Microstructure and wear properties of plasma nitrided low alloy steel tubes

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Abstract

Plasma nitriding is a surface treatment process, which can be applied on steel substrates with a wide variety of industrial applications. This study focuses on the properties of plasma nitrided low alloy steel of relatively long-small diameters tubes containing low amounts of Cr and Ni. The microstructure was investigated by using the optical microscopy (OM) and scanning electron microscopy (SEM). The results represented two nitride layers. In spite of the low amount of Cr and Ni, the compound layer demonstrated the maximum micro hardness of 800 HV. X-ray Diffraction (XRD) patterns, confirmed the presence of Fe₃N(ε), Fe₂N(γ’), Cr₂N, and Fe₃NiN in the nitrided layer of samples. Wear properties were evaluated by pin on disc wear test under the loads of 10, 20, 30, 60, and 90 N, and sliding distance and velocity of 1000 m, and 0.3 m s⁻¹, respectively. Results showed that for 10 and 20 N loads the wear loss of nitrided samples was significantly lower than that of the uncoated ones; for 60 to 90 N loads the wear loss was slightly lower while under 30 N load the wear performance of nitrided sample was not as good as expected.

1. Introduction

Plain carbon steels known as non-alloy steels have many applications in industries and are less expensive than the alloyed ones. Mechanical strength of non-alloyed steels is lower compared to alloyed steels which limits their applications. However, as a result of high brittleness they are not widely applicable in surface enhancement by nitriding process [1, 2]. Non alloy steels can be nitrided using some special nitriding elements such as chromium and aluminum in manufacturing process or some kind of interlayer deposition, however this methods are not cost effective. In some recent studies by utilizing alloyed stainless steel active screen in active screen plasma treatment without any additional treatment the nitriding capability of non-alloyed steel was improved [3–5]. Now drawing concern on Low alloy steels reveals the fact that they are one of the most important groups of steels. In spite of having desirable properties, the surface properties of these alloys can still be improved with many different heat treating techniques such as gas/plasma assisted nitriding, which provides a significant increase in hardness especially in the steels containing nitridable elements such as Cr, Ti, and Al [6]. The combination of nitrogen with these elements and producing hard compounds such as CrN, TiN, AlN and … leads to an increase in mechanical properties of nitrided alloys especially wear resistance, however, applying this processes for internal surfaces of the structures, especially those with small diameters has always been a big challenge for different industries [7, 8]. If low carbon steels are nitried, the hardness of the surface could increase up to 400HV. This is due to the nitrogen diffusion into the surface of the steel, at nitriding temperature, forming iron nitrides[9]. Elements such as chromium, could combine with nitrogen and limit the diffusion of nitrogen into the depth of the sample and make a thin and hard layer on the surface [10]. By increasing the amount of alloying elements, the hardness of the nitrided layer increases while its thickness decreases. Presence of chromium avoids the sudden reduction of hardness from the surface to the center of the sample [11]. Not only chromium but also presence of nickel contributes to the hardenability and also improving ductility and toughness of steels [12]. High temperature of the nitriding process could distort the parts [13]. By employing plasma medium, nitriding at lower temperatures is achievable which could minimize the distortion [14].
Plasma nitriding tubes of 1500 mm length and 25 mm inner diameter were cut from the primary rod and used for plasma nitriding studies and for optimizing the process parameters, as a benchmark, samples in the form of a disc with 60 mm and microstructural properties. In this work, low alloy steel with chemical composition listed in Table 1 was used as the substrate. For the primary setup configuration and process parameters of nitriding, samples from different parts of plasma nitrided tubes were taken and compared with bare samples. The results showed a significantly higher hardness values compared to the bare material (low alloy steel containing Cr and Ni). The wear loss of treated samples was studied, as well.

Table 1. Chemical composition of the samples.

|   | Fe | C  | Si | S     | P     | Mn | Ni | Cr |
|---|----|----|----|-------|-------|----|----|----|
| Substrate | 0.309 | 0.22 | >0.0002 | 0.0044 | 0.444 | 2.1 | 0.755 |

2. Experimental procedures

In this work, low alloy steel with chemical composition listed in Table 1 was used as the substrate. For the primary studies and for optimizing the process parameters, as a bench mark, samples in the form of a disc with 60 mm diameter and 10 mm thickness were cut from the primary rod and used for plasma nitriding (sample D). As schematically shown in Figure 1, the second set of samples (group P) were cut from the inner surface of the plasma nitride tubes of 1500 mm length and 25 mm inner diameter (samples P1, P2, and P3) which are listed in Table 2. Due to the similar results for hardness and phase composition of both sets of samples, wear tests were conducted on flat evidence samples (performing wear test on the concave inner surface of tubes via pin-on-disc method was not possible).

For sample D, prior to plasma nitriding process, the discs were ground and polished, using 60 to 2500 grit SiC papers and alumina suspensions, respectively. Samples were then degreased by ethanol solution and then sputtered in a chamber with 2.5 mbar pressure for 1 h in hydrogen and argon gases atmosphere. Conventional Pulsed-Plasma nitriding process was conducted in a plasma machine with 50 kW power. As can be observed in Figure 1, to enhance the uniformity of gas distribution along the tube length, a long stainless steel rod was used in the middle of the tube as the anode and the tube acts as the cathode. For the nitriding process, all the samples were processed for 12 h at 6 mbar pressure in the glow discharge plasma of 75%H₂ + 25%N₂ atmosphere at 510 °C.

Nitriding is also applicable on iron-based substrates and has found some applications in industries [15]. The lower treatment temperature makes it possible to process long and complicated parts with almost no distortion. Particularly, treatment of the inner surface of tubes, with tight dimensional tolerances [16]. Plasma nitriding process occurs at 500 °C–600 °C [17, 18]. The structure of the nitriding layer that forms on the surface is divided into two zones: compound and diffusion layer. The first layer is a compound layer that contains iron nitrides such as Fe₂₋ₓN(ε), FeₓN(γ) or a combination of both. Beneath the compound layer is the diffusion layer [19]. Diffusion zone with increased residual compressive stresses causes an increase of the fatigue resistance of the parts such as tubes [16]. In the work of Khyoupin Khoo et al., microwave plasma nitriding of the inner wall of hollow tubes (1, 1.5, 2, and 3 mm inner diameter), with lengths of 5, and 12 mm was conducted. They found that the hardness values were around 800 HV; measured at both ends of the nitrided tubes for all diameters. The internal wall surface hardness profile of the 3 mm hollow tube had almost uniform hardness above 700 HV all along the entire tube length due to the formation of Fe₂₋ₓN(ε), and FeₓN(γ) compound layers [20]. Borisyuk et al [21] found that hardening of the internal surfaces of tubes by nitriding at 400 °C–550 °C in abnormal glow discharge with a hollow cathode showed hardening all through the length of the tube at both sides of it. The thicknesses of the compound and hardened layers were about 2–5 μm, and 300–400 μm, respectively. The hardness of the material below the compound layer increased by 1.5–2.5 times after nitriding.

Nitride layer produced on the surface could tolerate high loads without causing severe wear [22]. Karaglu et al studied wear and structural behavior of plasma nitrided low alloy steel AISI 5140 with pin on disc method by measuring the weight loss versus sliding distance. They found that in 400–600 volts, and current density of 0.04–0.25 mA mm⁻², the wear resistance ability in plasma nitrided sample mainly increased compared to the initial sample [23].

In the present work, plasma nitriding is successfully employed to treat inside of a relatively long (1500 mm) and small inner diameter (25 mm) tubes of low alloy steels. This is the first time that such a process is successfully conducted on such long samples to the best knowledge of authors. Different processing parameters are examined and results showed that processing the samples with optimized conditions could increase the hardness and wear properties of the samples.

Monotonous distribution of plasma ionized gas concentration along long tubes are challengeable; however, it can be resolved by optimizing process parameters. This was successfully done, in this study, by changing the setup configurations and process parameters of nitriding. Samples from different parts of plasma nitrided tubes were taken and compared with bare sample. The results showed a significantly higher hardness values compared to the bare material (low alloy steel containing Cr and Ni). The wear loss of treated samples was studied, as well. The formation of phases was investigated and dominant wear mechanisms were suggested. In conclusion, this technique could be a potential method for coating the long tubes in order to enhance their inside wear resistance and microstructural properties.
The plasma was produced at 480 volts. During this process, gas pressure and current of the chamber were kept constant. The samples were kept in the chamber until they were cooled down to the room temperature.

To study the microstructure of the samples the cross-sections of the nitrided specimens were cut and mounted. Using SiC papers with grids of 60 to 2500 the cross-section of the samples were grinded and polished by alumina suspensions and then etched with the nital etchant. The microstructures of the samples were investigated with an optical microscope and a scanning electron microscope in the SE mode (a Camscan MV2300 connected to EDS Bruker XFlash GL10). The thickness and structure of the layers and the distribution of the elements such as Fe, Cr, and Ni were investigated.

To obtain the hardness profile from surface to the center of the nitrided samples, micro hardness test with the load of 100gf for 10 s was used.

X-ray diffraction (XRD, Phillips XPert-Pro) measurement was performed using Cu Kα irradiation (λ = 1.54 Å) at a scanning rate of 1 step/s and a step size of 0.02°/step at 40 kV and tube current of 30 mA to study the phase formation in the nitrided layers, on the surface and cross-section of the samples.

To conduct wear tests and measure weight loss of the samples and coefficient of friction, pin on disc method was employed. Pins with a diameter of 5 and a height of 10 mm were made from nitrided samples. The counterpart disc was made of quenched and tempered 52100 steel. The wear rate was calculated by dividing the weight loss by the sliding distance. The applied loads were 10, 20, 30, 60, and 90 N, the rotation speed was fixed at 0.3 m s

The coefficient of friction was calculated using the following equation.

$$\mu = \frac{F_f}{F_n}$$

where $F_n$ is normal load, $F_f$ is friction load, and $\mu$ is friction coefficient.

### 3. Result and discussion

#### 3.1. Microstructural observation

Cross-sectional optical microscopy images of samples D, P1, P2, and P3 are shown in figure 2. As it can be seen, the compound layer is formed on all surfaces with different thicknesses. The thickness of the compound layers P1, P2, P3 and D samples was measured in 10 different points of the layer in each sample by utilizing digimizer and the average value of the layer thickness is reported in table 3. In steels with a high percentage of nitriding elements, the formation of fine nitrided precipitates specifies the thickness of diffusion layer. In this work, due to the chemical composition of the used steel, the diffusion layer can’t be recognized under the optical microscope.

As it is clear, the thickness of compound layers in samples taken from various depths in nitride tubes are different which may have a couple of reason such as; First, dissipation of nitrided particles due to the sputtering
process and reducing the thickness of the compound layer. Second, reprecipitation of the sputtered atoms from plasma atmosphere onto the surface [24, 25]. The Thickness of the compound and diffusion layer on the surface of nitriding samples are shown in table 3.

From the tip (sample P1) to the center of the tube (sample P3), the thickness of the diffusion and the white layer (compound layer) decreases, due to an increase of surface sputtering process; and also, probably because the available amount of gas and nitriding ions for diffusing into the surface of the sample P1 is more than that of sample P3. As a result, the thickness of white and diffusion layers decreases from the tip to the center of the tube.

In the compound layer, a fine porous region is observed. According to the kolbel plasma nitriding model, plasma nitriding process causes iron particles cut off from the surface and combine with nitrogen to form FeN. However, this phase is not stable and is converted to Fe$_3$N and Fe$_{2.5}$N which are more stable phases [26]. Released nitrogen atoms in this process, diffuses into the metal. Increasing nitrogen atoms concentration on the surface of the metal leads to the production of nitrogen gas molecules which is the main reason for the appearance of porosity. Figure 3 shows these typical porosities on the surface of sample D.

Diffusion of nitrogen into the structure of iron is increased at 500 °C (nitrogen is an interstitial atom), whereas, diffusion of chromium into the iron structure decreased because chromium is a substitutional atom. A high concentration of nitrogen in iron structure causes high-pressure stress. In this case, chromium cannot precipitate as (Cr$_3$N, CrN), and it remains as solid solution. By increasing the temperature, Cr$_6$N, and Fe$_4$N are

| Samples | P1  | P2  | P3  |
|---------|-----|-----|-----|
| Thickness of compound layer (μm) | 2.26 ± 0.52 | 1.94 ± 0.44 | 0.94 ± 0.26 |
| Thickness of diffusion layer (μm) | 525 | 511 | 205 |
formed, due to the decomposition of supersaturated unstable austenite and as a result of reduction of compression stress, porosities are emerged \[27\]. CrN is a wear resistance phase. Another phase existed in this region of the compound layer is Fe₂NiN, which has the same crystal structure as Fe₁N and is a magnetic phase \[28–30\].

Figure 4 shows the x-ray diffraction patterns of bare sample and samples D, P1, P2, and P3. X-ray analysis for bare sample represents Fe (α iron), and interstitial compounds such as FeNi and Fe–Cr based on the chemical composition of the substrate (table 1). For sample D, it is clear that the main nitried components are ε, γ′, Cr₂N, and Fe₂NiN. According to the 75%H₂ + 25%N₂ nitriding atmosphere, the concentration of nitried species are low, and as a result, γ′ can be formed. When the concentration of active nitrogen ions for formation of ε isn’t sufficient, γ′ is formed. As seen in figure 5, epsilon phase peaks are emerged and their intensity increased from tip (sample P1) to the center (sample P3) of the tube. During plasma nitriding process carbon atoms move from surface to the center of the tube. Since carbon stabilizes the ε phase, the amount of this phase is increased in the center of the tube from sample P1 to P3.

Figure 5(a) shows surface hardness of samples P1, P2, P3, and D. The highest hardness value belongs to the sample P1 with the compound layer consisted of mostly γ′ phase. As previously mentioned, if the compound layer contains γ′ and ε, weak interface emerges that could cause hardness reduction \[31\]. Two factors could enhance the Formation of γ′ phase; firstly, decarburization on the surface of the samples occurs by surface sputtering (carbon stabilizes the ε phase), and therefore, formation of γ′ on the surface is promoted, and
secondly, when the amount of nitrogen in the atmosphere is low, the concentration of active nitride species in the plasma atmosphere decreases, and as a result, conditions to produce $\gamma'$ are achieved [32, 33]. Reduction of the hardness of sample P2, and P3 compared to sample P1 is due to having both $\gamma'$ and $\varepsilon$ phases which are formed in these samples as shown in x-ray diffraction patterns (figure 4). Different crystal structure of these two phases may cause an increase in residual stresses in the compound layer, and reduce toughness and make micro cracks in this layer.

Presence of alloying elements such as chromium reduces the thickness of the compound layer. These elements are combined by nitrogen on the surface of the steel and the nitrided compounds are formed and in this case, nitrogen cannot diffuse into the steel. As a result of the thinner compound layer in sample P3, chromium can diffuse and react with nitrogen to form chromium nitride ($\text{Cr}_2\text{N}$). Formation of $\text{Cr}_2\text{N}$ can cause hardenability of the compound layer. However, since the amount of $\varepsilon$ phase in x-ray diffraction pattern of sample P3 is significant, it can cause a brittle compound layer, and reduce the hardness.

For identifying the thickness of the diffusion layer according to the hardness curve, the hardness profile of nitrided samples were plotted. Figure 5(b) shows the hardness profiles. The thickness of the diffusion layer is up to the part that the variation of hardness is not more than 10%. Hardening mechanism in diffusion layer is based on solid solution strengthening. During the plasma nitriding process, nitrogen substitution in the martensitic phase and repartition of carbon in the diffusion zone cause an increase in the concentration of carbon compared to the surface. Carbon is an element which has 0.8 Å atomic radius. Substitution of carbon by nitrogen with 0.7 Å atomic radius, causes interior stresses in the samples due to the difference in atomic radii These stresses may prevent further diffusion of nitrogen atoms into deeper layers of material [34].

Increasing the number of interstitial atoms (nitrogen) near the surface of the sample, increase the hardness of this zone. As can be observed from figure 5(b) hardness near the compound layer is higher than the diffusion layer and substrate. It’s evident that different chemical potential between the compound layer and substrate causes an increase in nitrogen concentration near the compound layer leading to an increased hardness of
compound zone. This profile is divided into two parts: (1) 0–15 micron: In this region, the amount of nitrogen is high, and the compound layer is formed. The capability of nitrogen diffusion decreases by increasing the depth in this region. The supersaturated surface at high temperature in this region is the main reason for the higher diffusion rate of nitrogen. (2) 15–500 micron: Similar to region 1, in this region, the capability of nitrogen diffusion decreases by increasing the depths and reducing the temperature.

Figure 6 shows the distribution of different elements in the compound and diffusion layers of sample D. Figure 6(a) shows the cross-section of sample D. A high concentration of nitrided species can be observed in figure 6(b). The amount of nitrogen and congestion of nitrided species near the compound layer is high, and it can confirm the formation of iron nitride phases in this sample [35]. The elemental map analysis and distribution of elements Fe, Cr, N, and Ni (figure 6(c)) and result of line scan of nitrogen from surface to the diffusion layer (figure 6(d)) also show that the concentration of nitrogen is the highest near the compound layer and gradually decrease as reaches the end of the diffusion layer.

Because the nitriding process parameters are the same for all samples, only SEM and EDS pictures of sample P1 is selectively presented in figure 7. Figure 7 represents the white layer and dark matrix (diffusion layer). Results show that compound layer have high concentration of nitrogen that causes iron nitride formation. The amount of chromium increases from the compound layer to the substrate, and therefore, a nitride compound (CrN) is formed. CrN acts as a barrier against the diffusion of nitrogen into the layers beneath.

3.2. Wear properties

Preparation of the wear test pins from the inner surface of the nitrided tubes, due to its concave geometry was not possible, therefore wear tests were done on sample D instead. Figure 8 shows the wear rate of bare sample and sample D under 10, 20, 30, 60, and 90 N loads.

As can be observed, weight loss of plasma nitrided samples under various loads are different. Increasing the hardness and as a result, increasing the wear resistance of different steels were investigated by Esfandiar et al [36] and Budinski et al [37]. Hardness plays an important role in wear processes because it is an important factor for requisite elastic strain energy for plastic deformation. In fact, increasing hardness causes decreasing the wear rate. A simple analysis for sliding wear indicated by Archard [38] equation.

\[
Q = \frac{KWL}{H}
\]

Where Q is a volume of wear loss at a unit of sliding distance, W is a normal load, H represents the hardness, and K is the wear constant without dimension. In this equation, it can be recognized that, by increasing the hardness and the load wear rate is decreased. As can be observed in figure 5, hardness of sample D is high due to high amount of nitried phases. On the other hand, the crystal structure affects wear and friction processes. Crystal structure of ε and γ′ phases are HCP and FCC, respectively. HCP metals have lower friction coefficient than BCC, and FCC ones. It can be mentioned that a good tribological properties of these metals are referred to limited sliding routes. Therefore their capability to deformation is low. The number of active sliding systems for HCP at low temperature is 1, and for FCC are 12 [39].

Hardness, thickness and phases that form the coating layer are the effective factors on wear rate. Among samples which were nitrided, a sample with thicker compound layer shows a higher wear rate. In this sample, breaking the layer and creating the wear species, increases wear rate. These species that come from the adhesive and flaky wear mechanism can cause abrasive wear. Therefore, the wear rate increases with increasing the thickness of the compound layer. This is confirmed with Staraffelini’s results [40]. In figure 8 in comparison between bare and plasma-nitrided samples under 10, 20, and 30 N loads, it can be recognized that up to 20 N load, plasma nitrided sample has the lower wear rate than the bare one however, under 30 N the wear rate of nitrided sample is higher than the bare one (0.0114, and 0.0081 respectively). This increase in wear loss is mainly because of breaking the compound layer, which contains brittle phase (ε). Due to the breaking of the compound layer under 30 N, worn surface reaches the diffusion layer and then steady wear occurs. However, under higher loads (60, and 90 N) the wear rate of plasma nitrided sample in comparison with bare sample decreases, due to the reformation of the oxides and tribological films. Therefore, a new compact oxide layer forms and reduces the wear rate.

In figure 8 it can be observed that wear rate of nitrided sample under the load of 10 N is 0.0008 gr/1000 m, and is increased up to 0.0129 under the 60 N which corroborates with Archard equation. However, the wear rate of nitrided sample under 90 N due to accommodation of oxide layers, decreases. By increasing the loads up to 90 N wear rate of the nitrided pin is close to the bare one that is related to the full destruction of the compound layer.

Figure 9 shows the SEM images and EDS map analyses of sample D under loads of 30, 60, and 90 N after wear. Map analyses of these layers confirm the presence of oxygen. Presence of few plate species, confirm the
Figure 6. Scanning electron micrographs of (a) Plasma nitrided cross-section of sample D (b) EDS map analyses of sample D, (c) Map analysis of elements Fe, Cr, N, Ni in cross-section of Plasma nitrided sample D, (d) Map analysis of nitrogen in cross-section of Plasma nitrided sample D, (e) Results of EDS line scan across a depth of nitrided layer cross section of sample D.
flaking during the peel off wear under 90 N load. So it can be concluded that after the formation of oxide layer, this layer is broken and peeled off from the surface.

Map analysis of worn surface under load of 90 N (figure 9(c)) shows an area with almost no oxygen which could be due to oxide layer removal.

Figure 10 shows the SEM image of surface of sample D after sliding distance of 1000 m under the loads 90 N. As can be observed, in figure 10, compact and uniform tribological layers are formed on the surface. Mechanism of formation of tribological layer during the wear abrasion in pin and disc occurs. Some wear species are trapped between the pin and the sliding disc starting three-body abrasive wear mechanism. During the sliding, wear species which are oxides, are broken and reached the critical size [41]. Species in this size begin to be agglomerated and compressed via contact stresses. By Formation of these oxide crusts, and again breaking them during the wear process, wear species are created that can cause severe abrasive wear on the surface of the sample.
Figure 9. Scanning electron micrographs and map analyses of worn pins (Plasma nitrided sample D) in 1000 m sliding distance, sliding velocity of 0.3 m/s under loads of (a) 30, (b) 60, and (c) 90 N.

Figure 10. Scanning electron micrographs of worn pin (Plasma nitrided sample D) under load of 90 N.
Figures 11(a)–(c) shows the wear surfaces under 30, 60, and 90 N loads. As the wear process continues the oxide layer is formed and become thicker and because of compression stress and accommodation of dislocations in the oxide layer, some part of this layer is peeled off at a critical thickness and accommodated at the region of sliding direction. Strains on layers near the surface cause plastic deformation and consequently cutting and/or cracking of the oxide layer. Dislocations pile ups may lead to the formation of holes, as well (figure 11(d)). As time passes, these holes join and form cracks. When the size of the crack reaches a critical size, then can propagate and break the surface layer. Another theory is compression theory. When two surfaces contact, under repeated loads, fatigue phenomena occurs and as a result, the species on the surface are deformed or broken. With increasing these kinds of deformation, some cracks emerge on the surface and then proliferate and shear the surface [38].

3.3. Friction coefficient plot
At the beginning of the wear process, friction coefficient is high, this is due to the facts that: first, the contact surfaces between pin and disc at the beginning of the wear process may not be 100% in touch, and second, the formation of oxide at the beginning of the wear process is low. During wear process, temperature of contact area increases, with two different effects. First, reducing materials strength and increasing ductility of it which may cause to an increase of the wear rate. Second, increasing the oxidation rate at contact surfaces that can cause to reduce the metal-metal contact and reduce the wear rate and friction coefficient[38]. The plot of friction coefficient versus sliding distance for bare sample and plasma-nitrided sample (sample D) under the applied load of 10, 20, 30, 60, and 90 N is represented in figure 12.

In comparison between friction coefficient of bare sample and sample D under loads of 10, 20, 30, 60, and 90 N it can be understood that there is not any noticeable change. However, under load of 60 N, it can be seen that friction coefficient of bare sample is higher than the treated one. Under the 60 N load, existence of compact oxide layers on the surface acts as a lubricant and cause to reduce the friction coefficient of sample D compared to the bare sample. Friction coefficient under 10, and 20 N loads due to a lower amount of weight loss of pin did not change significantly . However, under 30, and 90 N the friction graphs show that in both loads the friction coefficient of the bare and sample D is practically the same. It can be conclude that, under 30, and 90 N loads,
compound layer and oxide layers of nitrided sample are destroyed so the friction coefficients of the bare sample and treated one are the same.

As can be observed in figure 12, at the beginning of the wear process due to low amount of oxide formation adhesion between pin and disc and the amplitude of vibration is high. In this region, the created noise, and sound is high, as well. Then, when films are formed, the adhesion is lower, and as a result, the amplitude of vibration is low. Also, the created noise and sound is low.

The friction coefficient vary due to surface conditions. \( \mu = \frac{F}{W} \) and \( F = A \tau \) [42]. Where \( \mu \) is friction coefficient, \( F \), Friction load, \( W \), normal load, \( A \), the real contact surface, and \( \tau \) shear stress. So the real contact surface, and shear stress can affects the friction coefficient. Lack of the nitrided layer or existence of it, creation of oxide layer, the hardness of the substrate and the roughness of the surface affect the friction coefficient remarkably. Creation of oxide layer and the effect of it on reduction of the strength of interface between pin and disc causes the reduction of the friction coefficient. Lacking or removing the oxide layer, and increasing the adhesion between pin and disc, increases the amplitude of the friction coefficient variation.

4. Conclusion

In this research according to the studies on wear properties of plasma nitriding process on low alloy carbon steel containing Cr and Ni results showed that:

Plasma nitriding of long tubes with small diameter is successfully done and the formation of nitrided phases occurred. Nitriding causes the formation of compound, and diffusion layer with different thicknesses inside the tube walls.
Plasma nitriding on special steels containing low amount of Cr and Ni significantly increases the hardness by formation of Fe₃N(γ), Fe₃N(γ'), Ce₃N, and Fe₃Ni₄N phases and increasing the hardness up to 800 HV.

Thickness of compound, and diffusion layer in tube sample (sample P1, P2, P3) are different. The compound layer in P3 is thinner than P1, and this sample contains of ε, γ' phases, and the hardness of this sample in comparison with sample P1 which contains only γ' phase is lower.

Investigating wear behavior of sample D shows that plasma nitriding can be appropriate for special applications however wear process parameters should be controlled.

By investigating the wear processes and with increasing the load from 30 to 60 N, oxide films are formed due to mechanical stresses and increasing surface temperature. These oxide films act as a lubricant and reduce friction coefficient.

Under 90 N load, the tribological layer is formed and this can reduce the wear rate rather than 60 N load. However, under 90 N load, compound layer is broken and surface destruction happens and as a result friction coefficient of both bare and treated sample under this load is equal.

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References

[1] Jenkins I R and Forrest R 1999 Ductile Iron. (ASM International) Metals Handbook10th edn 1, p 33–55
[2] Naeem M, Shaﬁq M, Zaka-ul-Islam M, Ashiq A, Díaz-Guillén J C, Shahzad M and Zakaullah M 2016 Enhanced surface properties of plain carbon steel using plasma nitriding with austenitic steel cathodic cage Mater. Des. 108 745–53
[3] Naeem M, Raza H A, Shaﬁq M, Shabbir F, Iqbal J, Díaz-Guillén J C and Lopez-Badillo C M 2019 Improved nitriding capability of nonalloyed steels assisted with active screen plasma treatment Surf. Rev. Lett. 1950118
[4] Naeem M, Iqbal J, Shabbir F, Khan M A, Díaz-Guillén J C, Lopez-Badillo C M and Shaﬁq M 2019 Effect of pulsed current on cathodic plasma nitriding of non-alloyed steel Mater. Res. Express 6 066537
[5] Naeem M, Shaﬁq M, Zaka-ul-Islam M, Bashir M I, Díaz-Guillén J C, Lopez-Badillo C M and Zakaullah M 2017 Novel duplex cathodic plasma nitriding of non-alloyed steel using aluminum and austenitic steel cathodic cages J. Alloys Compd. 721 307–11
[6] Razzak M A 2011 Heat treatment and effects of Cr and Ni in low alloy steel Bull. Mater. Sci. 34 1439–45
[7] Solecia M and Salak A 1999 Durability and failure of powder forged rolling bearing rings Wear 236 47–54
[8] de Andrade Manfredini A P, de Godoy G C D and de Arruda Santos I 2017 Structural characterization of plasma nitried interstitial-free steel at different temperatures by SEM, XRD and Rietveld method Journal of Materials Research and Technology 6 65–70
[9] Naeem M, Iqbal J, Abrar M, Khan K H, Díaz-Guillén J C, Lopez-Badillo C M, Shaﬁq M, Zaka-ul-Islam M and Zakaullah M 2018 The effect of argon admixing on nitriding of plain carbon steel in N2 and N2-H2 plasma Surface and Coatings Technology 350 48–56
[10] Gharavi M A et al 2018 Microstructure and thermoelectric properties of CrN and CrN/CrN thin ﬁlms Journal of Physics D: Applied Physics 51 355302
[11] Almeida A B D 2013 Detecção da transformação da austenita retida por deformação plástica em aços para gasodutos classe API 5L X80 através de medidas magnéticas Universidade de São Paulo, Master’s Dissertation (https://doi.org/10.11606/D.3.2013.tde-17102014-120751)
[12] de Ara J F, Almazor E, Palacio J F, Fuentes G G, Rodriguez R J and García J A 2014 Inﬂuence of temperature in arc-activated plasma nitriding of maraging steel in solution annealed and aged conditions Surf. Coat. Technol. 258 754–62
[13] Bell T, Sun Y and Suhadi A 2000 Environmental and technical aspects of plasma nitrocarburising Vacuum 59 14–23
[14] D L Heras E, Ybarra G, Lamas D, Cabo A, Dalilbon E L and Brühl S P 2017 Plasma nitriding of 316L stainless steel in two different N2–H2 atmospheres-Influence on microstructure and corrosion resistance Surface and Coatings Technology 313 47–54
[15] Lampe T, Eisenberg S and Cabo E R 2003 Plasma surface engineering in the automotive industry—trends and future prospectives Surf. Coat. Technol. 174 1–7
[16] Braceras I, Ibáñez I, Dominguez-Meister S, Sánchez-Garcia J A, Brizuela M, Larrañaga A and Garmendia I 2018 Plasma nitriding of the inner surface of stainless steel tubes Surf. Coat. Technol. 355 116–22
[17] Khusainov Y G, Ramazanov K and Budilov V 2016 Technology of local ion nitriding of chrome-nickel steel in glow discharge with hollow cathode effect 2016 27th Int. Symp. on Discharges and Electrical Insulation in Vacuum (ISDEIV) (Piscataway, NJ) (IEEE)
[18] Razi H, Ashrafzadeh F, Hosseini S R, Ghomashchi K and Liu R 2017 Characterization of simultaneous aged and plasma nitrided 17-4 PH stainless steel Mater. Charact. 133 33–43
[19] Zdralevá E, Slota J, Solfronk P and Kolnerová M 2017 Evaluation of the effect of different plasma-nitriding parameters on the properties of low-alloy steel J. Mater. Eng. Perform. 26 3588–96
[20] Khoob K, Takeuchi M, Onuki J and Urao R 2004 Microwave plasma nitriding of hollow tube inner wall Materials Transactions 45 599–601
[21] Borisuyk Y V, Kozlova V V, Mozgrin D V, Nenashev P S, Oreshnikova N M, Berdnikova M M and Psarev A A 2017 Pulsed abnormal glow discharge with hollow cathode for nitriding of internal cylindrical surfaces Journal of Physics: Conf. Series Vol 941p012036(Bristol) (IOP Publishing)
[22] Davis R R 2002 Surface Hardening of Steels: Understanding the Basics. (ASM International)
[23] Karaglou S 2002 Structural characterization and wear behavior of plasma-nitrided AISI 5140 low-alloy steel Mater. Charact. 49 349–57
[24] Kim Y M and Han J G 2003 Spectroscopic study for pulsed DC plasma nitriding of narrow deep holes Surf. Coat. Technol. 171 205–10
[25] Alves C J, de Andicha Rodrigues J and Martinelli A E 2000 Growth of nitrided layers on Fe–Cr alloys Materials Science and Engineering: A 279 10–5
[26] Sohi M H, Ebrahimii M, Raouf A H and Mahboubi F 2018 Comparative study of the corrosion behaviour of plasma nitrocarburised AISI 4140 steel before and after post-oxidation Mater. Des. 31 4432–7
[27] Alves C Jr, Lima J A, Hajek V, da Cunha J M and dos Santos C A 2007 Effect of cooling rate on properties of plasma nitrided AISI 1010 steel Surf. Coat. Technol. 201 7566–73

[28] Kamoona S S N 2016 Low Temperature Nitriding Heat Treatment Process of Austenitic Stainless Steel Grade Journal of Environmental Science, Computer Science and Engineering & Technology 15 501–10

[29] Diao X G, Li F S, Zhao Z J and Zhou S Q 1996 Preparation and characterization of γ-Ni₅Fe₅N Mater. Sci. Lett. 15 1590–1

[30] Friedlaender F and Leliakov I 1961 Initiation of flux reversal in magnetic-amplifier cores Transactions of the American Institute of Electrical Engineers, Part I: Communication and Electronics 80 269–72

[31] Cartier M 2003 Handbook of Surface Treatments and Coatings (HEF Group: Professional Engineering Publishing)

[32] Alves C Jr, da Silva E and Martinelli A 2001 Effect of workpiece geometry on the uniformity of nitrided layers Surf. Coat. Technol. 139 1–5

[33] Hirsch T, Clarke T and da Silva Rocha A 2007 An in situ study of plasma nitriding Surf. Coat. Technol. 201 6380–6

[34] Jeong B-Y and Kim M-H 2001 Effects of the process parameters on the layer formation behavior of plasma nitrided steels Surf. Coat. Technol. 141 182–6

[35] Mahboubi F and Abdolvahabi K 2006 The effect of temperature on plasma nitriding behaviour of DIN 1.6959 low alloy steel Vacuum 81 239–43

[36] Esfandiar M and Dong H 2007 Improving the surface properties of A286 precipitation-hardening stainless steel by low-temperature plasma nitriding Surf. Coat. Technol. 201 6189–96

[37] Budinski K G and Budinski M K 2009 Engineering materials Nature 25 28

[38] Hutchings I and Shipway P 2017 Tribology: Friction and Wear of Engineering Materials. (Butterworth-Heinemann)

[39] Inokuti Y, Nishida N and Ōhashi N 1975 Formation of Fe₃N, Fe₄N and Fe₁₆N₂ on the surface of iron Metall. Trans. A 6 773

[40] Straffelini G, Avi G and Pellizzari M 2002 Effect of three nitriding treatments on tribological performance of 42CrAlMo7 steel in boundary lubrication Wear 252 870–9

[41] Sun Y and Bell T 1991 Plasma surface engineering of low alloy steel Materials Science and Engineering: A. 140 419–34

[42] Xi Y-T, Liu D-X and Han D 2008 Improvement of corrosion and wear resistances of AISI 420 martensitic stainless steel using plasma nitriding at low temperature Surf. Coat. Technol. 202 2577–83