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RESEARCH

Study of the Tribological Properties of Diffusion Coated NiTi Intermetallic on Cp Titanium

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A B S T R A C T

In this work, dry tribological behaviors of commercial pure (Cp) titanium and diffusion coated titanium were studied and compared. Wear tests were performed by a pin on disk tribometer using 52100 steel pins, under various loads of 10, 20, and 40 N. Worn surfaces were examined by scanning electron microscope, equipped with EDS analyzer. The wear rates of the coated materials were lower than those of the Cp titanium. This is mainly attributed to the higher hardness of the NiTi layer (about 280 HV) which is 1.5 times higher than that of the untreated titanium (about 180 HV). In addition, the SEM analysis of the worn surfaces revealed that the formation of tribological layer on the surface of the diffusion treated material effectively lowered the wear rate. The results also demonstrated a lower coefficient of friction in the treated specimens compared to those of the Cp materials.

1. INTRODUCTION

Despite the high specific strength and almost good corrosion resistance of titanium alloys [1,2], these materials exhibit poor wear resistance and high friction coefficient which restrict their application in items subjected to wearing conditions [3,4]. To overcome these deficiencies, several surface modification techniques like plasma nitriding, ion implantation, physical vapor deposition, liquid phase surface treatments, laser cladding, electroless, gas tungsten arc welding, and pulsed plasma electrolysis have been applied on these alloys [5–12]. The formation of diffusion coatings like nickel titanium compound (NiTi) has also drawn significant attention to combat wear [13-16]. NiTi intermetallic shows pseudoelasticity behavior [17,18] which is elastic responses to applied stresses, due to reversible martensitic phase transformation [19], and this leads to the superior hardness and wear resistance [20,21]. A number of research groups have employed various techniques to fabricate NiTi intermetallic as reliable protective layers on titanium and its alloys [22-24]. Tran et al. [15], studied the possibility of the formation of NiTi intermetallic phase via ultra-fast reaction of nickel droplet deposited by thermal spray on a titanium substrate. Waghmare et al. [23], fabricated a thick NiTi coating on Ti-6Al-4V by tungsten inert gas (TIG) surfacing method. The clad layer possessed a hardness value of more than 500 HV and the wear resistance improved up to 9.5 times compared to that of the substrate. Mokgalaka et al. [24], produced NiTi intermetallic coating on Ti-6Al-4V alloy using a laser metal deposition process. Results revealed that the presence of NiTi, NiTi2, and NiTi3 led to improvement in wear resistance up to 80%. Nevertheless, the aforementioned methods have limitations such as the formation of micro pores and inherent defects like cracks, expensive equipment, and operating complexity. Therefore, applying a diffusion-based treatment, which improves adhesion strength to the substrate, can overcome the defects of the previous methods [25]. Nikbakht et al. [26] reported the formation of a composite Ni-Ti coating on the titanium substrate through a cold spray method followed by a heat treatment. Li et al. [27,28] proposed a new method for preparing Ni-Ti intermetallic compounds by nickel plating on titanium substrate followed by electron beam remelting. The authors of the present work have reported the formation of Ni-Ti intermetallic coatings by nickel plating on commercially pure titanium followed by heat treatment [29]. A 20 μm thick layer with equi-atomic Ni-Ti composition is produced on the surface of Cp titanium via diffusion treatment. The aim of this work is to study and compare the wear behavior of commercial pure (Cp) titanium and Ni-Ti coated material. In this regard, the wear mechanisms are discussed based on the microstructural studies which provide an explanation to the enhancement of wear resistance in the coated samples in comparison with Cp titanium.

2. EXPERIMENTAL

40 mm × 40 mm square shape specimens were cut from a 3mm commercial pure titanium (99.5 %) sheet. The specimens were grounded with emery paper up to 800 grid and then polished. To activate the titanium surface, the specimens were initially immersed in an aqueous solution containing copper sulfate 200 g/l, sulfuric acid 40 g/l, aluminum sulfate 24 g/l, and wetting agent 0.1% for one minute. They were then immersed in a second solution containing sodium dichromate 100 g/l, copper sulfate 5 g/l for one minute at 85 °C, and hydrofluoric acid (52%) 50 ml/l for five minutes.

The activated specimens were then electroplated with nickel using Watts bath (containing 240 g/l NiSO4.6H2O, 30 g/l NiCl2.6H2O and 30 ml/l H3BO3), at a current density of 20 mA/cm^2 for one hour at room temperature to produce a 30 μm nickel coating. The distance between the cathode and anode during electroplating was fixed at 2 cm. After the plating process, the samples were washed with deionized water and then dried. Based on the results obtained in our previous work [29], the coated specimens were then heat treated at 900 °C for 12 hours in a controlled atmosphere furnace and were consequently cooled to room temperature in the furnace.

The phase structure of the coated layers was identified by X-ray diffractometer (XRD) using Cu kα (step size = 0.01°, and time per step = 0.5 Sec) operated at 40 kV with a 30 mA emission current. Morphology and elemental composition of the coating layers and worn surfaces were studied using a scanning electron microscope (Cambridge stereo scan 440 SEM) equipped with an energy dispersive spectroscope (Philips PV 9800 EDS) employing an accelerating voltage of 30 kV. A typical microhardness profile of the coated materials was obtained at an applied load of 0.147 N for 15 s by a microhardness machine using a Vickers indenter. The hardness measurement was repeated five times for each point, and the average values were reported.

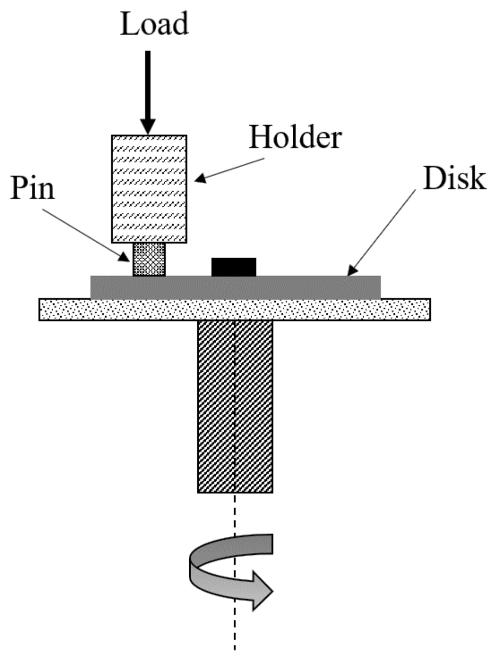


Fig. 1. Schematic of Pin-on-disk tribometer.

Pin-on-disk wear test machine was used to measure the room temperature dry sliding wear rate of titanium before and after coating. For this purpose, the bare and coated specimens were prepared as 30 mm diameter disks. AISI 52100 steel pins with 5 mm diameter and a hardness of approximately 740 HV were used as counterface. The schematic diagram of this test is presented in Fig. 1. Before each test, the pins and discs were ultrasonically cleaned in acetone for 6 minutes then dried and weighed with a precision of 0.1 mg. The wear tests were performed under 3 different loads (10, 20, 40N) at a sliding speed of 0.25 m/s for a sliding distance of 500 m. Weight losses of the specimens were measured using a high-precision balance. The coefficient of friction was also recorded by a computational system during the wearing process. In addition, Hommelwerk T-8000 roughness tester was used to obtain grooves depth by measuring the roughness before and after were testing.

3. RESULTS AND DISCUSSION

The cross sectional SEM image of the diffusion coated specimen in Fig. 2 shows two distinct layers from the surface to the core of the diffused layer on the titanium substrate. Based on the EDS results presented in Table 1, it appears that the outer layer is equiatomic NiTi with 15 µm thickness, and the bottom layer with a thickness of about 4 µm is $NiTi_2$ intermetallic. The X-ray diffraction pattern shown in Fig. 3 supports the EDS result which demonstrate the presence of NiTi, and $NiTi_2$ intermetallic in the treated layer. In fact, electroplating results in the formation of a pure nickel layer on the titanium surface. Post heat treatment results in the mutual diffusion of the coated layer and sublayer in each other. Based on the high diffusion coefficient of Ni in Ti, after 12 hours, the penetration of nickel in the titanium results in the formation of intermetallic compounds and disappearance of initial nickel plated layer.

The hardness profile along the top of the fabricated layer toward the CP titanium substrate in the treated material is shown in Fig. 4. The outer layer (NiTi layer) hardness is 280 HV, and then it drops with a steep gradient in the diffusion zone at a hardness level of about 200 HV. The hardness eventually reaches to 180 HV, which is the hardness value of the base material.

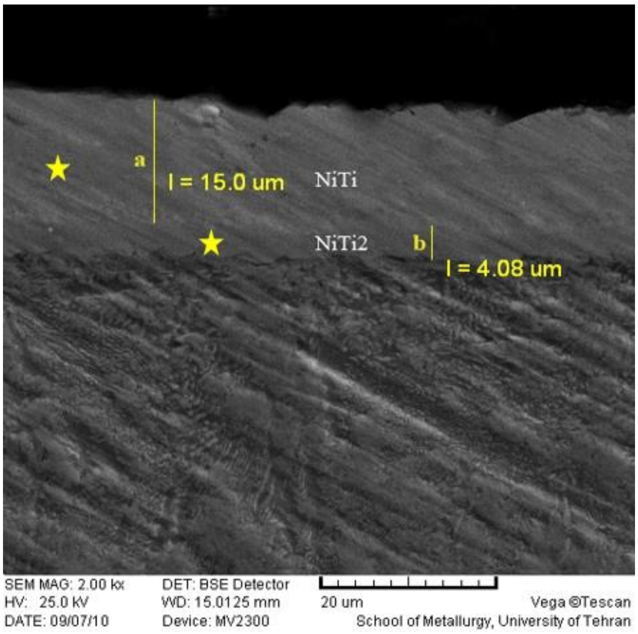


Fig. 2. Cross sectional SEM image of the nickel plated specimen after 12 h heat treatment at 900°C.

Table 1. EDS analysis of the marked points in layers a, and b of Fig. 2, formed after Ni plating followed by heat treatment.

Layer	Ni (at%)	Ti (at%)
a	49.8	50.2
b	33.9	66.1

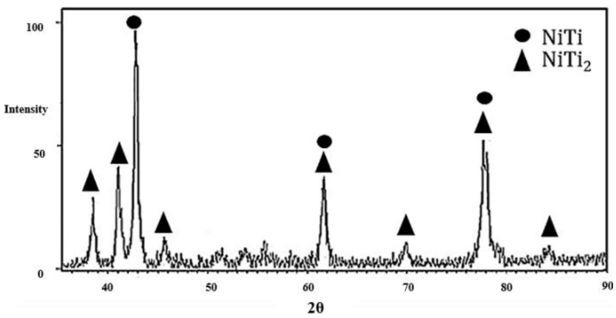


Fig. 3. XRD pattern of Ni plated CP titanium after 12 h heat treatment at 900°C.

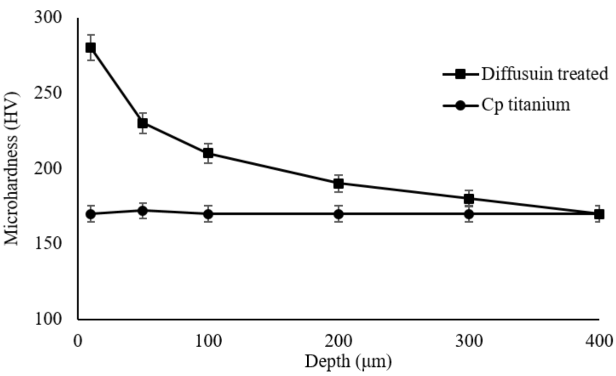
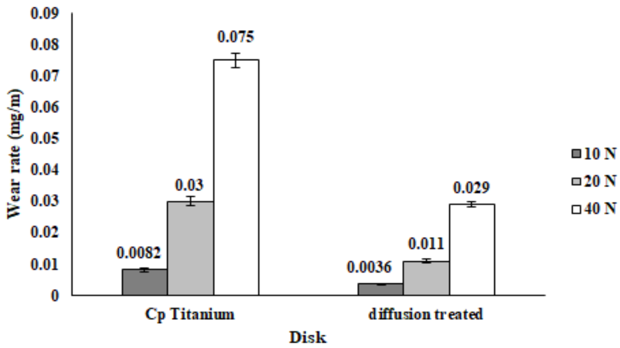
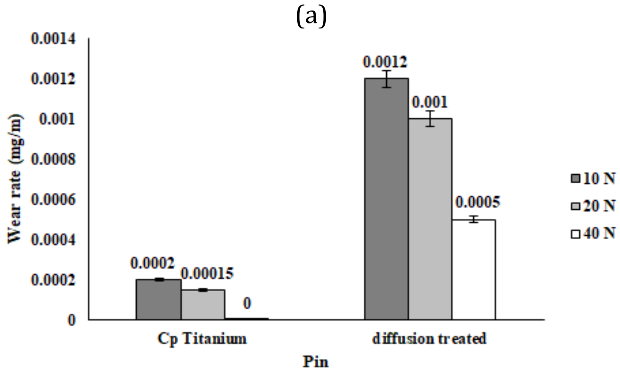


Fig. 4. Micro hardness profile along the cross-section of Cp titanium and the diffusion coated material.

The wear rates of the Cp titanium before and after diffusion treatment as a function of load for a sliding distance of 500 m are shown in Fig. 5a. As can be seen in this figure, there is a significant increase in wear rate with the load increasing for both materials. Nevertheless, the wear rates of the treated material under all applied loads are much lower than those of Cp titanium. The wear rates of the counterface pins are shown in Fig. 5b. It is obvious that under three different loads the wear rate of the pins against the bare samples are significantly lower than those used for treated specimens. This could be a result of the lower hardness of the untreated material in comparison with the surface treated ones. Besides, increasing the load up to 40 N reduces the wear rate of the pin against Cp titanium to zero. It is believed that applying high loads results in temperature rise that in turn deteriorates the mechanical strength of the bare titanium and consequently the wear rate of the counterface pin is reduced to zero [30].



(a)



(b)

Fig. 5. Variation of wear rates versus loads for (a) Cp titanium and diffusion treated materials, and (b) the counterface hardened steel pins.

In order to eliminate the effect of the sliding distance, the specific wear rate of disks was calculated by dividing the wear rate to the applying load. Fig. 6 shows the specific wear rates of the Cp and diffusion coated specimen as a function of load. The figure reflects a dramatic increase in wear resistance after the aforementioned surface treatment.

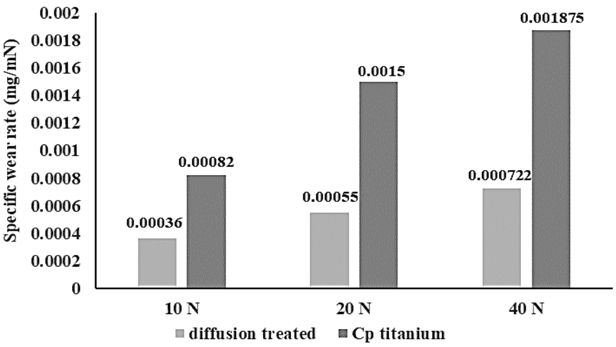


Fig. 6. Variation of specific wear rates as a function of loads for (a) diffusion treated and (b) Cp titanium samples.

Assessing the wear track of the diffusion coated sample achieved after 500 m sliding under 40N load (Fig. 7) revealed that, the groove depth is about 40 μm, which is significantly greater than the coating thickness (about 20 μm). It seems that the intermetallic layer has been totally removed. Therefore, to evaluate the wear mechanism before losing the intermetallic layer, the sliding distance decreased to 200m for this particular specimen.

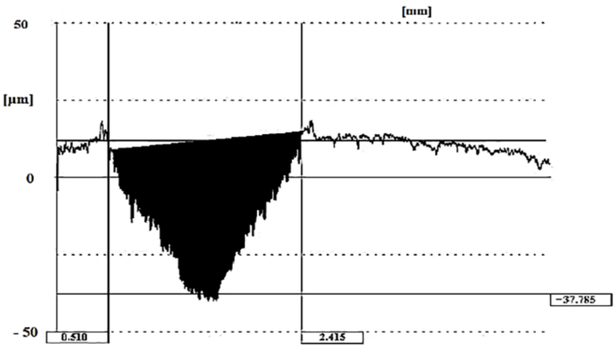
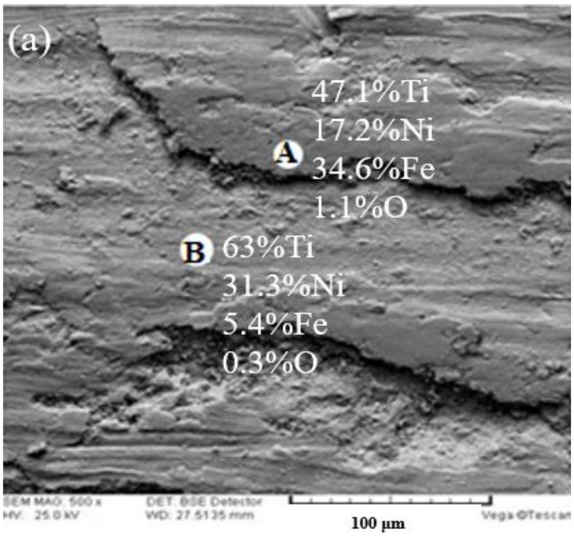


Fig. 7. Depth profile of the worn surface of the treated specimen after 500 m sliding under 40N load.

Fig. 8a and 8b, respectively show the SEM images of the worn surfaces of diffusion coated and Cp titanium achieved after wearing under 10 N. The respected EDS analysis of the worn surface of the diffusion treated specimens are also given in Fig. 8a. The EDS results are in favour of the formation of tribological layers on the worn surface of the coated specimen. These layers are mainly contained nickel, titanium, iron, and oxygen. With respect to the image of the treated sample after abrasion under 10 N (Fig. 8a), it becomes apparent that the tribological layers in area A are thicker than those of the area B, because the amount of iron and oxygen in area A are higher than those of the area B. Furthermore, cross sectional image in Fig. 9 also confirms the formation of the tribological layer with perfect bonding on the surface of the coated specimen during wear under an applied load of 10 N. It appears that the rate of the formation of oxides is higher than their removal. This can protect the surface from severe wear and results in the reduction of the wear rate to 0.0036 mg/m value, as can be seen in Fig. 5a. The remarkable wear rate of the pin against the diffusion treated sample which is about 0.0012 mg/m under 10 N load (Fig. 5b) also confirms the existence of a hard tribological layer. This observation is also in agreement with the results found in the literature [31], which demonstrates four times improvement in the wear resistance of the nickel diffusion treated titanium compared to that of untreated Ti-6Al-4V alloy. The authors stated that this was due to the formation of a tribological layer as well as an increase in the hardness compared to that of the untreated material.



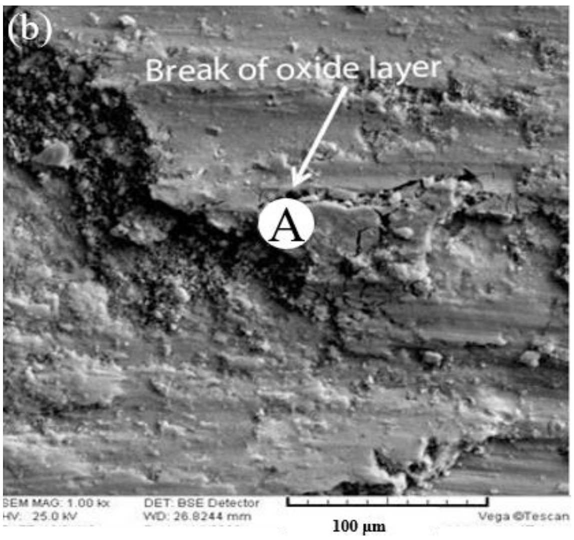


Fig. 8. SEM images of the worn surfaces of diffusion treated specimens (a) and Cp titanium (b) under 10N load.

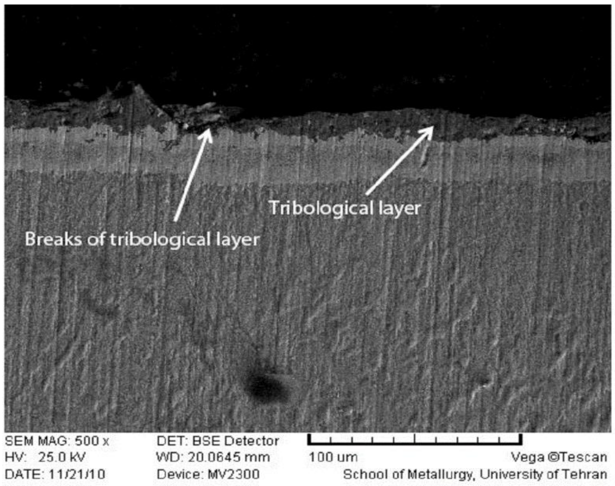


Fig. 9. SEM cross sectional image of the worn treated titanium under 10N load.

The SEM images of the worn surfaces of the Cp titanium produced under an applied load of 10N are also shown in Fig. 8b. The relevant EDS analyses of these worn surfaces reveal that the chemical composition of the region marked as A in this figure consists of 86.5% titanium, 12.5% iron, and 1% oxygen. These results demonstrate that a Ti-based tribological oxide layer was formed on the surface in regions like A. In addition, as is shown in figure 8b some cracks are formed on this tribological layer. It seems that the rate of the formation of the tribological layer is lower than its removal, so this layer could not protect the surface.

The micrograph and EDS analysis of the worn surface of the coated specimen under 20N are shown in Fig. 10a. These results reveal that a mixture of titanium, nickel, and iron oxides as a tribological layer is formed in the region marked as C. The EDS analysis of region D and some broken regions indicate that the tribological layer has been broken and removed. Therefore, the wear rate of the coated specimen under 20 N applied load is more than that of the 10 N. Furthermore, as can be seen in the SEM image of the worn surface of Cp Ti under 20N load (Fig. 10b), there is no oxide layer to protect the surface, and plastic deformation is the main wear mechanism which leads to increase of the wear rate under 20 N load in comparison with the diffusion treated specimen. The micrograph and the EDS analysis of the worn surface of the coated material under 40 N (Fig. 11a) show that the mixed oxide layer yet remains on the surface in regions like E. This figure also illustrates the formation of debris that were able to aggravate abrasion and as a result, no protection layer was left on the surface in the region marked as F, which led to a higher wear rate as compared to those of similar specimens under lower forces (as shown in Fig. 6a). In addition, with due attention to the micrograph of the worn surface of Cp Ti under 40 N in Fig. 11b, it becomes clear that there is not any tribological layer to protect the surface. The plastic deformation and fatigue cracks are the main wear mechanisms in this specimen.

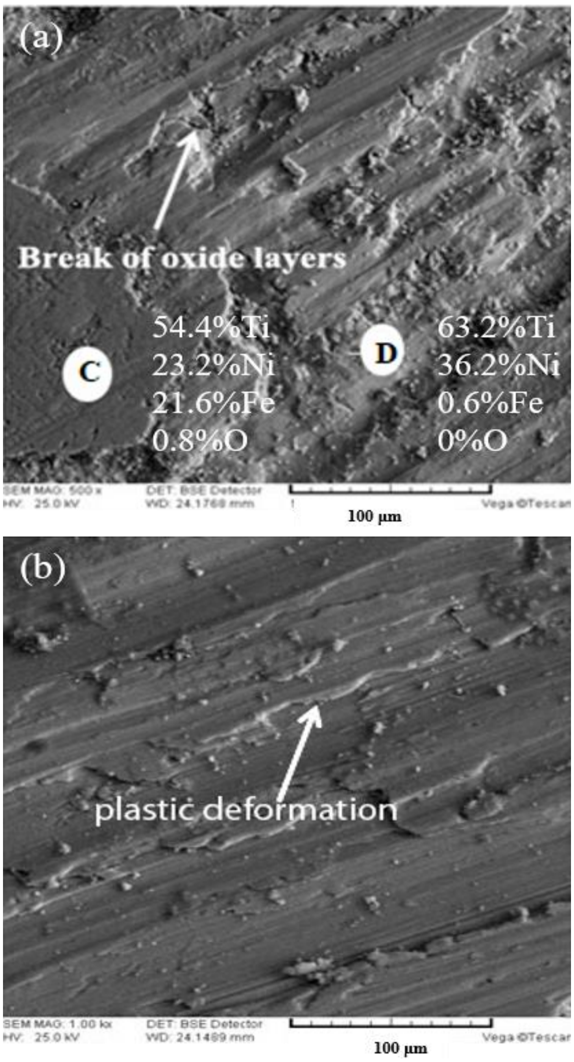


Fig. 10. SEM images of the worn surfaces of diffusion treated specimens (a) and Cp titanium (b) under 20N load.

Fig. 12 shows the cross sectional micrograph of the worn Cp Ti under an applied load of 40 N. It can be seen that holes and cracks are formed in depth below the surface and eventually result in the formation of debris with a possible delamination mechanism. In fact, by increasing the amount of the applied force, the surface temperature increases due to the heat generated by friction, and as stated in a previous research [26], the yield strength of titanium decreases by increasing temperature and as a consequence, the plastic deformation becomes more severe. Briefly, even under a high load (40N), NiTi coating can withstand and protect the surface from severe wear that occurs in bare titanium. This observation is in accordance with findings of the previous study [23].

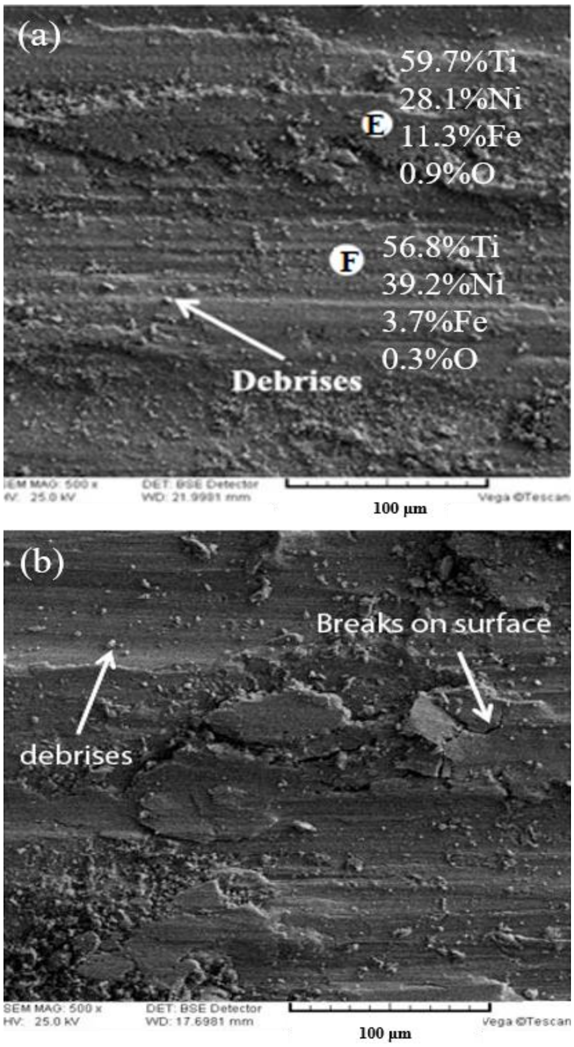


Fig. 11. SEM images of the worn surfaces of diffusion treated specimens (a) and Cp titanium (b) under 40N load.

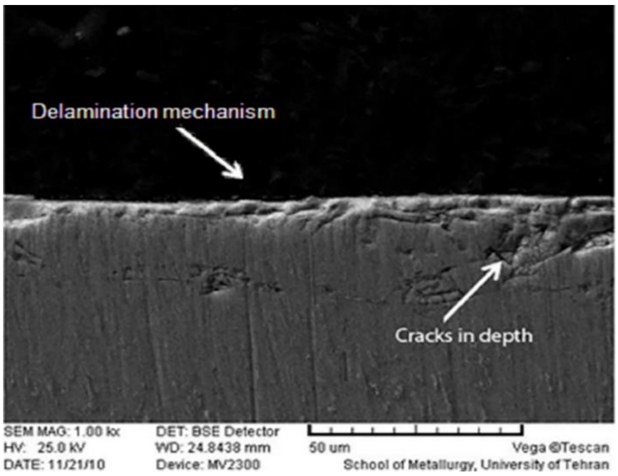


Fig. 12. SEM cross sectional image of the worn Cp titanium under 40N load.

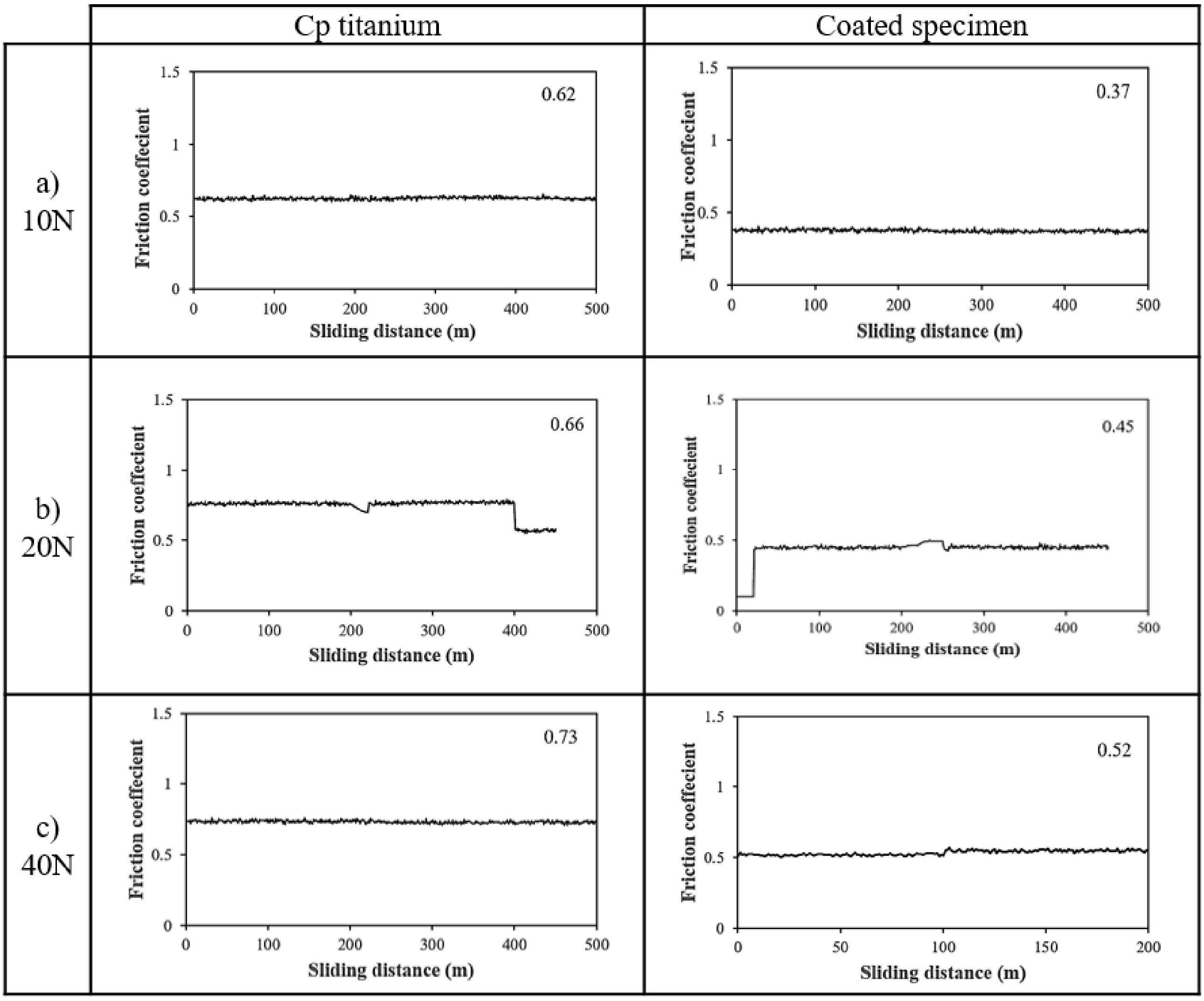


Fig. 13. Friction coefficient versus sliding distance of Cp titanium and treated specimens under three different loads: a) 10N, b) 20N and c) 40N.

Fig 13 presents the friction coefficient of treated and bare specimens versus sliding distance under 10, 20, and 40N loads. These figures confirm the transition of wear mechanism by increasing the applied forces. As can be seen, under 10N load (Fig. 13a) both the treated and the bare materials possess the lowest friction coefficient fluctuations, which indicate a low adhesion between these samples and the relevant counter facing pins during the wear test. These results also confirm the presence of the tribological layers on the surfaces of the samples, because the formation of this layer reduces the adhesion and friction coefficient by reducing metal-on-metal contact. By increasing the applied load up to 20N (Fig. 13b) some fluctuations are noticed. This is because the adhesion has increased after 200 m, which can be attributed to an increase in the metal-on-metal contact and subsequent failure of the oxide layer. Further rising in load value (Fig. 13c) and consequently increasing the temperature on the surface during the course of wear, results in the surface oxide layer failure. Therefore, the wear mechanism changes, and the plastic deformation becomes the dominant mechanism of wear according to Fig. 11b. Hiraga et al. believed that rising the load value causes an increase in the hardness of NiTi intermetallic phases. [32]. However, hardening does not seem to have a significant effect on the reduction of the wear rate, because the thickness of the treated surface layer decreases by increasing the sliding distance, and the effect of titanium substrate increases. The comparison between treated and bare materials reveals lower friction coefficients in all treated samples compared to those of the untreated ones.

4. CONCLUSIONS

Commercial pure (Cp) titanium was surface coated with equiatomic NiTi intermetallic layer via electroplating and subsequent heat treating. Morphological and dry tribological tests were conducted to evaluate the behavior of diffusion coated and Cp titanium and the results summarized as follow:

1. XRD analysis revealed the formation of NiTi and NiTi2 phases on the surface of the diffusion treated materials. Moreover, the hardness profile along the top of the fabricated layer toward the Cp titanium substrate showed a steep drop.
2. SEM observation suggested the formation of a tribological oxide layer on the surface of the diffusion treated materials under different loads in the course of wear.
3. The wear rate of Cp titanium and diffusion treated samples increased significantly by rising of the load, but there was a dramatic increase in wear resistance after the surface treatment.
4. Studying the worn surface of Cp titanium revealed the formation of a non-continuous oxide layer under 10 N load. By further increasing the load up to 40 N plastic deformation mechanism prevailed which led to increased wear rates.
5. Under 10 N load, treated material showed the lowest fluctuations in friction of coefficient, but by further rising of the load, the amplitude of these fluctuations increased.

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