiments were conducted in a standard three-electrode cell with 0.785 cm^2 of exposed area in the working electrode, having a platinum counter electrode and a saturated calomel reference electrode (SCE). Working electrolyte was simulated body fluid (1.5 SBF). All experiments were carried out at 37 °C. Before immersion, the specimens electrodes were polished with 1500 grit silicon carbide paper and covered with epoxy resin so that only one metal surface was in contact with the 1.5 SBF. Tafel scans were conducted from –500 to 500 mV at a scan rate of 1 mV/s. The corrosion potential and corrosion density were obtained from the Tafel analysis.

Results and Discussion

SEM Analysis

Figure 1 shows the morphology of the raw Ti and Mo mixture powders. The starting powders exhibited irregular morphology and inhomogeneous size distribution. The morphology and the change of the particle size of the milled powders with different milling times are illustrated in Fig. 2. After milling for 5 h (see Fig. 2a), the particles were more regular in shape compared with the starting powders. As the milling time increased from 5 to 20 h, the shape of the particles was round and flat as a result of continuous cold welding and fracturing during ball milling. The morphology of the largest particles is shown in Fig. 2(b), while smaller particles were also visible. There

Fig. 1 SEM images of the raw Ti and Mo mixture powders

Table 1 The designation of the sintering alloy samples in this study

Sample no	Milling time, h	Sintering temperature, °C	Holding time, h
Ti-10Mo-2	5	1200	2
Ti-10Mo-5	5	1200	5
Ti-10Mo-10	5	1200	10

Fig. 2 SEM images of Ti-10Mo powders with different milling times: (a) 5 h, (b) 20 h (c) 40 h, and (d) 60 h

was a significant scatter in particle size. At this milling time, the number of the big particles was always larger than that of the small particles. This indicates that welding of particles was predominant over fracturing during ball milling [23]. When the milling time increased to 40 h (see Fig. 2c), the size of particles decreased greatly. The average size was approximately 1–2 μm , and the shape changed into a more spheroidal type. However, certain large particles also existed, indicating that welding was still occurring. After continuous milling for 60 h (see Fig. 2d), the particles became finer and homogeneous and retained their granular morphology. The size of particles ranged between 200 and 500 nm. It was observed that the size of particles decreased and their shape gradually became spherical because of mechanical milling.

XRD Analysis

Figure 3 shows the XRD patterns of Ti-10Mo powder after milling for several times. It can be seen that the peaks of Ti and Mo gradually broaden and decrease in intensity with increasing milling time. This is caused by the refinement of the crystallite size, which occurs during the mechanical alloying process. In addition, the Ti peaks shifted toward higher diffraction angles, which indicated that some Mo atoms had dissolved into Ti lattice during milling and to form a Ti (Mo) solid solution. The XRD pattern of powders milled for 5 h revealed the presence of individual elemental Ti with a hexagonal close packed (hcp) structure and elemental Mo with a cubic structure (Fig. 3). After milling for 40 h, peaks for both Mo and Ti broadened because of the refinement of the grains and introduction of the internal strain. Furthermore, the TiN phase begins to appear at this time. The N contamination may result from

(1) N existing in the as-received titanium; or (2) adsorption and/or absorption of N during the exposure of the milled powder to air. During mechanical milling, the remaining air in the powders served as a source of reaction gas. As a result, this may have led to the formation of titanium nitride. This phenomenon had been observed by other researchers [24, 25].

The change of position and shape of Ti (1 0 1) diffraction peaks at the selected milling times are shown in greater detail in Fig. 4. The Ti peak was observed to shift toward higher diffraction angles, which indicates the dissolution of Mo into Ti. As the atomic radius of Mo is less than that of Ti, lattice contraction of Ti occurred during the mechanical alloying, resulting in the observed shift of the titanium peak to higher angles [26].

In general, during the mechanical alloying, Ti and Mo peaks became broader, and their intensities decreased. The broadening of the peak was due to the refinement of powders and the strains associated with intense plastic deformation in powder particles. The positions of Ti peaks moved to larger angles indicating that the lattice parameter of the Ti alloy had changed. This phenomenon was also observed in other mechanically alloyed alloys [23, 27–29] and was considered to be typical for the mechanical alloying process. Furthermore, after mechanical alloying only α -phase Ti formed, while the expected β -phase Ti did not appear.

Figure 5 shows the XRD patterns of Ti-10Mo sintered from powders milled for various times. In the sintered alloy using powders milled for 5 h (see Fig. 5a), no elemental Mo could be detected, indicating that the complete transformation occurred. Both hcp α -phase Ti and expected bcc β -phase Ti were observed. In the sintered alloy using powders milled for 40 h (see Fig. 5b), peaks for α -phase

Fig. 3 XRD patterns of Ti-10Mo powders with different milling times

Fig. 4 Change of position and shape of diffraction peaks after selected milling times Ti (1 0 1)

Fig. 5 XRD patterns of Ti-10Mo sintered alloy from different milled powders: (a) 5 h, (b) 40 h, and (c) 120 h

are observed along with peaks for the α'' and TiN phases, while the peak for β -phase disappeared. It was obvious from these data that the peak for TiN in the sintered alloy using powders milled for 120 h was stronger than observed for the sintered alloy using powders milled for 40 h (compare Fig. 5b and c). This might be ascribed to the fact that the longer milling time intensified the formation of TiN phase in the powder. This observation was agreement with XRD results shown in Fig. 3 for the as-milled powders. Furthermore, it was observed that the alloyed sintered from powders milled for 120 h was composed of α -Ti and TiN without the β -Ti phase (Fig. 5c). This might be because as the mixture powder milling time increased, the occurrence of TiN suppressed α to β phase transformation during the sintering process. As a result, the β -Ti phase of the sintered alloy gradually disappeared with increasing powder milling time. In general, the Ti-10Mo alloys with dual phases ($\alpha + \beta$ phase) exhibit excellent properties. Therefore, the sintered Ti-10Mo alloy using powders milled for 5 h, which was the only sample in this study to exhibit both the α and the β phases, was selected for further analysis.

Structural Features and Morphology of Sintered Alloy

The microstructures of the Ti-10Mo-2, Ti-10Mo-5, and Ti-10Mo-10 alloys (where 2, 5, and 10 refer to the hold time for sintering) are very similar; an example of a typical microstructure for the Ti-10Mo-5 alloy is shown in Fig. 6. Figure 6a, b shows light micrographs of the sintered Ti-10Mo alloy. The alloy had a fine-grained microstructure with an inhomogeneous size distribution, and β phase was dispersed on the matrix- α phase. It could be deduced from these figures that the content of β phase was a little less

Fig. 6 Light micrographs of Ti-10Mo after the sintering treatment: (a) low magnification, and (b) high magnification

than that of α phase, which was in agreement with the results of XRD (see Fig. 5a). In addition, some pores were observed; the density of pressed and annealed samples ranged from 97 to 98% of the theoretical density. Furthermore, it was noted that most of the pores were formed near the β phases. These pores consisted of a mixture of a small closed porosity and a large interconnected opened porosity of approximately 10 μm in size. There were two main reasons for the presence of these pores in the sintered alloy. First, according to the Ti-Mo phase diagram [30], no liquid phase could be formed at the sintering temperature. Hence, solid–solid diffusion reaction of Ti-Mo powder compacts was the dominant sintering mechanism. However, the diffusivity of Mo in Ti was much lower than that of the self-diffusion of Ti, and this would slow down the inter-diffusivity of Ti-Mo alloys. The slow diffusion rate of Mo in Ti at the sintering stage indicates that grain boundary migration was restricted during sintering, resulting in relatively substantial amount of sintered pores [31]. Second, the powder sintering technique is known to be susceptible to the formation of Kirkendall pores when the sintering

Fig. 7 Polarization curves of Ti-10Mo alloy samples in 1.5 SBF with pH value 7.4 at 37 °C

powder is compacted [32] and hence leads to the formation of residual pores in the compact. The light micrographs confirmed that the porosity rate in the alloy prepared by mechanical alloying was slightly higher than that prepared using the conventional methods.

Vickers Hardness

Vickers hardness of the bulk Ti-10Mo alloy sintered for different holding times was measured. There was a trend of increasing Vickers hardness with increasing holding time. The average Vickers hardness for the Ti-10Mo-2, Ti-10Mo-5, and Ti-10Mo-10 samples was 426, 427, and 473 HV, respectively. The change in hardness for the samples with hold times between the 2 and 5 h is negligible, but the large increase for the sample of the 10-h hold time is significant. This difference could be ascribed to the increased content of β phase that was observed for the 10-h-sintered sample. As mentioned previously, the solid–solid diffusion reaction of Ti–Mo powder compacts was the dominant sintering mechanism according to the Ti–Mo phase diagram [30]. A longer sintering time would provide a longer time for diffusion of Mo into the Ti lattice, which could then accelerate the α to β phase transformation. As a result, the content of β phase would increase as the holding time was increased from 5 to 10 h. In general, the hardness of β phase was higher than that of α phase. Hence, the Vickers hardness increased as the holding time increased from 5 h to 10 h.

Note that the hardness value of the bulk Ti-10Mo alloy was about 2.4 times higher than that of the pure titanium alloy (200 HV) [33]. Pure Ti has low resistance to plastic shearing and low work hardening; the enhancement in hardness of Ti with Mo addition could improve the resistance to the plastic shearing. The Ti-10Mo alloys

Table 2 Corrosion current density and corrosion potential for Ti-10Mo alloy samples

Samples	E_{corr} , V	I_{corr} , $\mu\text{A}/\text{cm}^2$
Ti-10Mo-2	−0.280	0.08
Ti-10Mo-5	−0.213	0.07
Ti-10Mo-10	−0.286	0.07

fabricated by mechanical alloying possessed sufficient hardness to make them potential candidates for artificial joint alloys.

Corrosion Behavior

Figure 7 shows the polarization curves of the Ti-10Mo alloy sintered for different holding times. After the Tafel region, broad and distinctive passivation regions are observed in the anodic polarization curves of all the samples. The polarization curves of these samples had wide passive region, indicating excellent corrosion resistance. It was noted that the holding time had little influence on corrosion behavior of the samples. It was clearly seen that the passivation current density of the samples increased in the following sequence: Ti-10Mo-5 to Ti-10Mo-2 to Ti-10Mo-10. The corrosion current density (I_{corr}) measured from the Tafel extrapolations, as well as the corrosion potential (E_{corr}) are shown in Table 2. The corrosion test results indicated that the holding time had little effect on the E_{corr} . The E_{corr} of the samples increased from −0.286 to −0.280 to −0.214 V as the holding time was changed from 10 to 2 to 5 h, respectively. These observations suggested that the alloy sintered at 5 h had the lowest corrosion tendency. The I_{corr} values were 0.08, 0.07, and 0.07 $\mu\text{A}/\text{cm}^2$, for the Ti-10Mo-2, Ti-10Mo-5, and Ti-10Mo-10 samples, respectively. The corrosion rates of the samples were very low. These results indicate that a passive film formed on the Ti-10Mo alloys was quite stable with minimal ion release to the electrolyte. According to the above analyses, it was inferred that the Ti-10Mo 5 sample had the best corrosion resistance among all the samples.

Conclusions

Ti-10Mo alloy was successfully prepared by mechanical alloying and then consolidated by powder metallurgy technique. The influence of milling time on material composition and morphology of the mixture alloy powder was investigated. In addition, the effect of the sinter holding time on the Vickers hardness and the corrosion

behavior of the Ti-10Mo alloy were also investigated. The primary results are summarized as follows:

- (1) With an increase in milling time, the crystallite size of mechanically alloyed Ti-Mo decreased. The steady-state phase exhibited a crystallite size of 200–500 nm. The particles could be reduced to nanometer after long milling time.
- (2) The XRD and light microscopy results confirmed that only the sintered Ti-10Mo alloy using powders milled for 5 h were composed of the dual phase: α -Ti and β -Ti. As the milling time increased to 40 h for more, the formation of TiN occurred in the powders and the sintered alloys, which suppressed the α -phase to β -phase transformation.
- (3) The Vickers hardness increased as the holding time increased because of the longer time available for Mo diffusion to occur. The hardness of the bulk Ti-10Mo alloys was about 2.4 times higher than that of the pure titanium alloy.
- (4) Electrochemical experiments revealed that the sintered Ti-10Mo alloy with the holding time at 5 h had the best corrosion resistance in all the samples.

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