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Araştırma Makalesi / Research Article

Ticari Saflıkta Titanyumun Akımsız Ni-B Kaplaması ile Yüzey Özelliklerinin Geliştirilmesi

Improvement of Surface Properties via an Electroless Ni-B Coating for Commercial Purity Titanium

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Ö*z-* **Bu çalışmada, ticari saf titanyumun (CP-Ti) yüzeyi akımsız Ni-B ile kaplandı. Yüzey morfolojisi, mikroyapı ve faz analizi, Taramalı Elektron Mikroskobu (SEM) ve X-Işınları Difraksiyonu (XRD) ile analiz edildi. Elde edilen Ni-B kaplama, işlem görmemiş CP-Ti'ye kıyasla önemli ölçüde daha düşük aşınma hızı ve kararlı sürtünme katsayısı sergilemiştir. CP-Ti yüzeyine oluşturulan Ni-B kaplamanın üstün aşınma direnci sergilemesi, oluşturulan kaplamanın yüksek sertlik ve kendinden yağlama özelliğinden kaynaklanmaktadır.**

Anahtar Kelimeler- **Aşınma Direnci, Sürtünme, Akımsız Ni-B, Titanyum.**

A*bstract-* **In the present study, the surface of commercial pure titanium (CP-Ti) was coated with electroless Ni–B. The surface morphology, microstructure and phase identification were analyzed by Scanning Electron Microscope (SEM) and X-Ray Diffraction (XRD). The obtained Ni-B coating demonstrated significantly lower wear rate and stable friction coefficient compared to bare CP-Ti. The superior wear resistance of Ni-B coating on the CP-Ti is dealt with the combination of its high hardness and self-lubricating property.**

Keywords- **Wear Resistance, Friction, Electroless Ni-B, Titanium.**

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I. INTRODUCTION

Owing to advantages of titanium and its alloys such as high specific strength, corrosion resistance and biocompatibility, components made of them are demanded by medical implants and other branches of marine, automotive and aerospace [1, 2]. However, the practical application of titanium based alloys has been limited due to the undesirable properties, including insufficient hardness, low wear resistance and high friction coefficient [3-5]. These concerns can create a practical issue when the alloys are considered to surpass the challenging needs for wear related engineering applications. Therefore, many researchers have attempted to develop high wear-resistance strategies. Various techniques are being pursued, focused on improving surface properties of the alloys [6-16].

Among the surface engineering methods used to protect titanium based alloys, electroless nickel plating is capable of providing wear resistant hard surface because of its simple process procedure [17]. Electroless has the ability to provide uniform deposits in intricate-shaped components [2]. Electroless nickel deposition processes are classified as pure Ni plating, Ni-P and Ni-B.

The present work aims to describe a procedure to coat commercially pure titanium (CP-Ti), by means of an electroless Ni-B process that is one of the most cost-effective approaches for improving the tribological properties of titanium alloys.

II. EXPERIMENTAL PROCEDURE

Electroless Ni-B coating was applied on the CP-Ti (Grade-2) substrate. Before plating, all samples were prepared to ensure reproducible surface condition: grinding with 1200 SiC abrasive paper; degreasing with acetone; etching in 6 vol. % HF for ~1 minute at room temperature and rinsing with deionized water before direct immersion in the plating bath for 60 minutes. More details on the bath composition have been given by Mindivan et.al [18]. The cross-section of the coating was included in a resin and polished by metallographic procedures.

Phase analysis and surface morphology of Ni-B coating were determined by X-Ray Diffraction (XRD) and Scanning Electron Microscope (SEM), respectively. The thickness of the deposited coating was measured by the Nikon Eclipse LV150 Light Optical Microscope (LOM). Hardness measurements were carried out using a Shimadzu HMV-G hardness tester equipped with Knoop indenter under a load of 10 g and load exertion time of 15 s. The indents were performed on polished cross sections to avoid the effect of the CP-Ti substrate.

Dry sliding wear tests were investigated by the ball-on-plate method with a home-made reciprocating wear tester by rubbing a 10 mm Al_2O_3 ball. Wear tests were carried out under a normal load of 5 N, a sliding speed of 1.7 cm s^{-1} and a sliding distance of 50 m. At the end of the wear tests, the wear tracks developed on the surfaces of the samples were monitored using a 2-D contact surface profilometer (Mitutoyo Surftest SJ-400). The cross section of every track was measured in three different locations. After determining the average depth and width of each wear track, the volume loss (mm^3) obtained by multiplying the cross-sectional area of wear track with its stroke length (12 mm) was used for calculating wear rate (mm^3/Nm) by considering normal load (5 N) and total sliding distance (50 m). SEM examinations were made on the worn surfaces. The contact surfaces of the Al_2O_3 balls were also examined by a LOM. Volume loss of the Al_2O_3ball (V_b) was determined according to the following equation;

$$V_b = \pi.d^4/64.R \quad (1)$$

where R is the radius of the Al_2O_3 ball and d is the average diameter of the wear scar formed on the ball.

III. RESULTS AND DISCUSSION

The Ni-B coating surface morphology, as well as its cross section, are shown in Figures 1 a and 1 b, respectively. In Figures 1 a and b, SEM and LOM images show a typical cauliflower like structure and a spherical nodular morphology. This kind of surface topography was produced due to the nodular grain growth [19, 20]. A layer with a thick of about 13 μm was formed on the surface as shown as Figure 1 b. The surface of the coating is wavy, exhibiting the typical nodular morphology of the electroless Ni-B coatings [18, 21]. Surface roughness measurements showed that the Ni-B has a high roughness value Ra = 2.59 ± 0.01 μm.

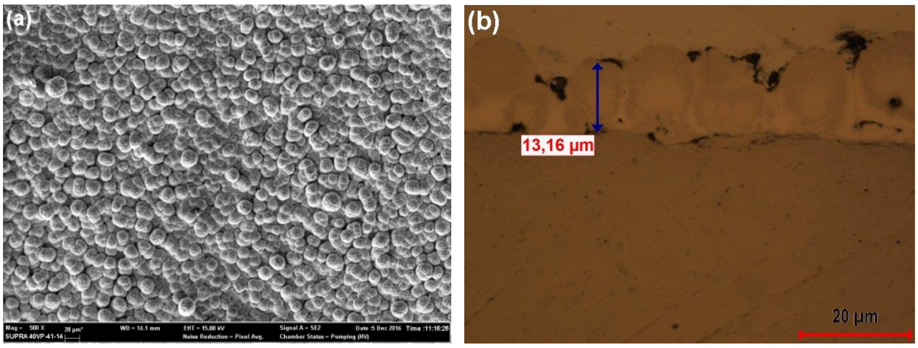


Figure 1. SEM and LOM micrographs of electroless Ni-B coating (a) surface morphology and (b) cross section.

Figure 2 depicts the XRD pattern of Ni-B coated CP-Ti sample. The broadening of diffraction peak at 2θ = 40 - 50° for Ni-B deposit shows the presence of amorphous structure. The single broad peak obtained by XRD analysis is corroborate well with Energy Dispersive X-ray Spectroscopy (EDS) analyses. Since the boron content in the Ni-B coating without heat treatment was higher than 5 wt. % [18], an amorphous structure was achieved. Similar results have been reported by Watanabe et al. [22] and Vitry et al. [23]. In terms of Knoop microhardness, the microhardness of the as-deposited coating of the present work was 818 ± 61 $HK_{0.01}$ and the uncoated CP-Ti sample had a microhardness of 279 ± 11 $HK_{0.01}$.

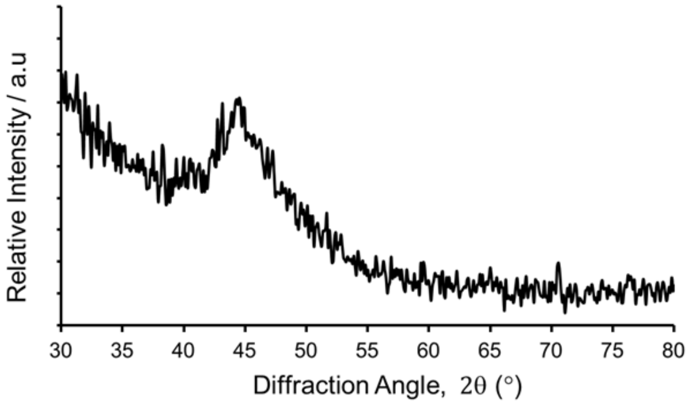


Figure 2. XRD pattern of Ni-B coated CP-Ti sample.

LOM images of wear tracks of the uncoated and Ni-B coated CP-Ti samples and corresponding Al_2O_3 balls are shown in Figure 3. It is clear that the track width was ~ 1.2 mm for the uncoated CP-Ti sample (Figure 3 a) compared with ~ 600 µm for the coating (Figure 3 c). Further, the contact surface of the testing ball used on the coating as the result of the relatively little amount of material removal from the Ni-B coated CP-Ti sample was relatively smooth (Figure 3 d), while the contact surface of the ball used on the uncoated CP-Ti sample was rough and covered with materials transferred from the uncoated CP-Ti sample and appeared as dark region within the wear scar (Figure 3 b).

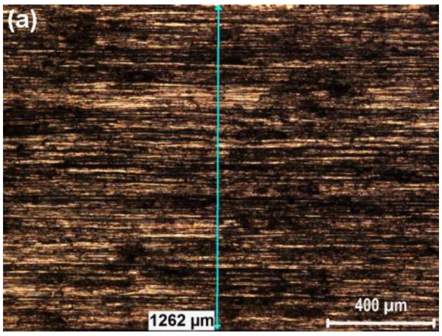
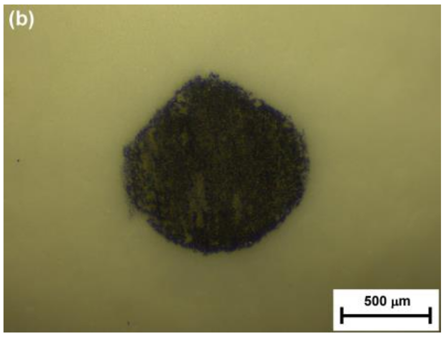
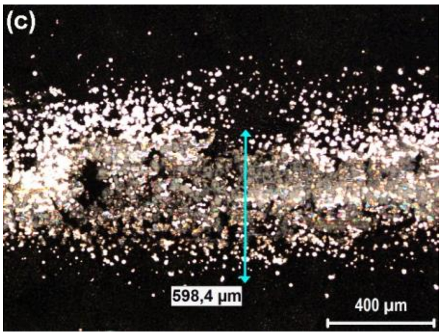
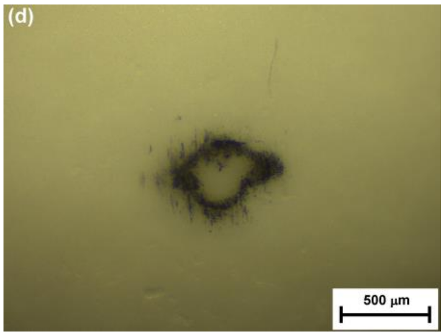
Sample	Wear Track	Counter Face
Uncoated CP-Ti		
Ni-B coated CP-Ti		

Figure 3. LOM images of wear tracks of the uncoated and Ni-B coated CP-Ti samples and corresponding Al_2O_3 balls.

The friction curves during the dry sliding wear tests of the uncoated and Ni-B coated CP-Ti samples are shown in Figure 4. The friction coefficient of the uncoated CP-Ti sample was approximately constant, while that of the Ni-B coated CP-Ti sample continued to fall, but at a reduced rate. There was a heavy fluctuation in the values of friction coefficient for the uncoated CP-Ti sample as compared with the Ni-B coated CP-Ti. Wear test results are listed in Table 1 in terms of friction coefficients, wear rates and wear volumes of Al_2O_3 balls. In general, steady state friction coefficients of about 0.84 and 0.32 were measured for the uncoated and Ni-B coated CP-Ti samples, respectively. As presented in Table 1, the uncoated CP-Ti sample exhibited 9 times higher wear rate than the Ni-B coated CP-Ti.

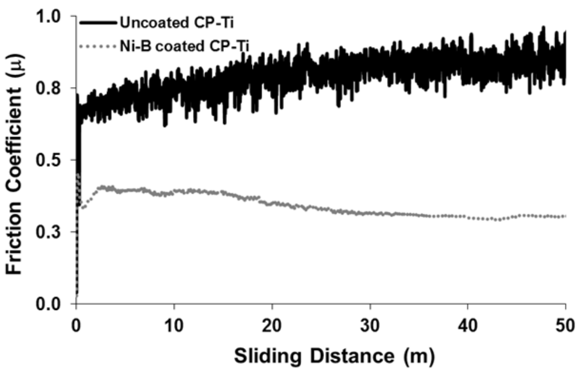


Figure 4. The friction curves of the uncoated and Ni-B coated CP-Ti samples.

Table 1. Wear test results of the uncoated and Ni-B coated CP-Ti samples.

Sample	**Steady state friction coefficient**	**Wear rate (mm^3/Nm)**	**Wear volume of Al_2O_3 ball (mm^3)**
Uncoated CP-Ti	0.84	73.92×10^{-5}	9.57×10^{-3}
Ni-B coated CP-Ti	0.32	8.16×10^{-5}	0.33×10^{-3}

The wear tracks of the uncoated and Ni-B coated CP-Ti samples are shown in Figure 5. As shown Figure 5 a and b, the uncoated CP-Ti sample exhibited severe wear. SEM inspection of the wear track on the bare substrate indicates that the worn surface was covered with continuous grooves parallel to the sliding direction (Figure 5 b). Conversely, at low SEM magnifications a smoother surface was observed in the wear track for the coating (Figure 5 c). At high magnification (Figure 5 d), it is possible to observe that flattening occurred for some nodules while others remained almost undamaged, as reported by previous studies [18, 21]. The flattening areas revealed the nodular grain growth of the Ni-B coating. Finally it can be concluded that a hard and self-lubricating Ni-B coating also led to a reduction in the actual contact area resulting lower wear rate and friction coefficient (Table 1). The cauliflower like surface morphology and nodular growth have been confirmed in several research works and the present observations corroborate well with them [17-23].

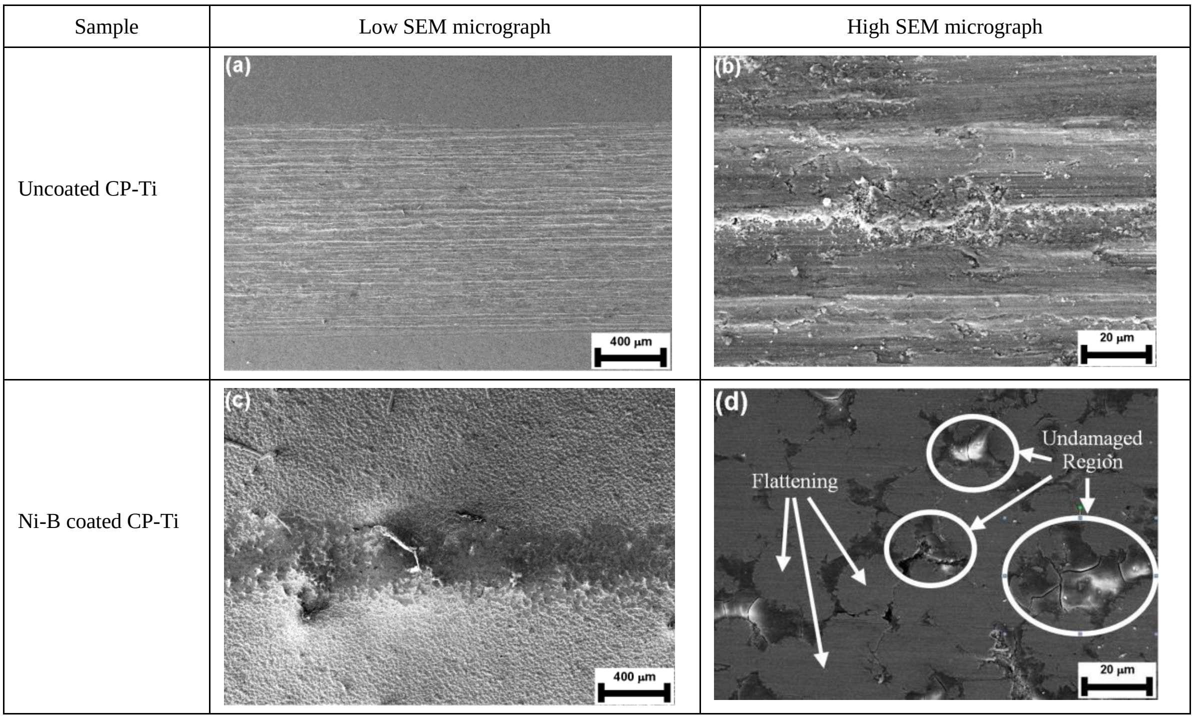


Figure 5. Low and high maginification SEM micrographs of wear tracks of the uncoated and Ni-B coated CP-Ti samples.

IV. CONCLUSIONS

Ni-B coating with the thickness of about 13 μm on CP-Ti sample was successfully prepared by electroless deposition bath. Conclusions might be drawn as follows.

- The surface morphology of the coating exhibited a typical cauliflower structure without any porosity.
- The Ni–B deposit was an amorphous structure.
- Exceptional value of microhardness (818 ± 61 $HK_{0.01}$) was obtained for the Ni-B coated CP-Ti sample.
- Tribological sliding tests conducted on CP-Ti sample coated with a Ni-B coating, indicated that a wear rate of approximately 8.16 x 10^{-5} mm^3/Nm was obtained, which was almost nine times smaller than that corresponding to the uncoated CP-Ti substrate.

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