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Investigation on the phase transformation of electroless Ni–B coating after dry sliding against alumina ball

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ABSTRACT

The purpose of the work was to evaluate the possibility of tribochemical reactions during dry sliding of electroless Ni–B coating against an alumina ball. The effect of probable tribochemical products on the tribological behavior of Ni–B/Al_2O_3 tribosystem was investigated. Wear experiments were performed in a ball-on-disc configuration pressing alumina balls towards rotating disc specimens. Wear tests were carried out at normal loads of 5, 20, 40 and 60 N at the sliding distance of 100 m. Electroless Ni–B coatings with a mean thickness of 50–60 μm were applied on the mild carbon steel substrates. The worn surfaces of Ni–B coatings were studied with Scanning Electron Microscope (SEM), Energy-Dispersive X-ray Spectroscopy (EDS), X-Ray Diffractometer (XRD) and X-ray Photoelectron Spectrometer (XPS). The results showed that the structure of the worn surfaces of the coatings changed from amorphous to the crystalline. The dominant wear mechanism in the present tribosystem was combination of adhesive and abrasive wear. As the load was increased the friction coefficient and its fluctuations were decreased. The wear rate of alumina balls changed from increasing to decreasing at the load of 40 N. XPS and XRD analysis revealed the phase transformation and production of Ni–Al compounds on the worn surface of electroless Ni–B coating. It was elucidated that tribochemical reactions between the mating surfaces were responsible for tribological behavior of Ni–B coating and unexpected wear behavior of alumina balls.

1. Introduction

Hard coatings have been developed to improve material's surfaces. Generally these coatings are applied on the substrate to control the wear and friction behaviors of it [1,2]. Among the deposition processes, electroless plating is capable of producing wear resistant hard coatings without the use of special equipment. Among the hard electroless coatings, electroless nickel especially electroless nickel-boron (Ni–B) coatings have attained the greatest attention due to their unique characteristics. Electroless Ni–B coatings are harder and more wear resistant than the hard chromium and electroless nickel-phosphorous (Ni–P) coatings [3]. In spite of the significant achievements made in the study of tribology of electroless Ni–B coatings, most of these studies were focused on the scratch test and Taber wear test [4–6] or on the tribological behavior of electroless Ni–B coating against hardened steel [3,7]. In recent years, joints of nickel alloys with alumina are the subject of many researches due to the wide range of their properties, i.e. high temperature strength and oxidation resistance [8,9]. One of the practical applications of ceramic-metal joints is in the micro electro mechanical systems (MEMS). In these systems nickel/alumina contacts prevail between the bottom side of the nickel rotor and the alumina ground plate. Survey of the literature elucidate that the relative humidity of the surrounding air, applied contact pressure and surface roughness had a strong influence on the tribological behavior of Ni/Al_2O_3 pairs in MEMS [10–12]. It has been frequently reported that unfavorable tribochemical reactions may be responsible for unusual wear rate of alumina in tribosystems [13]. Although tribological behavior of Ni–B/Al_2O_3 tribosystem has been investigated by Correa et al. [2,14], their attention was paid more to the tribological behavior of Ni–B at a constant load and sliding distance rather than tribochemical reactions between the mating surfaces. There are some studies in which their focus is on the tribochemical reactions and phase transformations in some tribosystems consisted of alumina or other ceramic sliders. Ravikiran et al. [15] studied the dry sliding of alumina pins against steel disks. They have reported that $FeAl_2O_4$ (spinel) and $FeAlO_3$ were formed as tribochemical products. Dong et al. [13] investigated the

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tribological behavior of Ti6Al4V/Al_2O_3 sliding pairs. They concluded that Ti_3Al and TiAl intermetallics were formed as products of tribochemical reactions between alumina balls and counterface material. Qu et al. [16] observed the formation of Si_5AlON_7 as a tribochemical product during dry sliding of Si_3N_4 balls against Ti6Al4V disks. Based on their XRD patterns, they also suggested the possibility of formation of Ti–Al intermetallic compounds during dry sliding of alumina balls against Ti6Al4V disks.

It seems that up to now, there is no report about investigation on the possibility of tribochemical reactions that may occur between the mating surfaces of the Ni–B/Al_2O_3 tribosystem. Accordingly the main objectives of the present work were to investigate the tribological behavior of Ni–B/Al_2O_3 tribosystem from tribochemical reactions point of view and to explore the wear mechanisms involved.

2. Experimental

2.1. Sample preparation

Electroless Ni–B coatings were applied on the Ck45 steel substrates. All the substrates were ground up to 600 grit SiC paper. Then, the specimens were ultrasonically cleaned in acetone for 10 min and rinsed in 10% sodium hydroxide solution at 50 °C for 3 min. The surfaces of the samples were activated in 15% HCl solution at room temperature for 1 min. Finally, the substrates were rinsed in distilled water and submerged vertically in the deposition bath. At each cleaning interval, the samples were rinsed in distilled water.

2.2. Bath preparation and operating conditions

The chemical composition of the plating bath and operating conditions are present in Table 1. Ni–B coatings were applied on the substrates during two consecutive deposition processes. The whole time of each plating process was 2 h. The thickness value of the coatings was about 50–60 μm in all cases. The bath solution was agitated at 600 rpm using a magnetic stirrer. The bath temperature was maintained at 95 ± 1 °C. During each process the ratio of deposition area to the volume of the plating solution was kept constant at 0.66 dm^2/L. After deposition process, the coated samples were rinsed in distilled water and dried using a stream of air.

2.3. Coating characterization

The boron and nickel content of Ni–B coatings was measured by Inductively Coupled Plasma-Optical Emission Spectrometer (ICP-OES, VARIAN 735).

Roughness measurements were carried out using a surface profilometer (T8000 Hommelwerke).

Microhardness tester (Micromet1, Buehler) equipped with Vickers indenter was used for hardness measuring. Hardness measurements were carried out on the specimens' cross sections under a load of 50 gf and load exertion time of 15 s. Measured microhardness values were reported as an average of three readings for each specimen.

Tribological behavior of the samples was investigated using a ball-on-disk tribometer without the use of any lubricants. The coated samples served as the disks and the counterparts were alumina balls with the hardness of 1400 HV. The sliding speed and the radius of the wear track were 0.2 m/s and 4 mm, respectively. Before starting the wear tests, the samples were cleaned in acetone for 2 min with an ultrasonic cleaner and then dried in air. Wear tests were carried out at 25 °C and 35% RH under normal loads of 5, 20, 40 and 60 N at the sliding distance of 100 m. The weight loss of the samples was measured by weighing them before and after each test on a balance with an accuracy of ±0.1 mg. Three wear tests were performed at each sliding condition to ensure repeatability of the tests. Good repeatability was obtained in almost all results. The average value of relevant weight losses was used for calculating the specific wear rate.

A scanning electron Microscope (SEM, TESCAN-VEGA) equipped with EDS (RONTEC) analyzer was used to study the surface morphology, wear tracks on the coated specimens, wear scars on the alumina balls and wear debris.

The crystal structure of Ni–B coatings and their worn surfaces was analyzed by X-Ray Diffractometer (XRD, Philips X'PERT PRO) using Cu K_α radiation with wavelength of 0.1542 nm. The tube voltage and current were set at 30 kV and 20 mA, respectively. The samples were scanned between 20° and 90° at a rate of 3°/min.

The surface atomic concentration and chemical composition of the worn surfaces were investigated using X-ray Photoelectron Spectrometer (XPS) equipped with an Al K_α X-ray source at energy of 1486.6 eV in an ultra-high vacuum system with a base pressure lower than 2×10^{-9} Torr. All binding energy values were calibrated by fixing the C (1s) core level peak at 284.8 eV.

Table 1
The chemical composition of electroless Ni–B plating bath and its operating conditions.

$NiCl_2 .6H_2O$	30 g/L
Ethylenediamine	90 g/L
NaOH	90 g/L
$NaBH_4$	1.0 g/L
Thallium acetate	18 mg/L
pH	14

3. Results and discussion

3.1. Characteristics of the coating

According to the results of ICP analysis, the chemical composition of the electroless Ni–B coatings was 92.5 wt.% nickel, 6.5 wt.% boron and the remainder contained traces of thallium and sodium.

XRD pattern of the coated samples (Fig. 1) showed a broad diffraction peak of Ni(111) at about 2θ = 45° as well as a high background which indicates a mixed crystalline/amorphous structure. It is well known that the amount of codeposited boron in Ni–B coatings plays a key role in determining the structure of the coatings [17]. Gaevskaya et al. [18] devied the Ni–B deposits into three groups according to their boron content. When the boron content is between 0.5 and 6 at.%, the structure of the coating is polycrystalline. When the boron content is between 6.0 and 20 at.% then the film structure is a mixture of crystalline and amorphous.

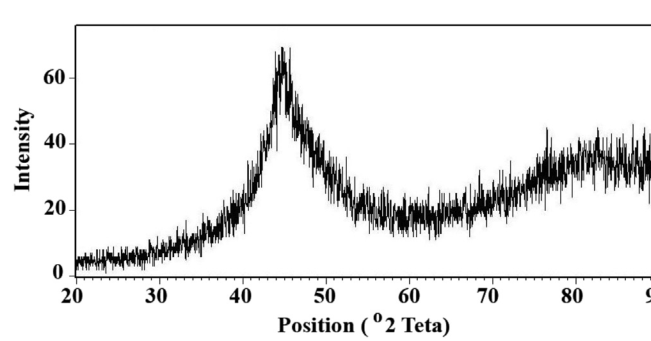

Fig. 1. X-ray diffraction pattern of electroless Ni–B coating.