

Effects of Chrome Plating, Boriding, Nitrocarburizing, Nitriding and Post-oxidation Treatments on The Tribological Properties of DIN 32CrMoV12-10 Steel

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Abstract

In this study, the effect of different surface treatments was investigated to improve the friction and wear properties of DIN 32CrMoV12-10 steel. For this purpose, chrome plating, boriding, nitrocarburizing+oxidation, nitriding+oxidation processes were applied to the samples prepared from DIN 32CrMoV12-10 steel. After the procedures, the structural, morphological and mechanical properties of the samples were determined by XRD, SEM, hardness measurement device and profilometer. In order to determine the tribological properties of the samples, pin-on-flat wear tests were carried out. It was observed that a chromium layer was formed on the surface of the material after chrome plating. In boronized samples, it was observed that a boron layer was formed on the surface of the material and a diffusion layer was formed below it. The formed layers contain Fe₂B and FeB phases. In nitrocarburized+oxidized and nitrided+oxidized samples, it was observed that the oxide layer on the material surface, a white layer just below it and a diffusion zone extending to the inner parts of the material were formed. The oxide layer formed on the surface consists of Fe₂O₃ and Fe₃O₄ phases, while the white layer and diffusion layers consist of ε-Fe₂₋₃N and γ'-Fe₄N phases. Due to the increased plastic deformation resistance with oxide layers, hard nitride phases and diffusion layers, the highest wear resistance was obtained from nitrocarburized+oxidized and nitrided+oxidized samples.

Keywords: Chrome plating, boronizing, nitrocarburizing, nitriding, oxidation, duplex treatment

Krom Kaplama, Borlama, Nitrokarbürleme, Nitrürleme ve Post-oksidasyon İşlemlerinin DIN 32CrMoV12-10 Çeliğinin Tribolojik Özelliklerine Etkileri

Öz

Bu çalışmada, DIN 32CrMoV12-10 çeliğinin sürtünme ve aşınma özelliklerinin iyileştirilmesi amacı ile farklı yüzey işlemlerinin etkisi araştırılmıştır. Bu amaçla, DIN 32CrMoV12-10 çeliğinden hazırlanan numunelere krom kaplama, borlama, nitrokarbürleme+oksidasyon, nitrürleme+oksidasyon işlemleri uygulanmıştır. İşlemler sonrasında, numunelerin yapısal, morfolojik ve mekanik özellikleri XRD, SEM, sertlik ölçüm cihazı ve profilometre ile belirlenmiştir. Numunelerin tribolojik özelliklerinin belirlenmesi amacı ile de pin-on-flat aşınma testleri gerçekleştirilmiştir. Krom kaplama sonrasında malzeme yüzeyinde krom tabakasının oluştuğu görülmüştür. Borlanmış numunelerde; malzeme yüzeyinde bor tabakası oluşumu ve düzey altında ise difüzyon tabakasının oluştuğu gözlemlenmiştir. Oluşan tabakalar Fe₂B ve FeB fazlarını içermektedir. Nitrokarbürleme+oksidasyon ve nitrürleme+oksidasyon işlemi uygulanmış numunelerde ise malzeme yüzeyinde en üstte oksit tabakası olacak şekilde bunun hemen altında beyaz tabaka ve malzemenin iç kısımlarına uzanan difüzyon bölgesinin oluştuğu görülmüştür. Yüzeyde oluşan oksit tabakası Fe₂O₃ ve Fe₃O₄ fazlarından oluşmakta iken beyaz tabaka ve difüzyon tabakaları ε-Fe₂₋₃N ve γ'-Fe₄N fazlarından oluşmaktadır. Oksit tabakaları, sert nitrür fazları ve difüzyon tabakaları ile artan plastik deformasyon direnci sebebiyle en yüksek aşınma direnci nitrokarbürleme+oksidasyon ve nitrürleme+oksidasyon işlemi uygulanmış numunelerden elde edilmiştir.

Anahtar Kelimeler: Krom kaplama, borlama, nitrokarbürleme, nitrürleme, oksidasyon, dubleks işlem

1. Introduction

Damages caused by friction and wear cause material and energy loss, and machine parts and structures cannot fulfill their duties due to these mechanisms. The wear damage mechanism is related to the surface properties of the materials. For this reason, it is necessary to improve the surface properties of the materials in order to eliminate or minimize the damage caused by wear. For this purpose, surface treatments are used that allow the creation of extra layers or structures on the surface of the materials. The selection of these surface treatments is determined by the type of feature to be gained. For this purpose, many different chemical, physical, electrochemical, thermo-chemical etc. methods are used to increase the wear, corrosion, fatigue and impact resistance of the surfaces of the materials (Burlacov et al., 2017; Genel, 2006; Ibrahim & Berndt, 2007; Remešová et al., 2020; Salgueiredo et al., 2013; Terres, Mohamed, & Sidhom, 2010; Xiang, Jonsson, Hedström, Zhu, & Odqvist, 2021).

DIN 32CrMoV12-10 steel is frequently preferred especially in high temperature applications because of its mechanical properties. DIN 32CrMoV12-10 has high toughness, hardness, impact resistance and shock resistance. For these reasons, it is frequently used as tool and mold material. (Qamar, 2015; Temmler et al., 2020). Despite these features, tools or molds made of DIN 32CrMoV12-10 steel can be subject to frequent wear during use. For this reason, as mentioned before, various surface treatments are applied to improve the tribological properties of tool steels. In the literature, there are different studies on this subject. In a study on plasma nitriding: it was stated that in case of nitriding treatment, wear properties would be improved due to increased surface hardness (Forati Rad, Amadeh, & Moradi, 2011). In another study, it was observed that post-oxidation treatment applied together with plasma nitriding increased the corrosion resistance of the material (Borgioli, Galvanetto, Fossati, & Bacci, 2003). Other studies on nitriding have generally indicated that the increased surface hardness and the phases formed after the process increase the wear behavior of the material (Karamş, Yildizli, & Çarkt Aydın, 2012; Tillmann, Lopes Dias, & Stangier, 2019; Yazdani, Soltanieh, Aghajani, & Rastegari, 2010; Yilbas & Nizam, 2000). In various studies examining the effects of nitrocarburizing process on the friction and wear behavior of tool steels, it was determined that the increase in surface hardness and the change in phase structure increase the wear properties of the materials, similar to the nitriding process (Chiu, Wu, & Chang, 2002; Fazlalipour, Shakib, Shokuhfar, & Niki Nushari, 2012; Yan, Zhao, Chen, Hu, & Yan, 2020; Zhang et al., 2014; Zlatanović, Popović, Bogdanov, & Zlatanović, 2004). In addition, it is stated in the literature that the chrome plating process will increase the wear properties of the materials if the required current density and anode-cathode distance are adjusted properly (Kir & Apay, 2019). In studies on boriding of tool steels, it has been shown in different studies that the increase in hardness and diffusion zone caused by boriding process increase the wear, corrosion and fatigue properties of materials (Balusamy, Sankara Narayanan, Ravichandran, Song Park, & Lee, 2013; Erdogan, 2019; Gök et al., 2017; Günen, 2020; P Jurči, Hudáková, & Kusý, 2012; Peter Jurči & Hudáková, 2009, 2011, 2020; Mishigdorzhyn, Chen, Ulakhanov, & Liang, 2020).

Although there are many studies on the surface treatment of tool steels in the literature, there are limited studies on nitriding, nitrocarburizing, boron plating and chrome plating of DIN 32CrMoV12-10 steel (Pokorný et al., 2011; Pokorny, Dobrocky, & Joska, 2020).

For this reason, in this study, the effects of chrome plating, boronizing, nitrocarburizing+oxidation, nitriding+oxidation processes on DIN 32CrMoV12-10 steel were investigated in detail. Chromium plating, boronizing, nitrocarburizing+oxidation, nitriding+oxidation processes were applied to DIN 32CrMoV12-10 steel and the effects of these processes on the structural, mechanical and tribological properties of DIN 32CrMoV12-10 steel were investigated. In addition, the effects of these processes on the tribological properties of DIN 32CrMoV12-10 steel were investigated comparatively. For these examinations, XRD, SEM, hardness tester, profilometer and wear tester were used.

2. Material and Method

In this study, DIN 32CrMoV12-10 steel was used and the chemical composition of the steel is given in Table 1. Samples were prepared before surface treatments. Cylindrical specimens with a diameter of 30 mm and a thickness of 5 mm were used in the study. Before the surface treatments, the samples were polished with 80-1200 mesh size abrasives and then polished with 3 µm alumina powders. After this step, the samples were cleaned with ethanol and dried.

Table 1. Chemical composition of DIN 32CrMoV12-10 steel

Element	Weight, %
C	0.36
Mn	0.51
Si	0.30
Cr	2.49
Ni	0.06
Mo	0.90
V	0.21
Cu	0.04
P	0.02
S	0.01
Fe	Remain

Four different surface treatments were applied to the prepared samples. The information about these treatments is as follows:

- **Chrome plating:** Chromium plating processes were carried out in a chrome plating bath. Hard chrome plating bath conditions are as follows: Operating temperature: 50-55 °C, Current density: 47-56 amp/dm², Chromic acid: 250-300 grams /lt, Sulfuric acid: 2-3 grams/lt, Anode: Pb.
- **Boronizing:** The samples were subjected to boriding processes at 850 °C for 6 hours with the pack boronizing method under nitrogen atmosphere conditions.
- **Nitrocarburizing+Oxidation:** The samples were subjected to nitrocarburization processes at 560°C for 4 hours under KN:3, CO₂ 5% conditions. Afterwards, the oxidation process was applied to the samples for 90 minutes. Here, KN is described as

nitriding potential that is a derived measurement of an atmosphere's potential to allow for the diffusion of nitrogen into the material. $K_N = p\text{NH}_3 / (p\text{H}_2)^{3/2}$; where, p denotes partial pressure.

- **Nitriding+Oxidation:** The samples were subjected to nitriding processes at 480°C for 4 hours under KN:3. Afterwards, the oxidation process was applied to the samples for 90 minutes.

Phase analyzes of the samples were performed with PANalytical EMPYEAN branded X-Ray diffractometer. The measurement parameters are as follows: 4 KW (max 60 kV, max 100 mA), 15-60 kV voltage and 20°-100° measurement range. Cross-sectional imaging and post-wear surface imaging of the samples were performed with the ZEISS Sigma 300 Scanning Electron Microscope (SEM). KLa Tencor Stylus Profiler P7 profilometer was used for surface roughness measurements. Future Tech FM 300E brand hardness tester was used for the hardness measurements of the samples. Hardness measurements were carried out using the Vickers method with 300 gr load, 15-micron penetration depth and 15 seconds waiting time.

Wear tests were carried out to determine the wear characteristics of untreated and surface-treated samples. Wear tests were carried out with TURKYUS pin-on-flat wear tester in dry conditions and at room temperature ($\approx 21^\circ\text{C}$). In the wear tests, a 6 mm diameter Al_2O_3 wear ball was used as the pin and the tests were carried out for 5000 seconds for a length of 141 m with a 5N load and 8 mm track diameter.

3. Results and Discussion

XRD graphics of untreated and surface treated samples are given in Figure 1. α -Fe peaks were observed in the untreated sample (Fig. 1-a). Fe_2B and FeB peaks were observed in the boronized sample (Figure 1-b). In addition, the α -Fe peak from the substrate material was also observed in this sample. Only chrome peaks were observed in the chrome-plated sample (Figure 1-c). XRD graphs of nitrocarburized+oxidized and nitrided+oxidized samples are given in Figure 1-d and Figure 1-e, respectively. From these graphs, it is seen that ϵ - Fe_{2-3}N and γ' - Fe_4N phases are formed on the surface of the samples after treatment. These results are in agreement with the literature studies (Forati Rad et al., 2011; Karakan, Alsaran, & Çelik, 2004). In addition, α -Fe peaks from the substrate (nitrocarburized+oxidized and nitrided+oxidized) were observed in both sample groups. However, since post-oxidation process is applied after nitriding and nitrocarburizing processes in both sample groups; It was observed that Fe_2O_3 and Fe_3O_4 phases were formed.

Cross-sectional images and average section thickness values of the samples are given in Figure 2 and Table 2, respectively. As can be seen in Figure 2 and Table 2, a chrome layer of approximately 7 μm was formed from the material surface after the chrome plating process. After the boriding process, it is seen that a 4 μm boron layer is formed in the sample. Also, unlike the chrome plating, a diffusion layer of 120 μm was obtained in this sample, since boriding occurs by diffusion. On the other hand, an average of 10 μm and 12 μm white layer and 150 μm and 180 μm diffusion layers were obtained in nitrocarburized+oxidized and nitrided+oxidized samples, respectively. In addition to these, it was observed that an oxide layer of about 3 μm was formed in both samples after post-oxidation treatment.

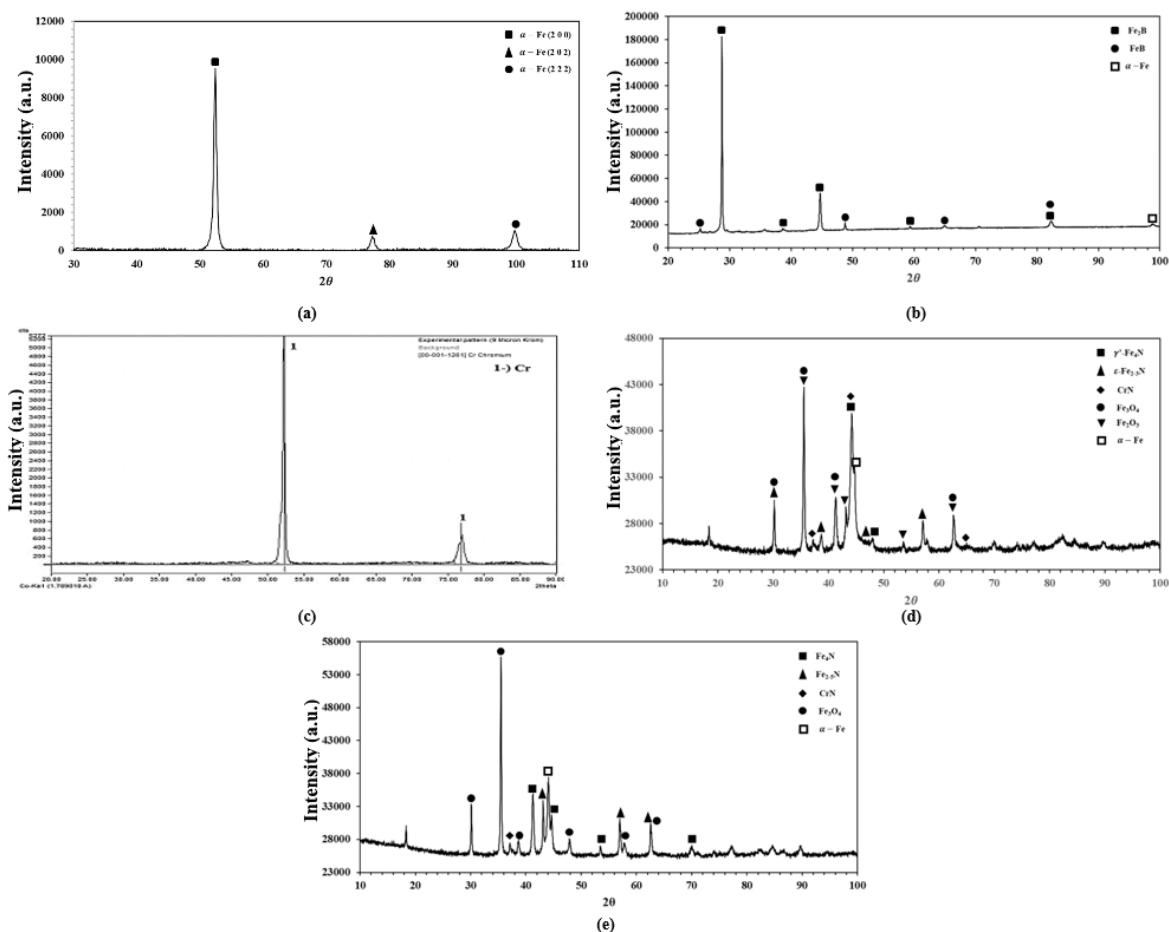


Figure 1. XRD graphs of (a) untreated, (b) boronized, (c) chrome plated, (d) nitrocarburized+oxidized and (e) nitrided+oxidized samples

After the applied surface treatments, depending on the type of process, different layer hardness values were obtained in the samples (Table 2). While the hardness for the untreated sample is 320 HV, the surface hardness as a result of chrome plating and boronizing is at 580 HV and 1100 HV, respectively. From these results, it is understood that boriding after both processes causes more hardness increase. This situation can be explained by two reasons. The first of these is that the Fe₂B and FeB phases formed on the surface as a result of boriding have higher hardness than chromium. The other reason is that the diffusion layer formed as a result of boriding increases the load carrying capacity of the material. Since the layer thicknesses of nitrocarburized+oxidized and nitrided+oxidized samples were close to each other, hardness values of 920 HV and 980 HV were obtained from these samples, respectively. However, the surface hardness of the boronized sample is higher than these samples. Although all three processes are based on diffusion, the reason why the surface hardness of boronized samples is higher than the other samples; the layers formed are different in terms of chemical content. Fe₂B and FeB based surface and subsurface layers formed as a result of boriding have higher hardness than structures formed by other processes. For this reason, the highest surface hardness was obtained as a result of boriding. In addition to these, an increase was observed in the surface roughness values of the samples after surface treatments, and it was observed that the surface roughness values of the treated samples were close to each other.

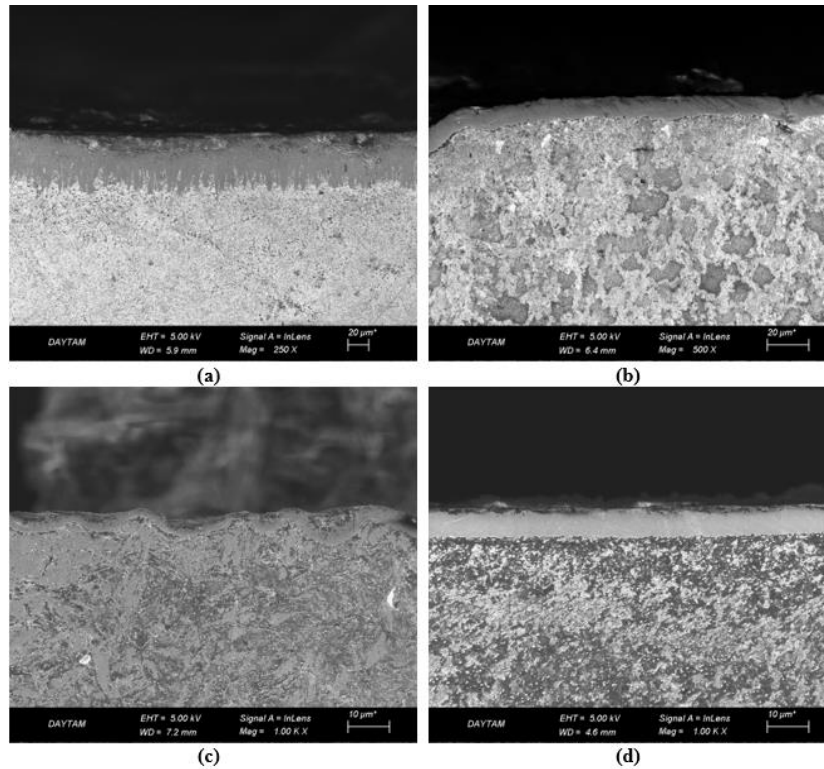


Figure 2. SEM images of (a) boronized, (b) chrome plated, (c) nitrocarburized+oxidized and (d) nitrided+oxidized samples

Table 2. The hardness, layer thickness and surface roughness of the untreated and surface treated samples

	Mean Surface Hardness (HV)	Mean Layer Thickness (µm)				Mean Surface Roughness-Ra (µm)
		White	Diffusion	Oxide	Boron/Chrome	
Untreated	320	-	-	-	-	0.09
Chrome plated	580	-	-	-	7	0.18
Boronized	1100	-	120	-	4	0.22
Nitrocarburized+oxidized	920	10	150	3	-	0.34
Nitrided+oxidized	980	12	180	3	-	0.36

Friction coefficient-time graphs of untreated and surface treated (chrome plated, boronized, nitrocarburized+oxidation and nitrided+oxidation) samples are given in Figure 3. As can be seen from here, the coefficient of friction for untreated DIN 32CrMoV12-10 steel is approximately 0.35, while this value is approximately 0.41, 0.50, 0.55 and 0.52 for chrome plated, boronized, nitrocarburized+oxidized and nitrided+oxidized samples, respectively. From this point of view, it is seen that the friction coefficient increases with the application of surface treatments. This can be explained by the increase in surface roughness after the processes. The average surface roughness values of the untreated, chrome-plated, boronized, nitrocarburized+oxidation and nitriding+oxidation treated samples were measured as 0.09, 0.18, 0.22, 0.34 and 0.36 (Ra-µm), respectively. It was seen that the friction coefficients increased with increasing surface roughness. In addition, the highest coefficient of friction

values were obtained from the samples treated with nitrocarburizing+oxidation and nitriding+oxidation.

In addition to these, another remarkable point is the variation of the friction coefficient with time. While the coefficient of friction in all samples did not show a stable behavior especially from the beginning of the wear until a certain moment (0-1000 seconds), it showed a stable behavior in the range of 1000-5000 seconds. In this regard, it was thought that the stick-slip event was an effective mechanism in the first stages of wear, especially in the first 250 seconds. In the later stages, it was seen that the wear/friction coefficient exhibited a more stable behavior with the effect of the transfer film formed on the pin.

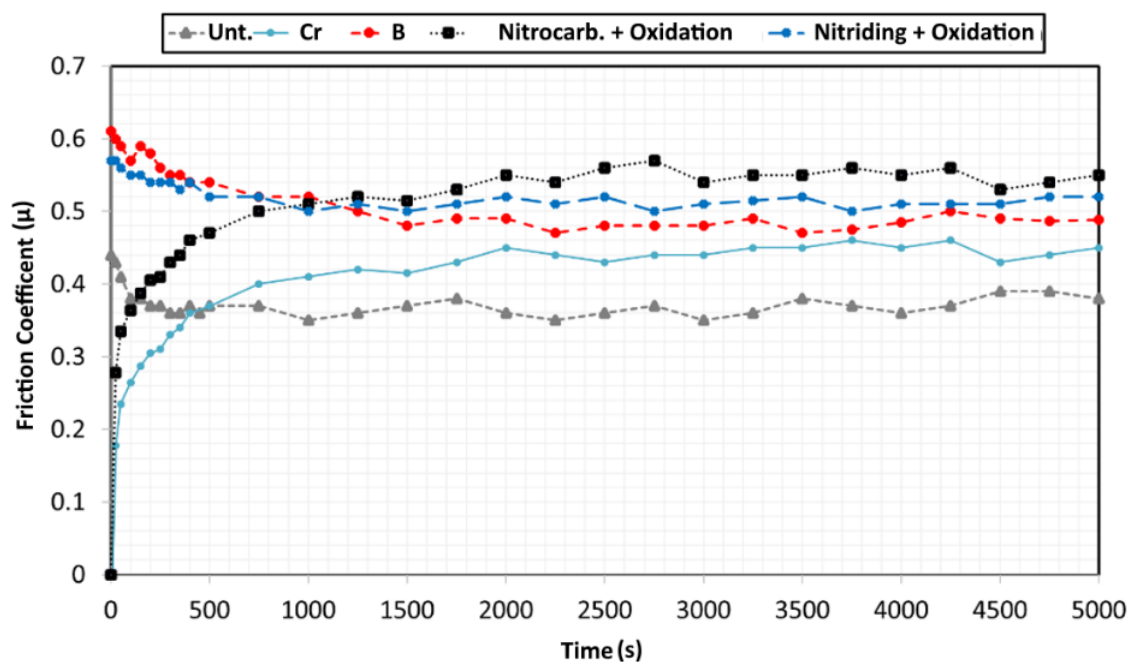


Figure 3. Friction coefficient-time graphs of untreated and surface treated samples

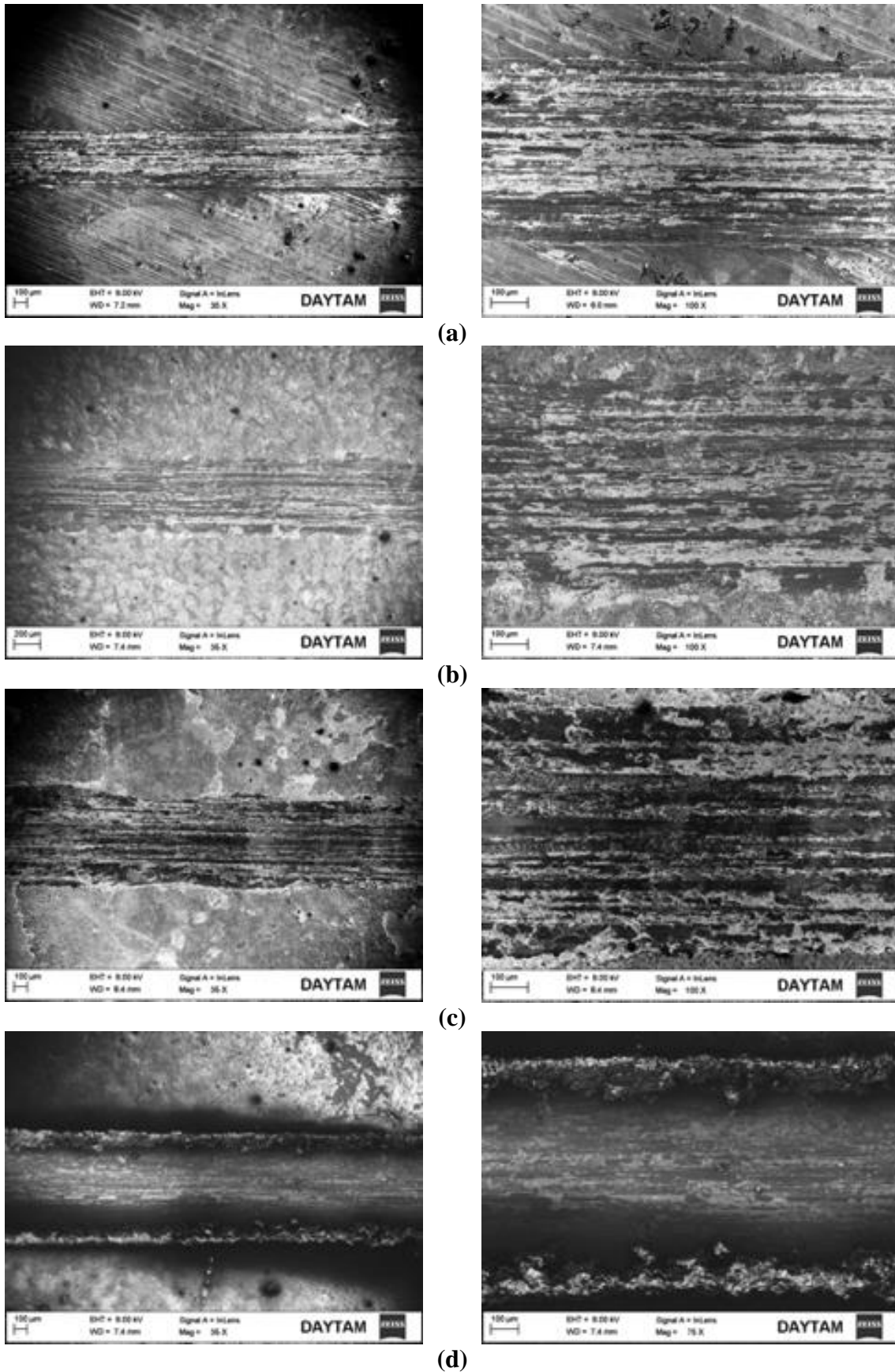
Surface SEM images of untreated and surface treated (chrome plated, boronized, nitrocarburized+oxidation treated and nitrided+oxidation treated) samples after wear tests are given in Figure 4. It can be seen that the wear is narrow and stable in the untreated sample (Figure 4-a). When the SEM image of the untreated sample was examined in detail (Figure 4-b), it was observed that the surface was crushed and partially scratched. Also, piles were observed at the edges of the wear track due to the effect of the wear ball. This indicates that the untreated sample is exposed to a high level of plastic deformation during wear due to the high contact pressure (Hertz pressure). As a result of this excessive plastic deformation, substrate piled up on the pin and formed a transfer film, resulting in adhesive wear. In addition, it was observed that there were scratches on the surface of the material as a result of the particles breaking off from the substrate during wear scratching the surface. It is understood that the substrate is subject to very low abrasive wear. When these conditions are examined together for the untreated sample, it can be said that this sample is exposed to adhesive wear.

When the surface SEM images of the chrome plated, boronized, nitrocarburizing+oxidation and nitriding+oxidation treated samples are examined after wear, similar to the untreated samples, it was observed that the wear in these samples took place in a narrow area and stable (Figure 4-b, c, d and e). However, when the surface detail images of these samples were examined, it was observed that there was either very little accumulation on the edges of the wear track or no material accumulation at all. It can be concluded that the resistance of these samples to plastic deformation caused by wear is higher than the untreated sample. Because, as mentioned before, as a result of each applied surface treatment, hard and homogeneous structures are formed on the surface of the samples and under the surface. As a result, the hardness of the material increases at the surface and near the surface (Castro, Fernández-Vicente, & Cid, 2007; Yetim, Celik, & Alsaran, 2010). As a result of this increase, the resistance of the materials to plastic deformation increased and no material accumulation was observed on the edges of the wear scar.

In addition to these, another remarkable point for the surface treated samples is the scratches formed within the wear marks. Although there are hardly any scratches in the wear trace in the untreated sample, it is observed that very high levels of scratches and traces occur within the wear trace in the treated samples, except for the chrome-plated sample. These scratches and marks can be considered as signs of abrasive wear. Because, as mentioned before, as a result of surface treatments, hard phases and structures with much higher hardness than the hardness of the material have been formed on the surface of the material and/or just under the surface. These structures are hard and brittle. For this reason, since these structures could not show plastic deformation with the effect of the applied wear load, they were directly broken and showed abrasive effect by forming scratches in the wear trace with pin movement in the wear trace. It can be said that the wear mechanism changes from adhesive to abrasive wear as a result of applied surface treatments. Although the treated samples showed adhesive wear, it can be said that the mechanism causing wear in these samples is abrasive wear. However, the wear surface appearance of the chrome plated sample is relatively similar to the untreated sample. As it is known, the chrome formed on the surface after the chrome plating process has a lower hardness value than the phases and structures formed on the surface after other surface treatments. For this reason, the wear mechanism for this sample was adhesive. Based on the research findings obtained, it was concluded that the main damage mechanism for the untreated and chrome-plated samples after the wear tests was adhesive and for the other samples abrasive.

Another situation observed as a result of SEM examinations is that the samples treated with nitrocarburizing+oxidation and nitriding+oxidation have a narrower wear track width than the untreated and other treated samples. In these samples, oxidation process was carried out after nitrocarburizing and nitriding processes. As a result of these processes, an additional oxide layer was formed on the surface of the relevant samples, which would resist wear more than the other samples. It is thought that this layer, which has a high resistance to plastic deformation with its hardness, causes the wear to occur in a narrower trace width compared to other samples.

The wear rates of the untreated and surface treated (chrome plated, boronized, nitrocarburized+oxidized and nitrided+oxidized treated) samples are given in Figure 5. It can



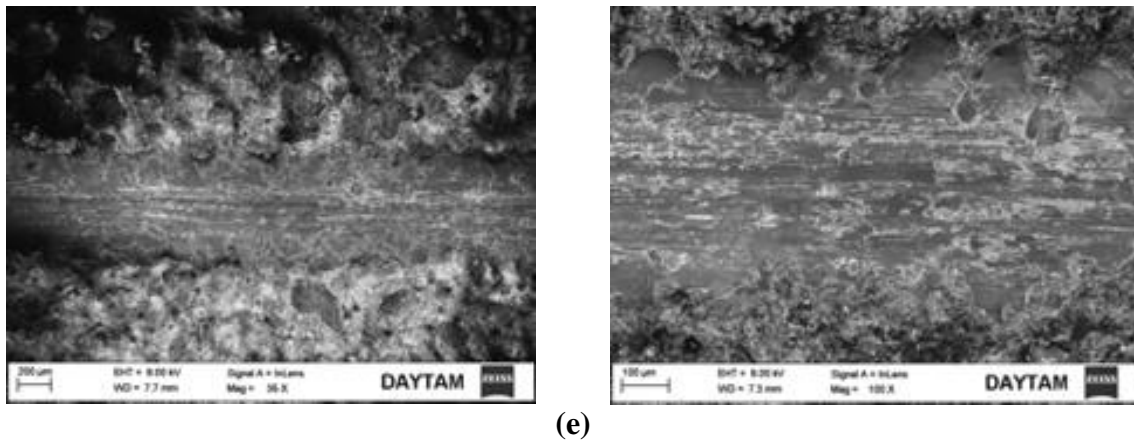


Figure 4. SEM images after wear tests: (a) untreated, (b) chrome plated, (c) boronized, (d) nitrocarburized+oxidized (e) nitrided+oxidized

be seen that the wear rates decrease as a result of each applied surface treatment compared to the untreated sample. The improvement of wear rate in chrome-plated sample is low compared to other processes. This situation also supports the wear SEM images obtained after wear tests. Because, the wear image and mechanism similar to the untreated sample were obtained in the chrome-plated sample. It was concluded that the chrome plating process did not change the wear rate at a very high level. However, wear rates of boronized, nitrocarburized+oxidized and nitrided+oxidized samples decreased by almost 50% compared to untreated samples. From this results, it is concluded that these processes increase the wear resistance of DIN 32CrMoV12-10 steel. This situation can be attributed to hard layers formed on the surface and under the surface of the material after these processes. As mentioned before, they prevent the plastic deformation of the materials and thus increase the wear resistance. In addition, as seen in Figure 5, the wear rate of the samples treated with nitrocarburizing+oxidation and nitriding+oxidation treatments decreased the wear rates more than the other samples. The reason for this is the oxide layer formed on the surface, which causes an increase in the resistance against plastic deformation.

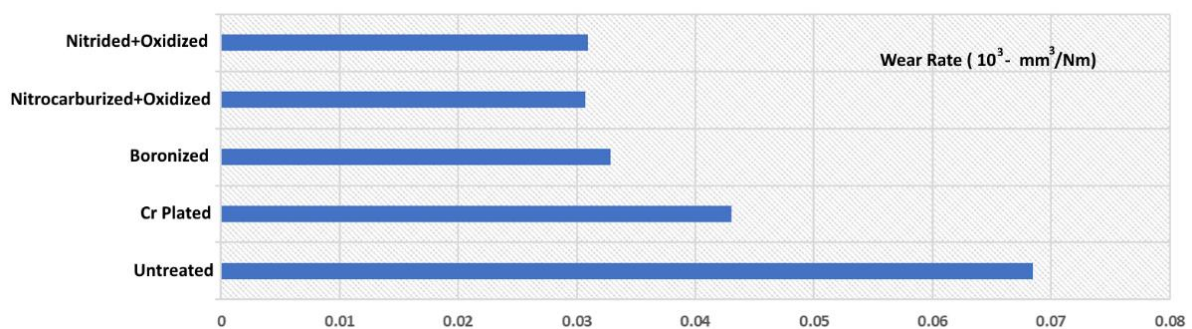


Figure 5. Wear rates of untreated and surface treated samples after wear tests

Thanks to this layer, the wear resistance of nitrocarburized+oxidized and nitrided+oxidized samples increased. This increase is higher for these samples compared to the boronized samples. Although the highest hardness value was realized in boronized samples, the oxide layer on the surface of nitrocarburized+oxidized and nitrided+oxidized samples created an extra barrier for friction and wear, resulting in a decrease in material loss during wear.

Therefore, these samples showed similar friction and wear behavior and had a lower wear rate than the boronized sample.

4. Conclusions

In this study, the effects of chrome plating, boronizing, nitrocarburizing+oxidation and nitriding+oxidation processes on the friction and wear properties of DIN 32CrMoV12-10 steel were investigated. The obtained results are given below:

- While α -Fe peaks were observed in the untreated sample, Fe_2B and FeB, chrome peaks and ϵ - Fe_{2-3}N and γ' - Fe_4N phases were obtained from the boronized, chrome-plated, nitrocarburized+oxidized and nitrided+oxidized samples. Since the post-oxidation process was applied, it was observed that Fe_2O_3 and Fe_3O_4 phases were formed in nitrided and nitrocarburized samples.
- The surface hardness of the material increased after the applied processes and the highest surface hardness was obtained from boronized samples due to both being a surface treatment created by diffusion and the high hardness of the boron-containing layer formed on the surface.
- It was observed that only a chromium layer was formed on the surface of the chrome-plated samples after surface treatments. In other samples, in addition to the boron layer and white layer formation on the surface of the materials, a diffusion layer was formed.
- It was found that the applied surface treatment changed the surface roughness of the material. While untreated sample showed the lowest roughness, duplex treated samples showed the highest roughness.
- Due to the increased surface roughness of the surface treated samples, an increase was observed in the friction coefficients compared to the untreated sample.
- The main damage mechanism in untreated and chrome plated samples is adhesive wear due to low plastic deformation resistance. In the other samples, the wear mechanism changed from adhesive to abrasive wear due to the effect of hard wear particles detached from the surface during wear.
- Nitrocarburized+oxidized and nitrided+oxidized samples showed the highest wear resistance due to increased plastic deformation resistance with oxide layers, hard nitride phases and diffusion layers on their surfaces.

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