

## TRIBOLOGICAL BEHAVIOR AND CHARACTERIZATION OF BORIDED COLD-WORK TOOL STEEL

### TRIBOLOŠKO VEDENJE IN KARAKTERIZACIJA BORIRANEGA ORODNEGA JEKLA ZA DELO V HLADNEM

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In the present study, tribological and characterization properties of the borides formed on cold-work tool steel were investigated. Boriding was performed in a solid medium consisting of Ekabor-II powders at 850 °C and 950 °C for 2 h and 6 h. The boride layer was characterized with light microscopy, X-ray diffraction technique and a micro-Vickers hardness tester. An X-ray diffraction analysis of the boride layers on the surface of the steel revealed the existence of FeB, Fe<sub>2</sub>B, CrB, Cr<sub>2</sub>B and MoB compounds. Depending on the chemical compositions of the substrates and the boriding time, the boride-layer thickness on the surface of the steel ranged from 13.14 μm to 120.82 μm. The hardness of the boride compounds formed on the surface of the steel ranged from 1806 HV<sub>0.05</sub> to 2342 HV<sub>0.05</sub>, whereas the Vickers-hardness value of the untreated steel was 428 HV<sub>0.05</sub>. The wear tests were carried out in a ball-disc arrangement under a dry friction condition at room temperature with an applied load of 10 N and with a sliding speed of 0.25 m/s at a sliding distance of 1000 m. The wear surfaces of the steel were analyzed using SEM microscopy and X-ray energy-dispersive spectroscopy (EDS). It was observed that the wear rate of the unborided and borided cold-work tool steels ranged from 11.28 mm<sup>3</sup>/(N m) to 116.54 mm<sup>3</sup>/(N m).

Keywords: cold-work tool steel, boriding, microhardness, wear rate, friction coefficient

V tej študiji so bile preiskovane tribološke in druge značilne lastnosti boridov, nastalih na orodnem jeklu za delo v hladnem. Boriranje je bilo izvršeno v trdnem mediju, ki ga je sestavljala prašina Ekabor-II pri 850 °C in 950 °C v 2 h in 6 h. Boriran sloj je bil preiskan s svetlobno mikroskopijo, rentgensko difrakcijo in izmerjena je bila Vickersova mikrotrdota. Rentgenska difrakcija boriranega sloja na površini jekla je potrdila prisotnost spojin FeB, Fe<sub>2</sub>B, CrB, Cr<sub>2</sub>B in MoB. Odvisno od kemijske sestave podlage in časa boriranja je bila debelina boriranega sloja na površini jekla med 13,14 μm in 120,82 μm. Trdota boridov, nastalih na površini jekla, je bila v razponu od 1806 HV<sub>0.05</sub> do 2342 HV<sub>0.05</sub>, medtem ko je bila Vickersova trdota neobdelanega jekla 428 HV<sub>0.05</sub>. Preizkusi obrabe so bili izvršeni na napravi krogla-disk pri suhem trenju pri sobni temperaturi z uporabljenimi obtežbo 10 N, hitrostjo drsenja 0,25 m/s in pri razdalji drsenja 1000 m. Površina z obrabo je bila analizirana s SEM-mikroskopijo in energijsko disperzijsko spektroskopijo rentgenskih žarkov (EDS). Ugotovljeno je bilo, da je hitrost obrabe neboriranega in boriranega orodnega jekla za delo v hladnem v razponu od 11,28 mm<sup>3</sup>/(N m) do 116,54 mm<sup>3</sup>/(N m).

Ključne besede: orodno jeklo za delo v hladnem, boriranje, mikrotrdota, hitrost obrabe, koeficient trenja

## 1 INTRODUCTION

Industrial boriding processes can be applied to a wide range of steel alloys including carbon steel, low and high-alloy steel, tool steel and stainless steel. Cold-work tool steels have a wide range of applications. Therefore, there has been an extensive research on the development of surface-treatment processes to improve the wear resistance, corrosion and oxidation resistance of the cold-work tool steels for high-temperature and high-pressure applications in recent years.<sup>1-7</sup> Boriding is a thermochemical surface treatment, in which boron atoms diffuse into the surface of a workpiece to form borides with the base material.<sup>8,9</sup> The main advantages of this technique are a high resistance to abrasion wear and a high oxidation resistance when compared with the other conventional surface treatments. The thermal diffusion treatments of boron compounds used to form iron borides typically require the process temperatures of 700 °C and 1000 °C. The process can be carried out in a solid, liquid, gaseous or plasma medium.<sup>10-14</sup>

In recent years, the boriding treatment has been used for a wide range of applications in industries such as the

manufacture of machine parts for plastics, food processing, packaging and tooling, as well as pumps and hydraulic machine parts, crankshafts, rolls and heavy gears, motor and car constructions, cold- and hot-working dies and cutting tools. The wear behavior of borided steels has been evaluated by a number of investigators.<sup>15-20</sup> However, there is no information about the friction and wear behavior of the borided cold-work tool steel. The main objective of this study was to investigate the friction and wear behavior of the borided cold-work tool 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 Boriding and characterization

The high-alloy cold-work tool steel essentially contained mass fractions 0.90 % C, 0.50 % Mn, 7.80 % Cr, 2.50 % Mo and 0.50 % V. The test specimens were cut into the cylinders with the dimensions of  $\varnothing 25$  mm  $\times$  10 mm, ground up to 1200 G and polished using a dia-

mond solution. The boriding heat treatment was carried out in a solid medium containing an Ekabor-II powder mixture placed in an electrical-resistance furnace operated at the temperatures of 850 °C and 950 °C for 2 h and 6 h under atmospheric pressure. Following the completion of the boriding process, the test specimens were removed from the sealed stainless-steel container and allowed to cool down in still air. The microstructures of the polished and etched cross-sections of the specimens were observed with a Nikon MA100 light microscope. The presence of the borides formed in the coating layer was confirmed by means of X-ray diffraction equipment (Shimadzu XRD 6000) using Cu K $\alpha$  radiation. The hardness measurements of the boride layer for each steel type and the unborided steel substrate were made on the cross-sections using a Shimadzu HMV-2 Vickers indenter with a load 50 g.

## 2.2 Friction and wear

To perform the friction and wear tests of the borided samples, a ball-on-disc test device was used. In the wear tests, WC-Co balls of 8 mm in diameter supplied by H. C. Starck Ceramics GmbH were used. The errors caused by a distortion of the surface were eliminated using a separate abrasion element (WC-Co ball) for each test. The wear experiments were carried out in a ball-disc arrangement under a dry friction condition at room temperature with an applied load of 10 N and with the sliding speed of 0.25 m/s at a sliding distance of 1000 m. Before and after each wear test, each sample and the abrasion element were cleaned with alcohol. After the tests, 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 a Taylor-Hobson Rugosimeter Surtronic 25 device. The wear rate was calculated with the following formula:

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

where  $W_k$  is the wear rate,  $W_v$  is the wear volume,  $M$  is the applied load and  $S$  is the sliding distance. The friction coefficients depending on the sliding distance were obtained through a friction-coefficient program. The surface profiles of the wear tracks on the samples and the surface roughness were measured with the Taylor-Hobson Rugosimeter Surtronic 25. The worn surfaces were investigated with scanning electron microscopy (SEM) and energy-dispersive X-ray spectroscopy (EDS).

## 3 RESULTS AND DISCUSSION

### 3.1 Characterization of boride coatings

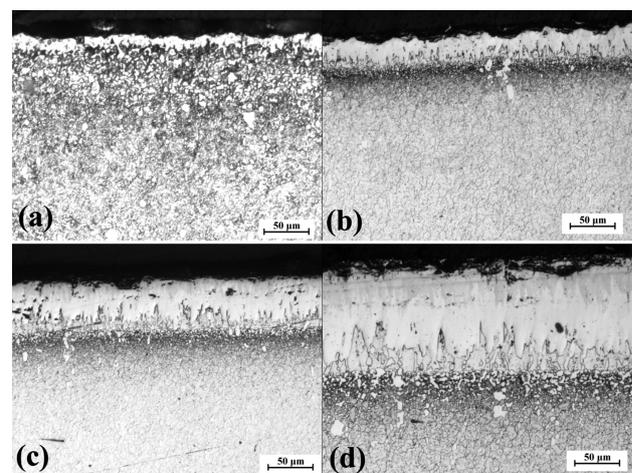
Light micrographs of the cross-sections of the cold-work tool steel borided at the temperatures of 850 °C and 950 °C for 2 h and 6 h are shown in **Figure 1**. As can be seen, the borides formed on the cold-work tool-steel substrate have a smooth morphology due to the

high-alloy content. It was found that the coating/matrix interface and the matrix could be significantly distinguished and that the boride layer had a columnar structure. Depending on the chemical compositions of the substrates and the boriding time, the boride-layer thickness on the surface of the steel ranged from 13.14  $\mu\text{m}$  to 120.82  $\mu\text{m}$ .

In this study, the presence of borides was identified using an XRD analysis (**Figure 2**). The XRD patterns show that the boride layer consists of the borides such as AB and A<sub>2</sub>B (A = metal: Fe, Cr). The XRD results showed that the boride layers formed on the steel contained the FeB, Fe<sub>2</sub>B, CrB, Cr<sub>2</sub>B and MoB compounds (**Figure 2**). The microhardness measurements were carried out along a line between the surface and the interior in order to see the variations in the boride-layer hardness, the transition zone and the matrix, respectively. The microhardness of the boride layers was measured at 10 different locations at the same distance from the surface, and the average value was taken as the hardness result. The microhardness measurements were carried out on the cross-sections, along a line between the surface and the interior (**Figure 3**). The hardness of the boride compounds formed on the surface of the steel ranged from 1806 HV<sub>0.05</sub> to 2342 HV<sub>0.05</sub>, whereas the Vickers-hardness value of the untreated steel was 428 HV<sub>0.05</sub>. When the hardness of the boride layer is compared with the matrix, the boride-layer hardness is approximately four times greater than that of the matrix.

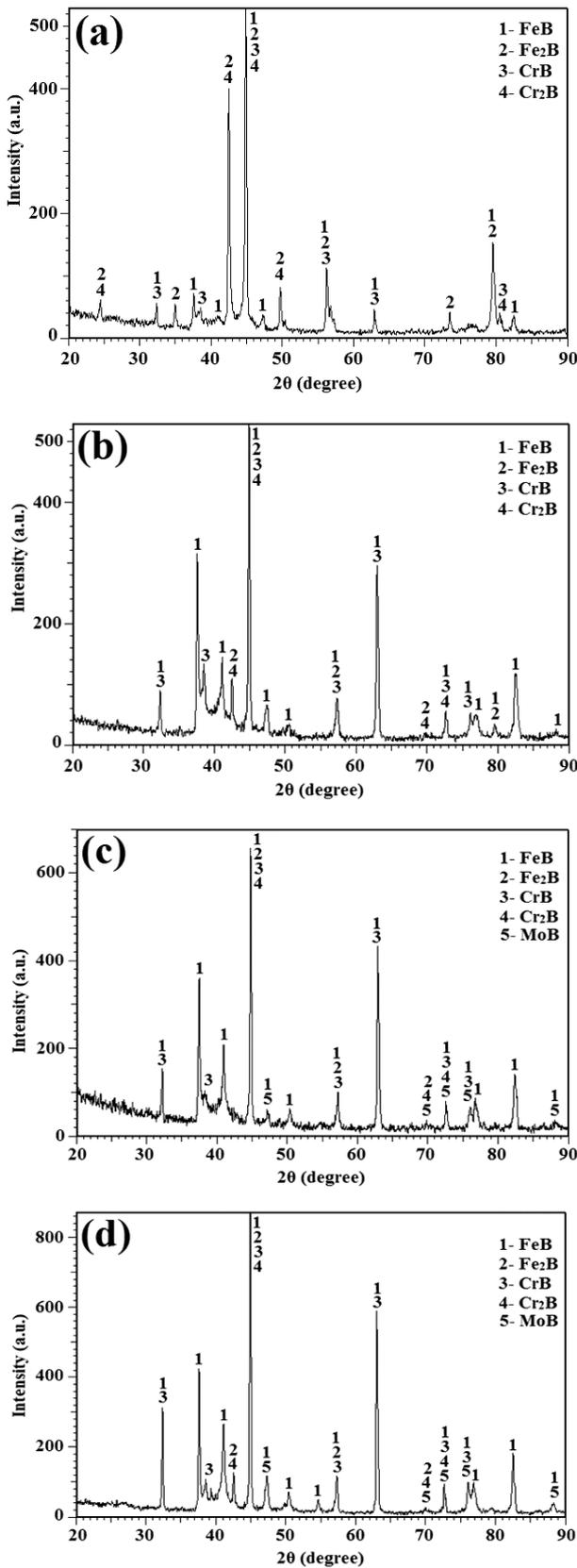
### 3.2 Friction and wear behavior

**Table 1** shows the surface-roughness values of the borided and unborided cold-work tool steels. The surface-roughness values of the unborided and borided cold-work tool steels varied from 0.12 to 0.54, as can be seen in **Table 1**. It was observed for the cold-work tool

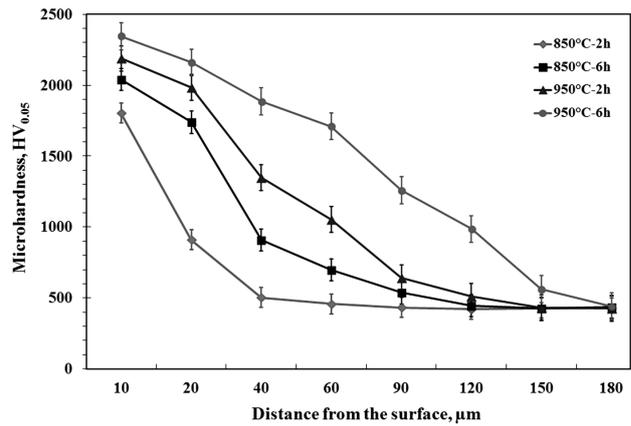


**Figure 1:** Cross-sections of borided cold-work tool steel: a) 850 °C, 2 h, b) 850 °C, 6 h, c) 950 °C, 2 h, d) 950 °C, 6 h

**Slika 1:** Prerez boriranega orodnega jekla za delo v hladnem: a) 850 °C, 2 h, b) 850 °C, 6 h, c) 950 °C, 2 h, d) 950 °C, 6 h



**Figure 2:** X-ray diffraction patterns of borided cold-work tool steel: a) 850 °C, 2 h, b) 850 °C, 6 h, c) 950 °C, 2 h, d) 950 °C, 6 h  
**Slika 2:** Rentgenska difrakcija boriranega orodnega jekla za delo v hladnem: a) 850 °C, 2 h, b) 850 °C, 6 h, c) 950 °C, 2 h, d) 950 °C, 6 h



**Figure 3:** Variation of the hardness depth for borided steel  
**Slika 3:** Spreminjanje trdote po globini boriranega jekla

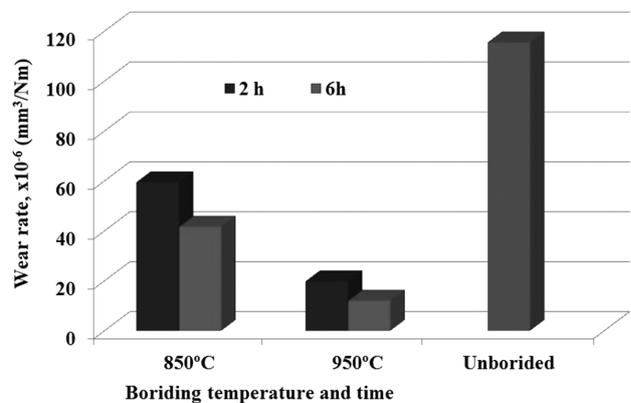
**Table 1:** Surface roughness values for unborided and borided steels  
**Tabela 1:** Vrednosti za hrapavost površine neboriranega in boriranega jekla

Unborided	Borided			
	850 °C, 2 h	850 °C, 6 h	950 °C, 2 h	950 °C, 6 h
0.12	0.32	0.37	0.43	0.54

**Table 2:** Friction coefficients for unborided and borided steels  
**Tabela 2:** Koeficient trenja za neborirano in borirano jeklo

Unborided	Borided			
	850 °C, 2 h	850 °C, 6 h	950 °C, 2 h	950 °C, 6 h
0.65	0.38	0.41	0.48	0.56

steel that the surface-roughness values increased with the boriding treatment and time. Gunes<sup>18</sup> studied plasma-paste-borided AISI 8620 steel and reported that the surface-roughness values increased with an increase in the boriding temperature. On the other hand, the friction coefficients of the unborided and borided cold-work tool steels varied from 0.38 to 0.65, as can be seen in **Table 2**. With the boriding treatment, a slight reduction was observed in the friction coefficients of the borided steels.



**Figure 4:** Wear rate of unborided and borided cold-work tool steels  
**Slika 4:** Obraba neboriranega in boriranega orodnega jekla za delo v hladnem

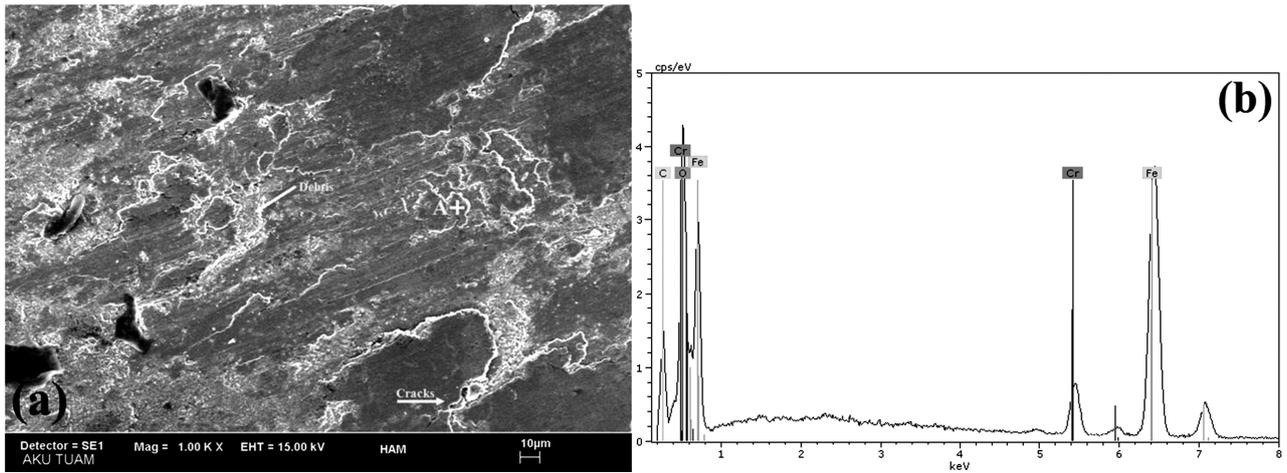


Figure 5: a) SEM micrograph and b) EDS analysis of the wear surfaces of unborided steel  
 Slika 5: a) SEM-posnetek in b) EDS-analiza obrabljenih površin na neboriranem jeklu

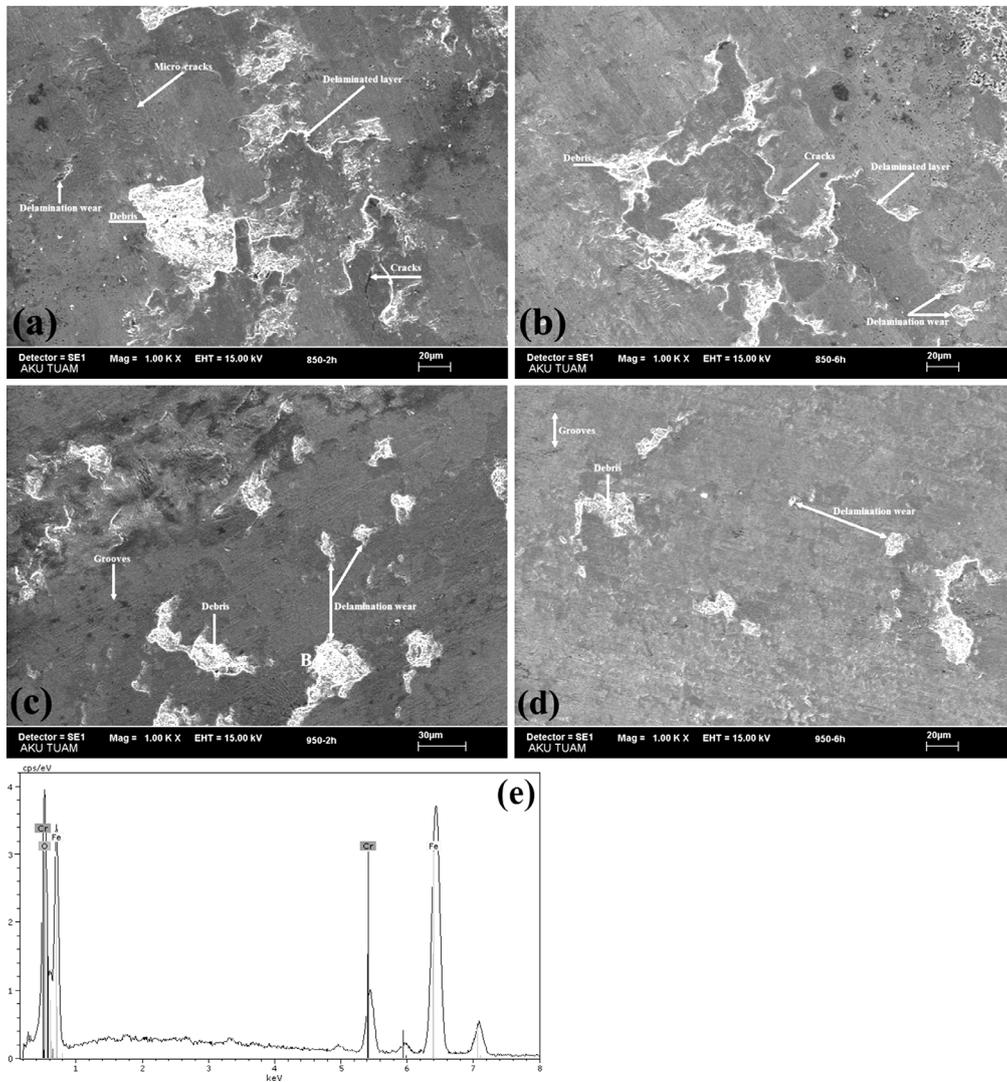


Figure 6: SEM micrographs and EDS analysis of the wear surfaces of borided steel: a) 850 °C, 2 h, b) 850 °C, 6 h, c) 950 °C, 2 h, d) 950 °C, 6 h, e) EDS  
 Slika 6: SEM-posnetki in EDS-analiza obrabljene površine na boriranem jeklu: a) 850 °C, 2 h, b) 850 °C, 6 h, c) 950 °C, 2 h, d) 950 °C, 6 h, e) EDS

**Figure 4** shows the wear rate of the unborided and borided cold-work tool steels. The reductions in the wear rates of the borided steels were observed with respect to the unborided steels. Due to the toughness of the FeB, Fe<sub>2</sub>B, CrB, Cr<sub>2</sub>B and MoB phases, these steels showed a larger resistance to wear. The lowest wear rate was obtained for the steel borided at 950 °C for 6 h, while the highest wear rate was obtained for the unborided steel. The wear test results indicated that the wear resistance of borided steels increased considerably with the boriding treatment. It is well known that the hardness of the boride layer plays an important role in the improvement of the wear resistance. As shown in **Figures 3** and **4**, the relationship between the surface microhardness and the wear resistance of borided samples also confirms that the wear resistance was improved with an increased hardness. This is in agreement with the reports of the previous studies.<sup>20–23</sup> When the wear rate of the borided steel is compared with the unborided steel, the wear rate of the borided steel (950 °C, 6 h) is approximately nine times lower than that of the unborided steel.

SEM micrographs of the worn surfaces of the unborided and borided cold-work tool steels are in **Figures 5** and **6**. **Figure 5a** shows a SEM micrograph of the wear surface of the unborided steel. In the wear region of the unborided steel, deeper and wider wear scars, debris, surface grooves and cracks can be observed on its surface. **Figure 5b** shows an EDS analysis obtained from **Figure 5a** (point A). Fe-based oxide layers formed as a result of the wear test.

**Figure 6** shows SEM micrographs of the wear surfaces of the borided cold-work tool steel. In **Figure 6a**, the worn surface of the borided steel is rougher, and coarser wear debris is present. There are microcracks, a delamination layer and wear, grooves, cracks and abrasive particles on the worn surfaces of the boride coatings (**Figures 6a** to **6d**). In the wear region of the borided cold-work tool steel there are cavities, probably formed as a result of the layer fatigue (**Figure 6**) and cracks formed due to the delamination wear. **Figure 6c** shows an EDS analysis obtained from **Figure 6c** (point B). The Fe-based oxide layers formed as a result of the wear test. The spallation of the oxide layers in the sliding direction and their orientation along the wear track were identified. When the SEM image of the worn surface of the unborided sample in **Figure 5a** is examined, it can be seen that the wear marks are larger and deeper.

#### 4 CONCLUSIONS

In this study, the wear behavior and some of the mechanical properties of the borides on the surface of the borided cold-work tool steel were investigated. Some of the conclusions can be drawn as follows:

- Boride types formed on the surface of the cold-work tool steel have a smooth morphology.

- The boride-layer thickness on the surface of the cold-work tool steel was between 13.14–120.82 μm, depending on the chemical compositions of the substrates.
- The multiphase boride coatings that were thermochemically grown on the cold-work tool steel were constituted of the FeB, Fe<sub>2</sub>B, CrB, Cr<sub>2</sub>B and MoB phases.
- The surface hardness for the borided steel was in the range of 1806–2342 HV<sub>0.05</sub>, while for the untreated steel substrate, it was 428 HV<sub>0.05</sub>.
- The lowest wear rate was obtained for the steel borided at 950 °C for 6 h, while the highest wear rate was obtained for the unborided steel.
- The wear rate of the borided steel was found to be approximately nine times lower than the wear rate of the unborided steel.

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