

EFFECT OF SLIDING SPEED ON THE FRICTIONAL BEHAVIOR AND WEAR PERFORMANCE OF BORIDED AND PLASMA-NITRIDED W9Mo3Cr4V HIGH-SPEED STEEL

VPLIV HITROSTI DRSENJA NA VEDENJE IN OBRABO BORIRANEGA IN V PLAZMI NITRIRANEGA HITROREZNEGA JEKLA W9Mo3Cr4V

Ibrahim Gunes

Department of Metallurgical and Materials Engineering, Faculty of Technology, Afyon Kocatepe University, 03200 Afyonkarahisar, Turkey
igunes@aku.edu.tr

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This study investigated the effect of sliding speed on the friction and wear behaviors of plasma-nitrided (PN) and borided (W9Mo3Cr4V) steels. The PN process was carried out in a dc-plasma system at a temperature of 923 K for 6 h in a gas mixture of 80 % N₂-20 % H₂ under a constant pressure of 5 mbar. The boriding process was carried out in Ekabor-II powder at a temperature of 1223 K for 6 h. X-ray diffraction analysis on the surface of the PN and borided steels revealed the presence of FeB, Fe₂B, CrB, MoB, WB, FeN, Fe₂N, Fe₃N and Fe₄N compounds. The wear tests were carried out in a ball-disc arrangement under dry friction conditions at room temperature with an applied load of 10 N and with sliding speeds of (0.1, 0.3 and 0.5) m/s at a sliding distance of 1000 m. The wear surfaces of the samples at the different sliding speeds were analyzed using SEM microscopy and X-ray energy-dispersive spectroscopy EDS. The friction coefficients of the nitrided and borided W9Mo3Cr4V steels varied from 0.42 to 0.61, and from 0.33 to 0.52, respectively. As a result of the wear, the friction coefficient was observed to decrease with an increase in the sliding speeds of the PN and the borided AISI W9Mo3Cr4V steel while an increase was observed in the wear resistance.

Keywords: plasma nitriding, boriding, W9Mo3Cr4V, sliding speed, wear rate

V tej študiji je bil preučevan vpliv hitrosti drsenja na trenje in vedenje v plazmi nitriranega (PN) in boriranega jekla (W9Mo3Cr4V). PN-postopek je bil izvršen v enosmernem plazemskem sistemu 6 h na temperaturi 923 K in v plinski mešanici 80 % N₂-20 % H₂ pri konstantnem tlaku 5 mbar. Postopek boriranja je bil izvršen s prahom Ekabor-II pri temperaturi 1223 K in trajanju 6 h. Rentgenska difrakcijska analiza na površini PN in boriranega jekla je odkrila spojine FeB, Fe₂B, CrB, MoB, WB, FeN, Fe₂N, Fe₃N in Fe₄N. Preizkus obrabe je bil izvršen na napravi s kroglo na plošči pri suhem trenju na sobni temperaturi z obtežbo 10 N in s hitrostjo drsenja (0,1, 0,3 in 0,5) m/s pri razdalji drsenja 1000 m. Obraba na površini pri vzorcih z različno hitrostjo drsenja je bila analizirana s SEM-mikroskopijo in rentgensko energijsko disperzijsko spektroskopijo. Koeficient trenja nitriranega in boriranega jekla W9Mo3Cr4V je bil med 0,42 in 0,61 oziroma 0,33 in 0,52. Kot rezultat obrabe PN in boriranega jekla AISI W9Mo3Cr4V se je koeficient trenja zmanjševal pri naraščanju hitrosti drsenja, odpornost proti obrabi pa se je povečala.

Ključne besede: nitriranje v plazmi, boriranje, W9Mo3Cr4V, hitrost drsenja, stopnja obrabe

1 INTRODUCTION

Plasma nitriding and boriding are thermo-chemical surface treatments widely used to improve the tribo-mechanical properties of engineering components via a modification of their surface microstructure.¹⁻⁶ During the plasma-nitriding process, the nitriding reaction not only occurs on the surface but also in the sub-surface owing to the long-distance diffusion of nitrogen atoms from the surface towards the core. Nitrogen diffused into a steel surface is combined with alloying elements to form a fine dispersion of alloy nitrides.^{7,8} As a result, two different structures have been identified, the so-called "white" or "compound" layer and the "diffusion zone". The first one is the outermost layer, and consists of one or two iron nitrides (Fe₄N-Fe_{2,3}N), depending on the process parameters. The second layer results from the reactions produced by the N diffusion, such as precipitation of nitrides, α -Fe saturation, changes in the residual tensions and C redistributions.^{9,10}

During the boriding process, boron atoms can diffuse into ferrous alloys due to their relatively small size and very mobile nature. They can dissolve in iron interstitially, but can react with it to form FeB and Fe₂B intermetallic compounds. Depending on the potential of the medium and the chemical compositions of the base materials, a single or a duplex layer may be formed. Borided steel components display excellent performance in several tribological applications in the mechanical engineering and automotive industries.¹¹⁻¹⁴

Plasma nitriding and boriding are used in numerous applications in industries such as the manufacture of machine parts for plastics and food processing, packaging and tooling, as well as pumps and hydraulic machine parts, crankshafts, rolls and heavy gears, motor and car construction, cold- and hot-working dies and cutting tools. The wear behavior of plasma-nitrided and borided steels has been evaluated by a number of researchers.¹⁵⁻²⁰ However, there is no information about

the effect of sliding speed on the friction and wear behaviors of plasma-nitrided and borided W9Mo3Cr4V steel. The main objective of this study was to investigate the effect of sliding speed on the friction and wear behaviors of plasma-nitrided and borided W9Mo3Cr4V steel. Structural and tribological properties were investigated using light microscopy, XRD, SEM, EDS, microhardness tests and a ball-on-disc tribotester.

2 EXPERIMENTAL METHOD

2.1 Plasma Nitriding, Boriding and Characterization

The W9Mo3Cr4V steel contained mass fractions (w) 0.82 % C, 4.10 % Cr, 3.80 % Mo, 7.40 % W and 1.30 % V. The test samples were cut into \varnothing 25 mm \times 8 mm dimensions and ground up to 1000 G and polished using a diamond solution. The PN process was carried out in a dc plasma system at a temperature of 923 K for 6 h in a gas mixture of 80 % N₂-20 % H₂ under a constant pressure of 5 mbar. The boriding heat treatment was carried out by using a solid boriding method with commercial Ekabor-II powders. The samples that were to be borided were packed in the powder mixture and sealed in a stainless-steel container. The boriding heat treatment was performed in an electrical resistance furnace under atmospheric pressure at 1223 K for 6 h followed by air cooling.

The microstructures of the polished and etched cross-sections of the samples were observed under an Olympus BX-60 light microscope. The presence of nitrides and borides formed in the coating layer was detected by means of X-ray diffraction equipment (Shimadzu XRD 6000) using Cu K_{α} radiation. The diffusion zone on the nitrided sample and the thickness of borides were measured by means of a digital thickness measuring instrument attached to a light microscope (Olympus BX60). Micro-hardness measurements were performed from the surface to the interior along a line in order to see variations in the hardness of the nitride and boride layers, the transition zone and the matrix, respectively. The microhardness of the boride layers was measured at 8 different locations at the same distance from the surface by means of a Shimadzu HMV-2 Vickers indenter with a load of 50 g and the average value was taken as the hardness.

2.2 Friction and Wear

A ball-on-disc test device was used to perform the friction and wear tests of the borided samples. In the wear tests, WC-Co balls of 8 mm in diameter supplied by H. C. Starck Ceramics GmbH were used. Errors caused by the distortion of the surface were eliminated by using a separate abrasion element (WC-Co ball) for each test. The wear experiments were carried out in a ball-disc arrangement under dry friction conditions at room temperature with an applied load of 10 N and with

sliding speeds of (0.1, 0.3 and 0.5) m/s at a sliding distance of 1000 m. Before and after each wear test, each sample and abrasion element was cleaned with alcohol. After the test, the wear volumes of the samples were quantified by multiplying the cross-sectional areas of the wear by the width of the wear track obtained from the Taylor-Hobson Rugosimeter Surtronic 25 device. The wear rate was calculated using the following formula:

$$W_k = W_v / (M \cdot S) \text{ mm}^3 / (\text{N m}) \quad (1)$$

where W_k is the wear rate, W_v the worn volume, M is the applied load and S is the sliding distance.

Friction coefficients depending on the sliding distance were obtained through a friction coefficient program. Surface profiles of the wear tracks on the samples and surface roughness were measured by a Taylor-Hobson Rugosimeter Surtronic 25. The worn surfaces were investigated by scanning electron microscopy and energy-dispersive X-ray spectroscopy (EDS).

3 RESULTS AND DISCUSSION

3.1 Characterization of Plasma Nitriding and Boriding Coatings

The cross-sections of the optical micrographs of the nitrided and borided W9Mo3Cr4V steel are given in

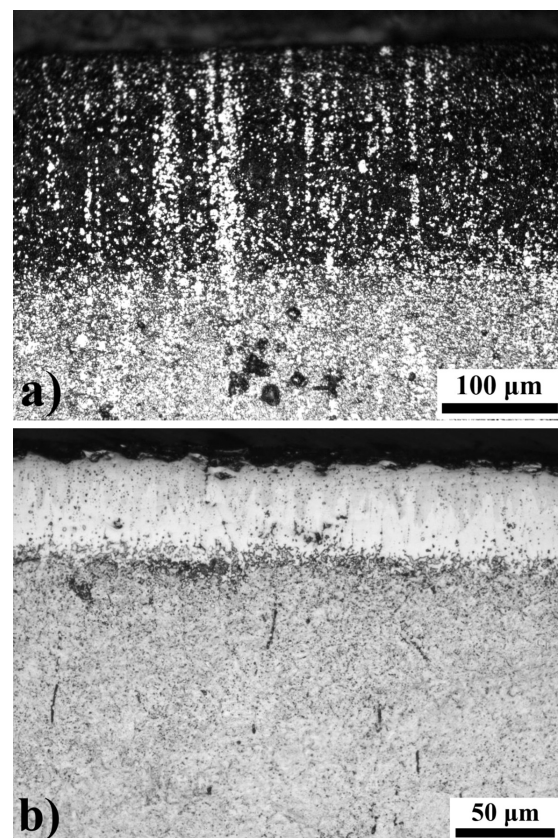


Figure 1: Cross-section of plasma-nitrided and borided W9Mo3Cr4V steel: a) PN, b) borided

Slika 1: Prerez v plazmi nitriranega in boriranega jekla W9Mo3Cr4V: a) PN, b) borirano

Figure 1. A typical light photograph of the cross-sectional microstructure of the plasma nitrided W9Mo3Cr4V sample is given in **Figure 1a**. This photograph shows the formation of a diffusion zone on the sample. However, the formation of a continuous compound (white) layer on top of the nitrided layer of the sample is not visible in **Figure 1a**. Following etching with a 3 % nital reagent, the nitrided (diffusion) layer appears as a dark zone on the sample. Around 250 μm depth of diffusion was obtained from the plasma-nitrided sample. As can be seen in **Figure 1b**, the boride layer was formed on the W9Mo3Cr4V steel borided with a solid boriding method. Depending on the process time, the temperature and the chemical composition of substrates, the thickness of the boride layer was found to be 55 μm .

The X-ray diffraction patterns of the nitrided and borided W9Mo3Cr4V steel are given in **Figure 2**. The XRD results showed that the nitrided W9Mo3Cr4 steel contained FeN, Fe₂N, Fe₃N, Fe₄N and M₆C phases (**Figure 2a**). The XRD results showed that the boride layers formed on the W9Mo3Cr4V steel contained FeB, Fe₂B, CrB, MoB and WB phases in **Figure 2b**. The microhardness and the wear resistance of the nitride and boride layers formed on the steel surface are to a large extent known with the help of these phases.²¹⁻²⁴

Microhardness measurements were carried out on the cross-sections from the surface to the interior along a

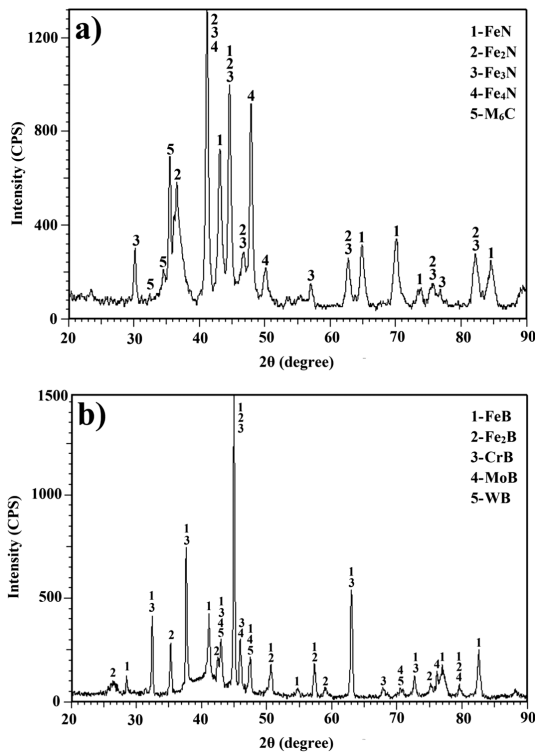


Figure 2: X-ray diffraction patterns of plasma-nitrided and borided W9Mo3Cr4V steel: a) PN, b) borided

Slika 2: Rentgenska difrakcija v plazmi nitriranega in boriranega jekla W9Mo3Cr4V: a) PN, b) borirano

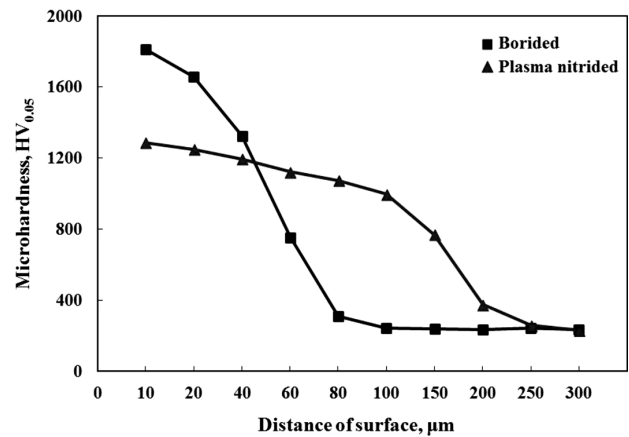


Figure 3: The variation of hardness depth in the plasma-nitrided and borided W9Mo3Cr4V steel

Slika 3: Spreminjanje trdote po globini v plazmi nitriranega in boriranega jekla W9Mo3Cr4V

line, as can be seen in **Figure 3**. The hardness of the nitrided W9Mo3Cr4V steel varied between 995 HV_{0.05} and 1286 HV_{0.05}. The hardness of the boride layer on the W9Mo3Cr4V steel varied between 1378 HV_{0.05} and 1814 HV_{0.05}. On the other hand, the Vickers hardness values were 230 HV_{0.05} for the untreated W9Mo3Cr4V steel. When the hardnesses of the nitride and boride layers are compared with the matrix, the nitride layer's hardness is approximately five times greater and the boride layer's hardness is approximately eight times greater than that of the matrix.

3.2 Friction and Wear Behavior

The wear mechanism is defined as the physical and chemical phenomena that take place during wear. Abrasive wear refers to the mechanism where two bodies rubbing against each other break off pieces from each other. The hardness, shape and size or roughness of the

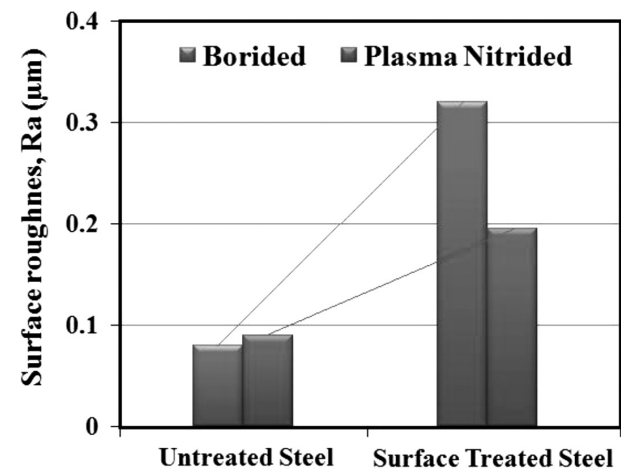


Figure 4: Surface roughness values of the plasma-nitrided and borided W9Mo3Cr4V steel

Slika 4: Hrapavost površine v plazmi nitriranega in boriranega jekla W9Mo3Cr4V

abrasive material, the angle of contact, the normal load applied, the sliding speed and the fracture toughness of the material are all among the important factors in wear mechanisms.²⁵ **Figure 4** shows the surface roughness values of the nitrided and borided and unborided W9Mo3Cr4V steel. For the nitrided and borided W9Mo3Cr4V steel, it was observed that the surface roughness values increased, as can be seen in **Figure 4**. It was observed that the surface roughness of the nitrided W9Mo3Cr4V steel was higher than that of the borided W9Mo3Cr4V. These results indicate that both nitriding and boriding treatments affect the surface roughness of the specimens. While the surface roughness value of the borided sample is 0.84 μm , it increased to 0.33 μm as a result of the boriding process. While the roughness level of the surface of the non-nitrided sample is 0.93 μm at the end of the boriding process, it has increased to 0.198 μm .

Figure 5 shows the friction coefficients of the nitrided and borided W9Mo3Cr4V steels at different sliding speeds. The friction coefficients of the nitrided and borided W9Mo3Cr4V steels varied from 0.61 to 0.42 and from 0.52 to 0.33, respectively. The friction coefficients of both borided and nitrided W9Mo3Cr4V steels reduced with the increase of the sliding speed up to 0.5 m/s. The reason behind this may be the oxides on the surface of the sample. During the wear test, oxides formed on the steel surface, allowing for low friction coefficients. The wear tracks on the samples may have been oxidized due to the frictional heat.

As the sliding speed increased, the steady-state friction coefficient values slightly decreased. This may be explained by a decrease in the shear strength at the sliding interface. Decreasing the shear strength may occur in two ways. Firstly, the hardness and hence the shear strength of the coatings decrease with increasing temperature. It is reported that the hardness of the boride coatings decreases by a factor of more than two with

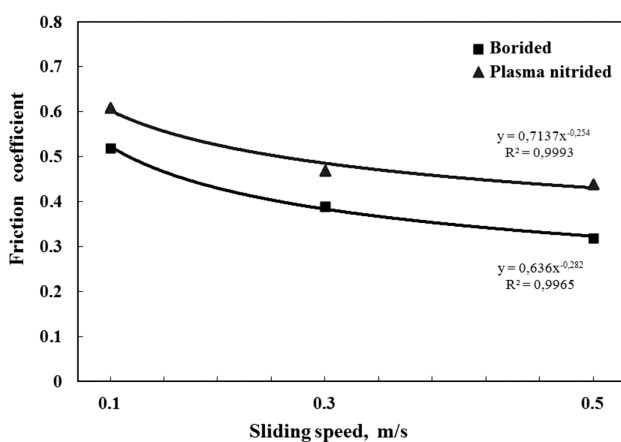


Figure 5: The effect of the sliding speed on the friction coefficient of the plasma-nitrided and borided W9Mo3Cr4V steel

Slika 5: Vpliv hitrosti drsenja na koeficient trenja jekla W9Mo3Cr4V, nitriranega v plazmi ali boriranega

increasing temperature up to 1000 °C.¹² Secondly, oxidation products (iron, boron, molybdenum, wolfram and chromium oxides) can reduce the friction. In fact, many of the oxides have high friction.

Figure 6 shows the effect of sliding speed on the wear rate of plasma-nitrided and borided W9Mo3Cr4V steel. It was observed that the borided W9Mo3Cr4V steel had a lower wear rate than that of the plasma-nitrided W9Mo3Cr4V steel. For the microhardness of the FeB, CrB and WB phases, the borided steel showed more resistance to wear due to the nitrided steel. The wear test results indicated that the wear resistance of the nitrided and borided steels was higher than that of the untreated steel. It is well known that the hardness of the nitride and boride layers plays an important role in the improvement of the wear resistance.

As shown in **Figures 3** and **6**, the relationship between the microhardness and the wear resistance of the nitrided and borided samples also confirms that the wear resistance was improved by the increase in the hardness. This is in agreement with reports of previous studies.²⁶⁻³³ In addition, reductions in wear rates were observed with an increasing rate of sliding speed. The oxides formed on the samples borided at high sliding speeds may have caused a reduction in the friction coefficient and an increase in the wear rate because oxides on the sample surface act as a solid lubricant. Molinari et al.³⁴ investigated the tribological properties of a plasma-nitrided Ti-6Al4V alloy at different loads and sliding speeds. It was observed that the increase in the sliding speed at low loads did not significantly affect the wear volume, while the wear volume decreased with an increase in the sliding speed at high loads. Straffellini et al.³⁵ investigated the impact of sliding speed and contact pressure on the oxidative wear of austempered ductile iron and reported that the friction coefficient and wear rate decreased with an increasing rate of sliding speed.

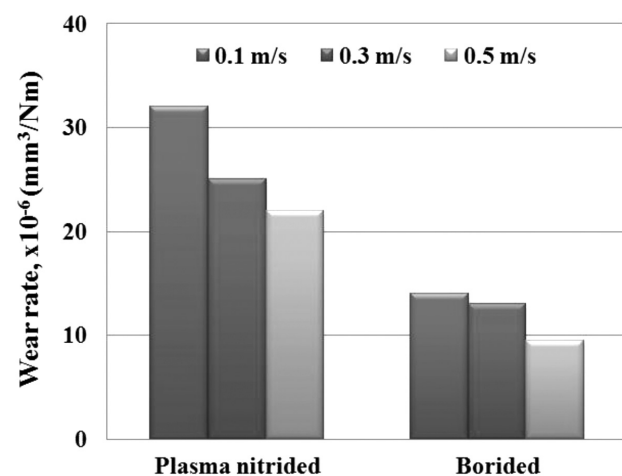


Figure 6: The effect of the sliding speed on the wear rate of the plasma-nitrided and borided W9Mo3Cr4V steel

Slika 6: Vpliv hitrosti drsenja na stopnjo obrabe v plazmi nitriranega ali boriranega jekla W9Mo3Cr4V

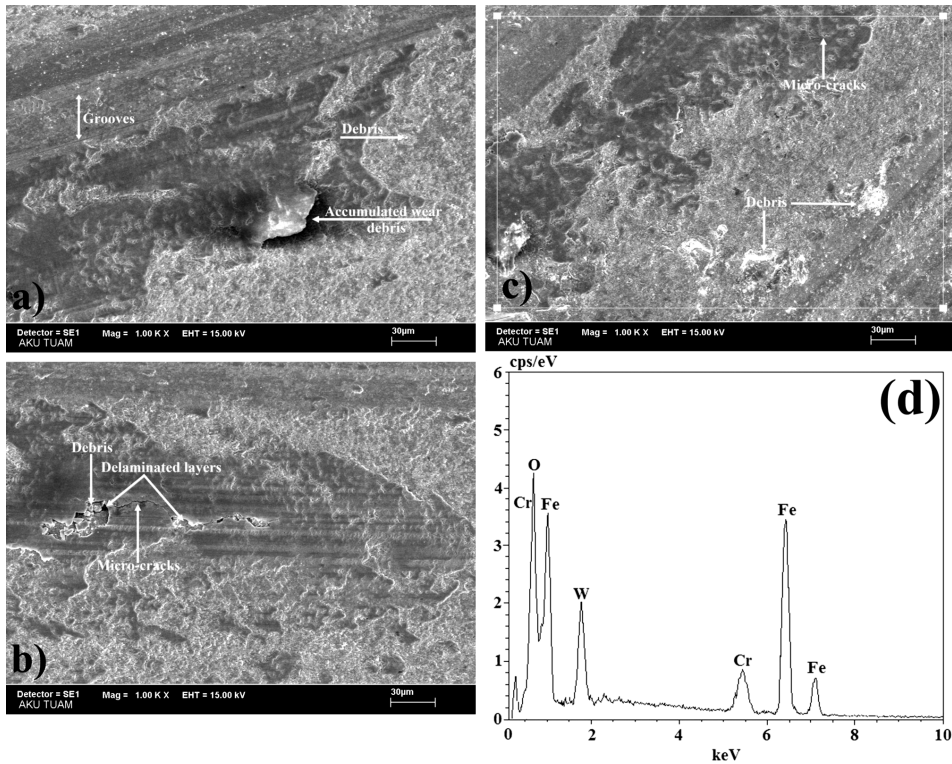


Figure 7: Sliding-speed-dependent SEM micrographs of the wear surfaces of the nitrided W9Mo3Cr4V steel at 923 K for 6 h: a) 0.1 m/s, b) 0.3 m/s, c) 0.5 m/s, d) EDS analysis

Slika 7: SEM-posnetki obrabljene površine v odvisnosti od hitrosti drsenja za 6 h nitrirano jeklo W9Mo3Cr4V pri 923 K: a) 0,1 m/s, b) 0,3 m/s, c) 0,5 m/s, d) EDS-analiza

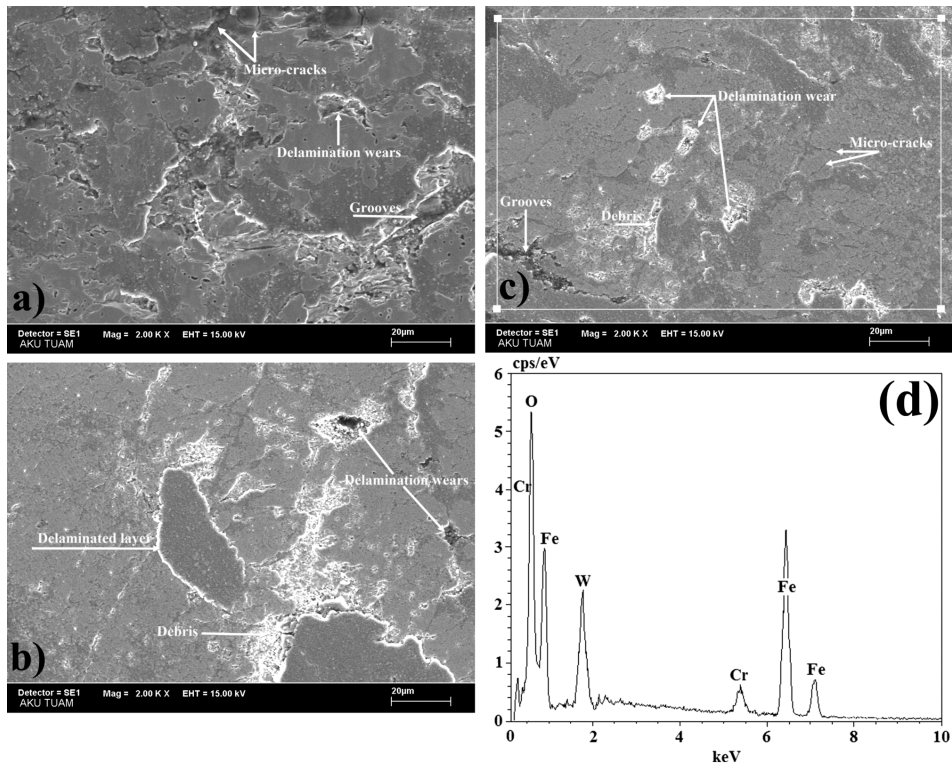


Figure 8: Sliding-speed-dependent SEM micrographs of the wear surfaces of the borided W9Mo3Cr4V steel at 1223 K for 6 h: a) 0.1 m/s, b) 0.3 m/s, c) 0.5 m/s, d) EDS analysis

Slika 8: SEM-posnetki obrabljene površine v odvisnosti od hitrosti drsenja za 6 h borirano jeklo W9Mo3Cr4V pri 1223 K: a) 0,1 m/s, b) 0,3 m/s, c) 0,5 m/s, d) EDS-analiza

The SEM micrographs and X-ray energy-dispersive spectroscopy EDS of the worn surfaces of the nitrided and borided samples are illustrated in **Figures 7** and **8**. **Figure 7** shows the SEM micrographs of the wear surfaces of nitrided W9Mo3Cr4V steel worn at different sliding speeds. The wear region of the nitrided samples, debris, surface grooves and cracks on the layer are shown in **Figures 7a to 7c**. It was observed that delamination wears, which form as a result of the progress of microcracks, occurred on the wear tracks of the nitrided samples. The wear intensity on the surfaces of the nitrided samples was observed to decrease with an increase in the sliding speed (**Figures 7a to 7c**). Oxidation products (iron, boron, wolfram and chromium oxides) formed as a result of the wear test in **Figure 7d**.

Figure 8 shows the SEM micrographs of the wear surfaces of borided W9Mo3Cr4V steel worn at different sliding speeds. There were microcracks, abrasive particles and small holes on the worn surface of the boride coatings (**Figures 8a to 8c**). In the wear region of the borided W9Mo3Cr4V steel, there were cavities probably formed as a result of layer fatigue (**Figure 8b**) and cracks concluded in the delaminating wear (**Figure 8c**). It was observed that delamination wear, which forms as a result of the progress of microcracks, occurred on the wear tracks of the borided samples. Oxide layers formed as a result of the wear test in **Figure 8d**. The spallation of the oxide layers in the sliding direction and their orientation extending along the wear track were identified (**Figures 7d and 8d**).

4 CONCLUSIONS

The following conclusions may be drawn from the present study.

- The multiphase nitride coating that thermo-chemically occurred on the W9Mo3Cr4V steel consisted of FeN, Fe₂N, Fe₃N, Fe₄N and M₆C phases, while the boride coating consisted of FeB, Fe₂B, CrB, MoB and WB phases.
- The surface hardness of the nitrided W9Mo3Cr4V steel was in the range of 995–1286 HV_{0.05}, the surface hardness of the borided W9Mo3Cr4V steel was in the range 1378–1814 HV_{0.05} and the untreated W9Mo3Cr4V steel substrate was 230 HV_{0.05}.
- The surface roughness values of both the plasma-nitrided and borided W9Mo3Cr4V steel increased compared to the untreated W9Mo3Cr4V steel.
- The friction coefficients of the plasma-nitrided and borided W9Mo3Cr4V steel varied from 0.42 to 0.61, and from 0.33 to 0.52, respectively.
- Reductions were observed in the wear rates of both the nitrided and borided W9Mo3Cr4V steel.
- As a result of an increase in the sliding speed, it was observed that the friction coefficient decreased while

the wear resistance of the plasma nitrided and borided W9Mo3Cr4V steel increased.

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