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The effect of post aging on wear properties of a plasma nitrided ferromagnetic steel under DC magnetic field

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ABSTRACT

Plasma nitriding is an effective surface treatment to improve unsatisfactory wear properties of machine elements. Such machine elements may also subject to magnetic fields in many industrial applications. Therefore, AISI 4140 ferromagnetic steel samples were plasma nitrided. Then, the samples were quenched in water and then post-aged at a temperature of 400 °C for 30 and 120 min in plasma environment. Reciprocating wear tests were performed under different oriented DC magnetic field to determine wear behavior of the samples. It was obtained that the magnetic field and its orientation during wear test were the most effective parameters on wear behavior. It was observed that post-aged samples showed better wear resistance and lower friction coefficient values than the merely nitrided samples. Hard $Fe_{16}N_2$ precipitations were formed in the nitrided layer after post-aging treatment and their amount increased with magnetic field. This gave rise to improvement the wear resistance.

1. Introduction

Low alloy steels are widely used for producing many machine elements such as gears and crankshafts and they are frequently subjected to wear. Friction and wear problems shorten their service life and increase waste of time and costs. Therefore, service life of machine elements should be increased. Several surface treatments such as physical vapor deposition [1–3], plasma diffusion treatments [4,5] and ion implantation [6] have been applied on material surfaces in order to solve tribological problems. Among these methods, plasma nitriding is one of the most used methods because of advantages such as easy application, minimum distortion with lower process temperatures, being relatively inexpensive, environment friendly and ability of excellent dimensional control. Plasma nitriding involves nitrogen diffusion into the surface of the material and produce both a compound layer consisting mainly of ε-$Fe_{2-3}N$ and γ'-Fe_4N phases and a diffusion zone under this layer. While improvement in tribological and corrosion properties is provided with compound layer, diffusion layer increases fatigue life of nitrided materials [7]. However, it is often required better mechanical and tribological properties than achieved with plasma nitriding. In this case, some post processes are applied after plasma nitriding. Post-aging treatment is suggested as one of these treatments [7,8]. In the post-aging treatment, samples are quenched after plasma nitdiring and then it is followed by an aging treatment. After this post-aging treatment, α''-$Fe_{16}N_2$ phase precipitates in the modified layer. Previous studies were focused on the formation and magnetic properties of α''-$Fe_{16}N_2$ phase [9–12]. Also, there are some limited papers can be found on mechanical and tribological properties of this phase [7,8,10]. On the other hand, many machine elements may be subjected to the magnetic or electrical field if they are working close to an external magnetic field and/or electrical field generation. In these circumstances, performance and service life of these machine elements are determined by taking their working conditions into account. In the scientific literature, mechanical and tribological properties of untreated ferromagnetic steel samples were investigated in some studies [13–21]. They pointed out that magnetic field changed the material properties and accelerated the oxidation rate of wear debris during wear tests. Zaidi et al. [19] presented a magneto-tribological model of ferromagnetic/ferromagnetic sliding couples both in ambient air and vacuum conditions. They showed that magnetized contact enhanced ferromagnetic Fe_3O_4 oxide growth. Also, it was seen that mean friction coefficient decreased in ambient air and increased in vacuum under magnetic field. Chin et al. [20] determined that oxide film formation in magnetized sliding steel/steel contact changed wear mode from metal/metal contact to metal/oxide contact and reduced the friction coefficient values and wear rate. Kumagai et al. [15] determined that increment of oxidation rate with magnetization decreased both coefficient of friction and wear rate

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values. Hiratsuka and Sasada [14] performed the wear tests in air and Ar atmospheres to prove the effect of magnetization on oxidation rate. They confirmed while wear rates decreased in air with chemisorption of oxygen on material surface, Argon atmosphere did not have any effect. Bi and Wang [13] showed that magnetization promoted the oxygen activation and this resulted in formation of the fine oxidized particles. They also stated that this kind of debris were stuck on the wear scar and prevented further wear loss. In another study, Stolarski and Makida [16] indicated that magnetic field exposure direction was very important on tribological properties. They noted that wear resistance increased if external magnetic field was applied in the horizontal direction to the sliding direction. In our previous study, tribological properties of ferromagnetic steel samples that were pre-magnetized and magnetized during sliding were investigated [17]. It was determined that wear resistance of the samples magnetized during sliding was improved. Also, it was seen that as magnetic field intensity increased, wear rates decreased. Zaidi and Senouci [21] investigated the effect of presence of magnetic field on friction behavior of a ferromagnetic/paramagnetic sliding contact. They found that while application of magnetic field decreased friction coefficient, it increased the surface hardness and wear rates of sliding couples. As seen in scientific literature, whole previous studies focused on the effect of magnetic field on untreated (non-surface treated) samples. In the light of this information, the main aim of this study was to investigate the effect of magnetic field on tribological properties of untreated, plasma nitrided and post-aged samples and to contribute to information in this research area.

2. Experimental details

AISI 4140 steel specimens with $25 \times 25 \times 5$ mm^3 dimensions were used in this study. The chemical composition of the AISI 4140 is tabulated in Table 1. The specimens were polished by alumina powder with 1 μm grain size followed by grinding with 220–1200 mesh emery papers. After cleaning with ethanol, the specimens were placed in a plasma nitriding chamber, which was evacuated to 2.5 Pa. Prior to treatments, hydrogen sputtering for 15 min at 500 V and a pressure of 5×10^2 Pa was applied to the specimens in order to remove any surface contaminants. Then, the specimens were plasma nitrided at 575 °C for 1 h in a gas mixture of 50% N_2 and 50% H_2 under a constant pressure of 5×10^2 Pa. Subsequently, the specimens were quenched in water and then post-aged at a temperature of 400 °C for durations of 30 and 120 min. For the post-aging treatments, a gas mixture of 50% Ar and 50% H_2 was used [7]. Thus, four groups of specimens were obtained i.e. untreated, plasma nitrided, plasma nitrided and post aged for 30 min and post aged for 120 min.

The surface hardness measurements were conducted by using a Bruker Universal Mechanical Tester-UMT at a constant load of 25 g and a dwell time of 15 s. For phase analyses, XRD-Panalytical/Empyrean operated at 30 kV and 30 mA with CuKα radiation. The treated specimens were studied by using ESEM, FEI QUANTA-FEG 250.

The reciprocation wear tests were carried out on a Turkyus PODWT&RWT reciprocating tribo-tester according to ASTM G133-02. Al_2O_3 ball with a diameter of 6 mm was used as the pin. The system ran in ambient air, at room temperature (~18 °C) and a relative humidity of approximately 50%. At this stage, three different wear tests were performed to determine the effect of magnetic field on the wear properties of the specimens. The first test was performed without the magnetic field under dry sliding conditions as the control group. The other tests were conducted with the application of magnetic field in two different directions: reciprocation direction and perpendicular to reciprocation direction. DC electro-magnet, which is produced by coiling copper on iron core, was used as the magnetic field source. The magnetic field intensity of 1500 G measured by a gaussmeter was applied to the specimens during tests. The wear tests were conducted at a normal load of 2 N, total sliding distance of 141 m and a wear track length of 8 mm. The wear load of 2 N corresponds to maximum Hertzian contact pressures of 954, 997 and 1009 MPa and average contact pressures of 634, 665 and 676 MPa for untreated, plasma nitrided and plasma nitrided-post aged specimens, respectively. That value was calculated by using the following equation: $P_{max} = 3F/2\pi a^2$ where P_{max} is the maximum contact pressure, F is the wear load and a is the equivalent contact radius [22]. Wear rates were calculated through the Archard equation $V = k(ws/H)$; where V is the worn volume, k is the wear coefficient, w is the normal load, s is the distance moved and H is the surface hardness [17]. After the wear tests, the worn specimens were examined by using ESEM, FEI QUANTA-FEG 250. Also, 3D surface profiles were recorded by using the Bruker ContourGT-I Optical Microscope.

Table 1
Chemical composition of AISI 4140 (wt%).

C	Mn	Cr	Si	Ni	Mo	V	S	Cu	Fe
0.36	0.80	0.014	0.005	0.30	0.85	0.075	0.07	0.143	Balance

3. Results and discussion

3.1. Structural characterization of modified layer

Fig. 1 shows the XRD patterns of untreated, plasma nitrided and post-aged samples. α-Fe peaks diffracted from the low alloy steel untreated samples. After plasma nitriding, modified layers consisted of face centered cubic (fcc) γ'-Fe_4N and hexagonal closed packet ε-$Fe_{2-3}N$ nitride phases with solid solution of nitrogen in α-ferrite. After quenching which is the first stage of post-aging, nitrogen-supersaturated ferrite was formed. Then, it transformed to more stable body centered tetragonal (bct) α''-$Fe_{16}N_2$ phase after decomposition of compound layer. XRD results of post-aged samples showed that the intensities of α-ferrite peaks decreased while the aging time increased the peak intensity of α''-$Fe_{16}N_2$ phase.

Fig. 2 shows the cross-sectional SEM images of plasma nitrided and post-aged samples. As well known, after plasma nitriding a continuous compound layer on top of the surface and diffusion layer beneath the compound layer is formed. It was observed that a distinct and visible line separated the compound layer from diffusion layer. Alsaran et al. [7] pointed out that the diffusion layer mainly composed of γ′ nitride while the compound layer was composed of ε nitride. After post-aging treatment, α''-$Fe_{16}N_2$ phase formed in the modified layer as a result of transformation of nitrogen-supersaturated α-ferrite. In Fig. 2b, α''-$Fe_{16}N_2$ phase was observed as darker areas under diffusion layer. SEM images showed that while compound layer thickness was about 13–15 μm, visible diffusion layer thickness was about 18–20 μm. Case depth values are different from the visual observations because plasma nitriding and post-aging treatments are diffusion-based processes. Therefore, case depth values were measured with microhardness tests. Microhardness values were measured from the surface to the core of treated specimens. Distance from the surface to the point of core hardness was accepted as case depth.

The microhardness measurements of plasma nitrided and post-aged samples are given in Fig. 3. Both plasma nitriding and post-aging treatments increased the surface hardness values of the samples. Microhardness versus distance from the top of surface curves of all surface treated samples showed similar tendency. But, the highest microhardness value and higher diffusion depth were obtained from the sample post-aged for 120 min. Increase of micro-hardness values after plasma surface treatments was attributed to the changes of chemical composition of the surface and strains inside