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Microstructure, mechanical properties and dry wear resistance of β-type Ti−15Mo−xNb alloys for biomedical applications

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Abstract: In order to study the effect of element Nb on the microstructure and properties of the biomedical β-type Ti−Mo based alloys, Ti−15Mo−xNb (x=5, 10, 15 and 20 in %) alloys were investigated. The dry wear resistance of β-type Ti−15Mo−xNb alloys against Gr15 ball was investigated on CJS111A ball-disk wear instrument. Experimental results indicate that crystal structure and morphology of the Ti−15Mo−xNb alloys are sensitive to their Nb contents. Ti−15Mo−xNb alloys match those for β phase peaks and no any phases are found. The Vickers hardness values of all the Ti−15Mo−xNb alloys are higher than HV200. The compression yield strength of the Ti−15Mo−5Nb alloy is the lowest and that of the Ti−15Mo−10Nb alloy is the highest. For all the Ti−15Mo−xNb alloys, the friction coefficient is not constant but takes a higher value. In dry condition, SEM study reveals deep parallel scars on the wear surfaces of all the Ti−15Mo−xNb alloys under different loads. The friction coefficient of the Ti−15Mo−5Nb alloy under 1 N is the lowest. The wear principal mechanism for Ti−15Mo−xNb alloys is adhesive wear.
Key words: β-type Ti−Mo−Nb alloys; wear resistance; adhesive wear; microstructure; mechanical properties

1 Introduction

Due to their many advantageous properties, the application of commercially pure titanium and titanium alloys in the medical fields has dramatically increased over the last few decades. The superior qualities of these alloys, such as low specific gravity, high corrosion resistance, adequate mechanical properties, and good biocompatibility are all highly desirable for biomedical materials [1−3]. Commercially pure (CP) titanium is insufficient for high-stress applications，because of low strength, difficulty in polishing, and poor wear resistance [4]. Because of its higher strength with sufficient corrosion resistance, Ti−6Al−4V alloy has been tested as a replacement for CP−Ti. However, the cytotoxicity of elemental vanadium is questionable [5,6]. Subsequently, Ti−6Al−7Nb alloy has developed as a biomedical application material. The mechanical properties of Ti−6Al−7Nb alloy are quite similar to Ti−6Al−4V alloy. The biocompatibility of Ti−6Al−7Nb alloy advantage is over Ti−6Al−4V owing the absence of vanadium, but it still contains aluminum [5−7].

The preferred titanium alloy for biomedical applications should have low modulus of elasticity, excellent mechanical strength, corrosion resistance, formability, and no potential toxic elements. As a result, Nb, Ta, Mo, Zr and Sn are selected as the safest alloying elements to titanium [7−9]. Thus, Ti alloys such as Ti−Mo and Ti−Nb alloys have been extensively investigated as biomedical materials due to their low elastic modulus, high biocompatibility and non-toxicity [10,11]. Recently, a number of studies have focused on the development of non-toxic and low elastic modulus β or near-β type titanium alloys for biomedical applications, such as Ti−Mo based alloys [12,13]. The different studies focused on the phase transformations, stress release, and mechanical properties of Ti–Mo based alloys, but there are only a few studies about their wear characteristics [13−15]. Therefore, Ti–Mo–X alloys composed of non-toxic elements such as Zr, Nb, Ta and Sn with lower elastic modulus and higher strength should

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be developed. It is of academic interest to investigate the effect of these elements on the microstructure and properties of the Ti–Mo based alloys. In the present study, the microstructure and properties of a series of Ti−15Mo−*x*Nb alloys with Nb contents ranging from 5% to 20% were investigated. The dry wear resistance of Ti−15Mo−*x*Nb alloys against Gr15 ball was investigated on CJS111A ball-disk wear instrument.

2 Experimental

Ti−15Mo−*x*Nb alloys with four concentrations of niobium (x=5, 10, 15 and 20 in mass fraction, %) were selected for this study. All the materials were prepared from raw titanium (99.8%), molybdenum (99.9%), and niobium (99.9%) by using a commercial arc-melting vacuum-pressure casting system. The melting chamber was first evacuated and purged with argon. Appropriate amounts of metals were melted in a copper hearth with a tungsten electrode. The ingots, of approximately 30 g each, were re-melted thrice to improve their chemical homogeneity.

The specimens were cut from as-cast ingots by electrospark-erosion. The dimensions of specimen were d12 mm×5 mm. The specimens for observing microstructure were sanded on SiC paper (grade from 240 to 1200), finished by polishing with diamond powder and eroded with 8%HF+15%HNO_3+77%H_2O. The solidification microstructures were observed by an OLYMPUS BH2-UMA optical microscope (OM). The Vickers hardness measurements were measured using a HV−5 Vickers tester with a load of 49 N for 30 s. Compression testing was conducted at room temperature with a rate 0.5 mm/min on a INSTRON 5500 compression instrument. The dimensions of specimen were d4 mm×6.5 mm.

The dry sliding wear tests were carried out on a CJS111A ball-disk wear instrument, by using Gr15 ball as a counterface material (3 mm in radius and hardness HV750). The specimens for the experiments (disks 12 mm in diameter and 2 mm in thickness) were cut from the castings. The experimental parameters were estimated as follows: dry condition, normal load 1 N and 2 N, test-disk rate 100 r/min, test-disk wear radius 3.5 mm. Friction coefficient was automatically recorded as a function of cycle time. Wear scars were examined by SEM.

3 Results and discussion

3.1 Microstructure and mechanical properties of Ti−15Mo−*x*Nb alloys

The microstructures of the Ti−15Mo−*x*Nb alloys are shown in Fig. 1. The equiaxed crystal grain was observed. The effect of Nb content on the microstructures of the Ti−15Mo−*x*Nb alloys is obvious. With the Nb content increasing, the sizes of equiaxed crystal grain of the Ti−15Mo−*x*Nb alloys are obviously decreased. Figure 2 shows the XRD patterns of the series of Ti−15Mo−*x*Nb alloys. For the Ti−15Mo−*x*Nb alloys,

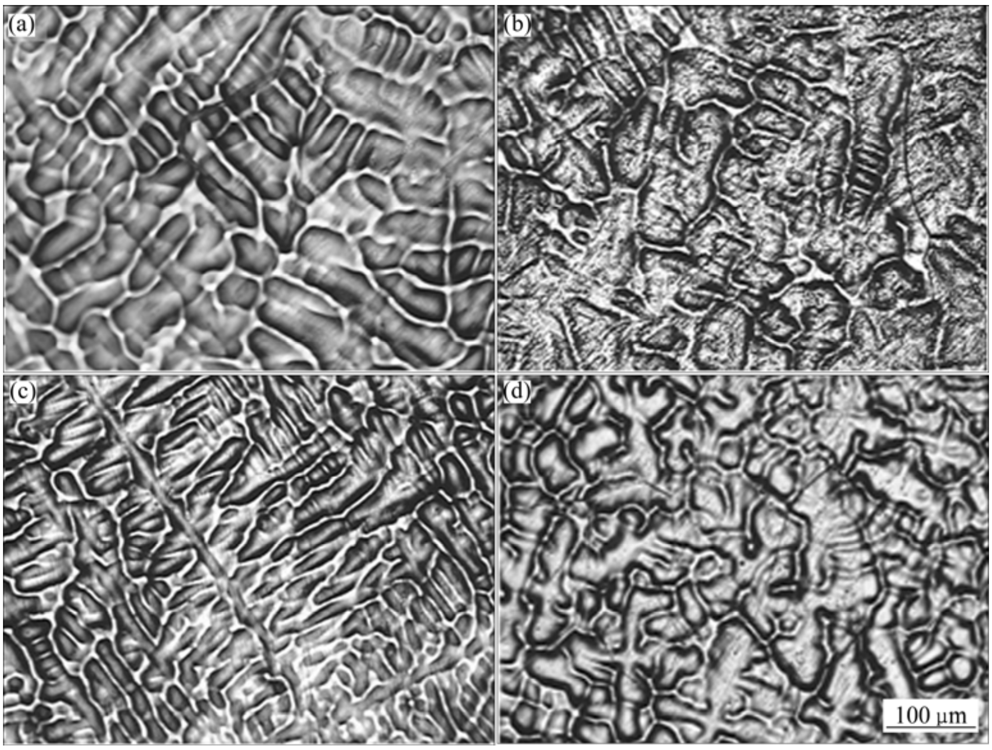

Fig. 1 Microstructures of Ti−15Mo−*x*Nb alloys: (a) Ti−15Mo−5Nb; (b) Ti−15Mo−10Nb; (c) Ti−15Mo−15Nb; (d) Ti−15Mo−20Nb

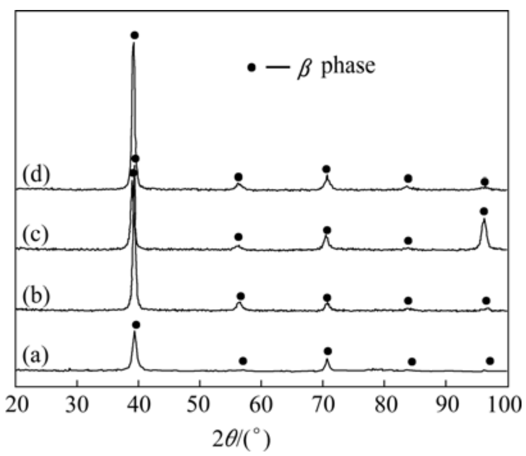

Fig. 2 XRD patterns of alloys: (a) Ti−15Mo−5Nb; (b) Ti−15Mo−10Nb; (c)Ti−15Mo−15Nb; (d) Ti−15Mo−20Nb

all the diffraction peaks matched β phase. There was no indication of α-phase peaks or any intermediate phases were included in any of the present XRD patterns.

Figure 3 shows the room-temperature compression stress—strain curves of the Ti−15Mo−*x*Nb alloys. During the compression test, fracture did not happen for all the Ti−15Mo−*x*Nb alloys. The mechanical characteristics of Ti−15Mo−*x*Nb alloys obtained by compression and Vickers hardness tests are summarized in Table 1. The compression yield strength of the Ti−15Mo−5Nb alloy is the lowest and that of the Ti−15Mo−10Nb alloy is the highest. The Vickers hardness values of all the Ti−15Mo−*x*Nb alloys are higher than HV200. The Vickers hardness value of the Ti−15Mo−10Nb alloy is the highest (HV257) and that of the Ti−15Mo−20Nb is the lowest (HV208). In a study by CHEN et al [11], the Vickers hardness values of Ti−15Mo alloy after centrifugal casting into graphite mold is HV381 and the compression yield strength of the Ti−15Mo is 1221 MPa. The Vickers hardness values and the compression yield strength are higher than those of

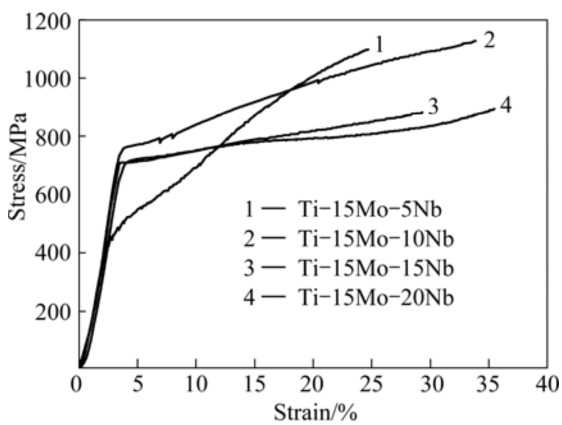

Fig. 3 Room-temperature compression stress—strain curves of Ti−15Mo−*x*Nb alloys

Table 1 Main properties of Ti−15Mo−*x*Nb alloys

Alloy	Phase	Vickers hardness(HV)	Compression yield strength/MPa
Ti−15Mo−5Nb	β	246	473
Ti−15Mo−10Nb	β	257	756
Ti−15Mo−15Nb	β	235	702
Ti−15Mo−20Nb	B	208	710

the Ti−15Mo−*x*Nb alloys. This result may be associated with the molten Ti−15Mo alloy pressed into graphite mold and the microstructure of the Ti−15Mo alloy is finer. Therefore, other evaluations of the Ti−15Mo−*x*Nb alloy will be performed in a future study, including assessments of its microstructure and mechanical properties after centrifugal casting into graphite mold.

3.2 Dry wear properties of Ti−15Mo−*x*Nb alloys

Variation of friction coefficients of the Ti−15Mo−*x*Nb alloys against Gr15 ball under different loads with the test lasting time is shown in Fig. 4. For all the Ti−15Mo−*x*Nb alloys, the friction coefficient was not constant but took a higher value. In dry condition, at the early stage the alloy structure is slightly pressed. In the second stage, some particles gradually detach at the two edges of contact, more and more particles are detached. The friction coefficient of the Ti−15Mo−5Nb under 1 N is lower than that under 2 N. The effect of load on the friction coefficients of the other three alloys is not obvious. Among all of the Ti−15Mo−*x*Nb alloys, the friction coefficient of the Ti−15Mo−5Nb alloy under 1 N is the lowest.

Figures 5 and 6 show the SEM images of wear surfaces of the Ti−15Mo−*x*Nb alloys against Gr15 ball under different loads. In the present study, SEM examination of the wear surface in dry condition showed smeared wear surface of all the Ti−15Mo−*x*Nb alloys with ridged wear scars parallel to the sliding direction (grooving wear) and superficial plastic deformation along the direction of the sliding. The wear particles of all the Ti−15Mo−*x*Nb alloys presented aggregation during the wear tests.

Figure 7 shows the EDS energy spectrum analyses of wear surface of Ti−15Mo−20Nb alloy under 1 N and 2 N. The energy spectra of the Ti, Mo, Nb, Cr and Fe elements were observed. During the wear tests, the exfoliation of debris of Gr15 ball happened, which shows that Cr and Fe pick up from the counterface material by the wear debris during wear testing. LONG and RACK [16] reported that adhesive wear is the principal mechanism of dry wear of Ti–6Al–4V ELI and Ti–6Al–7Nb ELI alloys articulated against hardened steel. MAJUMDAR et al [17] reported that the dominating wear mechanism of heat-treated Ti–13Zr–13Nb

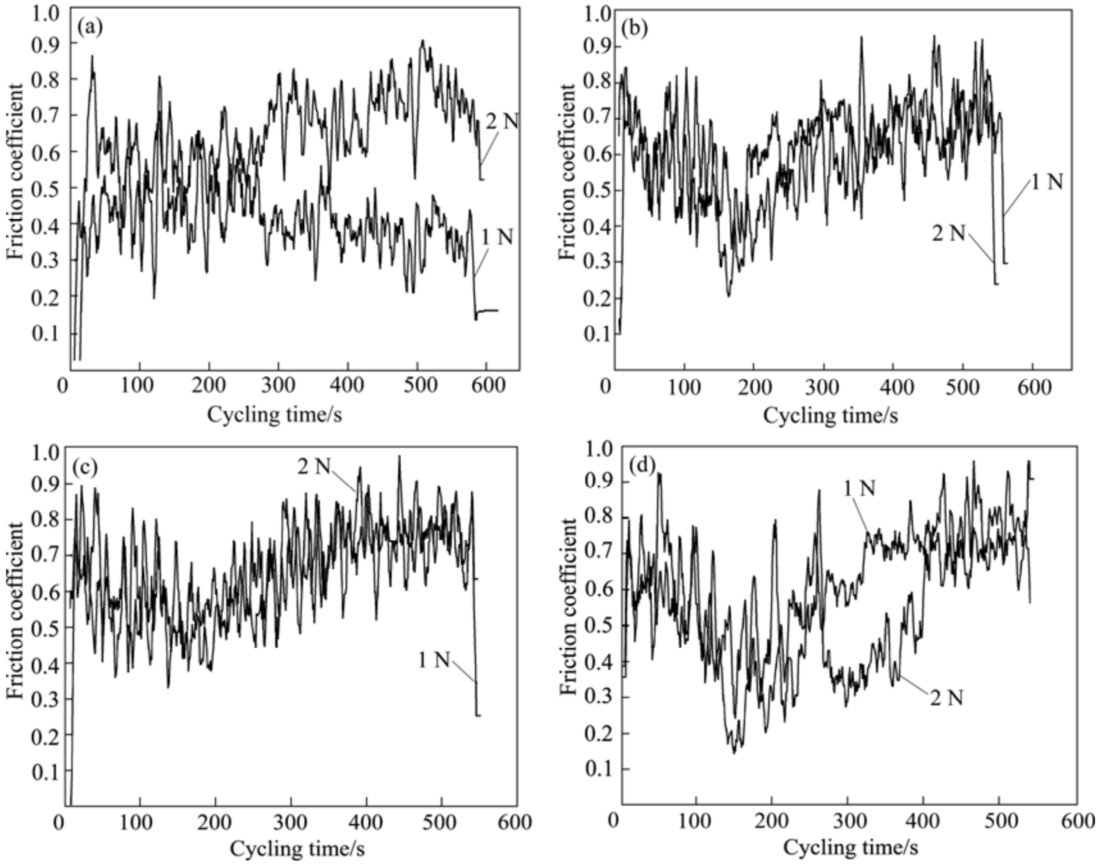

Fig. 4 Variation curves of friction coefficients of titanium alloys against Gr15 ball under different loads with test lasting time: (a) Ti−15Mo−5Nb; (b) Ti−15Mo−10Nb; (c) Ti−15Mo−15Nb; (d) Ti−15Mo−20Nb

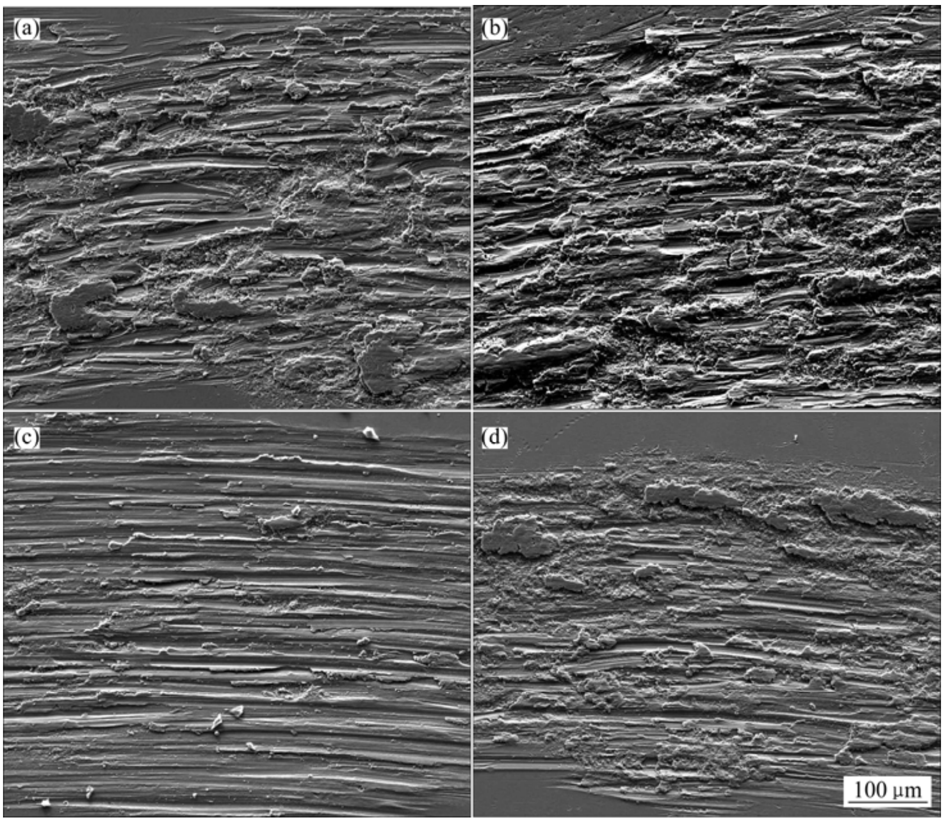

Fig. 5 SEM images of worn surfaces of Ti−15Mo−*x*Nb alloys against Gr15 ball under 1 N: (a) Ti−15Mo−5Nb; (b) Ti−15Mo−10Nb; (c) Ti−15Mo−15Nb; (d) Ti−15Mo−20Nb

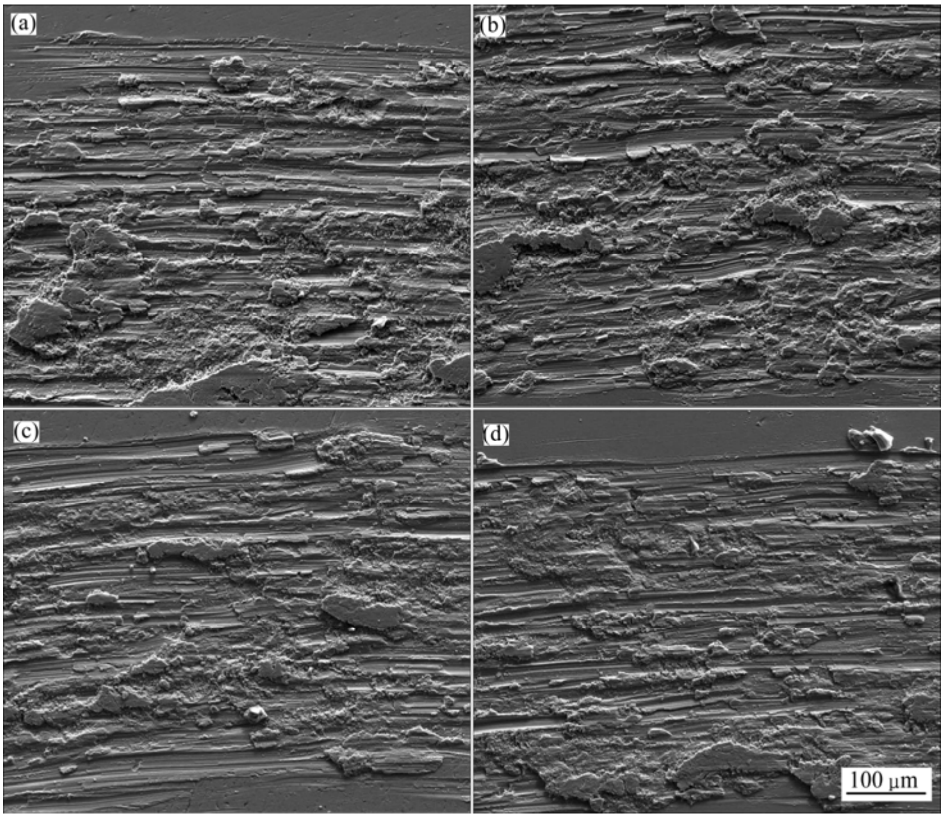

Fig. 6 SEM images of worn surfaces of Ti−15Mo−*x*Nb alloys against Gr15 ball under 2 N: (a) Ti−15Mo−5Nb; (b) Ti−15Mo−10Nb; (c) Ti−15Mo−15Nb; (d) Ti−15Mo−20Nb

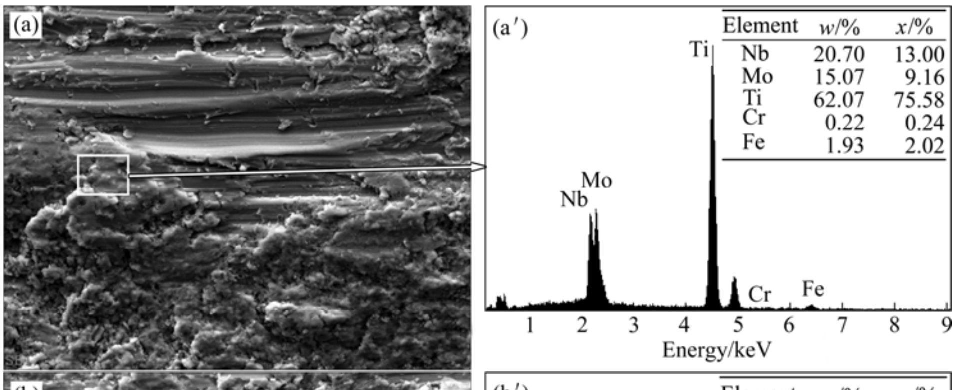

Fig. 7 SEM images and EDS energy spectrum analysis of worn surface of Ti−15Mo−20Nb alloy under different loads: (a) 1 N; (b) 2 N

alloy in dry condition was microcutting. In simulated body fluids, the abrasive scratches with transfer layer are in agreement with the observation of CHOUBEY et al [18]. As soon as the surface of the titanium alloy is exposed to environment it rapidly forms an oxide layer on the surface due to its high reactivity. The repassivation capacity of the surface layer during the wear process also plays a major role during the wear process, and the wear mechanism is mainly abrasive [17,19]. In the present study, the wear principal mechanism for Ti−15Mo−*x*Nb alloys is adhesive wear.

The wear resistance of biomedical materials is one of the important characteristics that must be studied. However, wear testing procedures are not standardized; many types of wear testing have been used [1]. Generally, around or cone-shaped specimen acting as a stylus opposes a flat specimen [20]. In general, metals with low theoretical tensile and shear strengths exhibit higher coefficients of friction than higher-strength materials [1]. An association between wear characteristics and hardness was reported that wear value decreased with increasing hardness. When the properties of titanium materials were evaluated by parameters obtained from indentation depth, depth was found to decrease with increasing content of impurities. The change in depth value is caused by the effect of yield strength and flow stress [21]. However, in previous studies of OHKUBO et al [1] on CP titanium and some titanium alloys, they were not able to find any correlations between their amount of wear and their strength data. They also found that there was no correlation between their bulk hardness and their wear. In addition, there are other factors for wear characteristics. Wear particles have influence by a surface-damage mechanism, and the surface condition was reported to be influential in the wear characteristics of dental-amalgam alloys [22,23]. In the present study, we can find the friction coefficient of the Ti−15Mo−5Nb alloy with low compression yield strength among Ti−15Mo−*x*Nb alloys is the lowest under 1 N, But, we also found that there was no correlation between their bulk hardness and their wear of Ti−15Mo−*x*Nb alloys. In the future study, we will focus on the correlations among wear, strength and bulk hardness of Ti−15Mo−*x*Nb alloys.

4 Conclusions

1) For the Ti−15Mo−*x*Nb alloys, only the β phases were observed in the XRD patterns. With the Nb content increasing, the sizes of equiaxed crystal grain of the Ti−15Mo−*x*Nb alloys are obviously decreased.

2) The Vickers hardness values of all the Ti−15Mo−*x*Nb alloys are higher than HV200. The compression yield strength of the Ti−15Mo−5Nb alloy is the lowest and that of the Ti−15Mo−10Nb alloy is the highest.

3) The friction coefficient of the Ti−15Mo−5Nb under 1 N is lower than that under 2 N. The effect of load on the friction coefficients of the other three alloys is not obvious. Among all of the Ti−15Mo−*x*Nb alloys, the friction coefficient of the Ti−15Mo−5Nb alloy under 1 N is the lowest. The wear principal mechanism for Ti−15Mo−*x*Nb alloys is adhesive wear.

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生物医用 *β* 型 Ti−15Mo−*x*Nb 合金的显微组织、力学性能和干态耐磨性

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摘 要：为了研究元素 Nb 含量对 *β* 型 Ti−Mo 基合金显微组织和性能的影响，制备了 4 种 Ti−15Mo−*x*Nb(*x*=5%, 10%, 15%和 20%)合金，并研究其显微组织和性能。采用 CJS111A 型球−盘式摩擦磨损试验机评价了 Ti−15Mo−*x*Nb 合金与 Gr15 对磨时的干摩擦磨损性能。结果表明：Nb 含量对 Ti−15Mo−*x*Nb 合金显微组织结构形态的影响较大，4 种合金主要由 *β* 相组成，其硬度值均高于 HV200；Ti−15Mo−5Nb 合金的压缩屈服强度最低，Ti−15Mo−10Nb 合金的压缩屈服强度最高；在干磨状态下 Ti−15Mo−*x*Nb 合金的摩擦因数不稳定但其值较高；在不同的加载条件下，磨损表面均出现了较深的平行磨痕，在加载载荷为 1 N 时 Ti−15Mo−5Nb 合金的摩擦因数最低；Ti−15Mo−*x*Nb 合金的磨损机制为粘着磨损。

关键词：*β* 型 Ti−Mo−Nb 合金；耐磨性；粘着磨损；显微组织；力学性能

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