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Araştırma / Research

INVESTIGATION OF WEAR BEHAVIOR OF BORONIZED AISI 316 STAINLESS STEEL

Polat TOPUZ[1] (ORCID: 0000-0001-9715-6682)[*]
Tuna AYDOĞMUŞ[2] (ORCID: 0000-0002-8736-2949)

[1] İstanbul Gedik Üniversitesi Gedik MYO
[2] Sülüntepe Mah. Yunus Emre Cad. No: 1/1 Şeyhli Pendik/ İSTANBUL

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ABSTRACT

In this study, the wear behavior of boronized AISI 316 stainless steel was investigated. The chemical analysis of the sample to be used in the experiment was carried out with optical emission spectrometry before starting the boronizing experiment. Boronizing experiment using pack-boronizing method were carried out at 950°C for 3 hours. As the boron source, a powder mixture having the commercial name Ekabor 2 consisting of SiC, B_4C and KBF_4, was used. As a result of the boronizing treatment, the boride layer formed on the surface of AISI 316, morphologically by Scanning Electron Microscope (SEM-BEI), thickness by optical microscope integrated visual analyze system, hardness by Vickers indenter and chemical analysis by XRD (X-ray diffraction) device, were investigated. Then, friction tests were carried out to determine the wear resistance of the boride layer. As a result, it is determined that the boron layer formed on the surface of boronized AISI 316 stainless steel, consists of a double phase iron-boride layer. This layer is a columnar morphology and homogeneous thickness. Also it has been determined to have ultra-hard and high wear resistance.

Keywords: AISI 316 stainless steel, pack-boronizing, Ekabor 2, iron-boride layer, wear resistance

BORLANMIŞ AISI 316 PASLANMAZ ÇELİĞİN AŞINMA DAVRANIŞININ İNCELENMESİ

ÖZ

Bu çalışmada borlanmış AISI 316 paslanmaz çeliğin aşınma davranışı incelenmiştir. Borlama işlemi öncesi, optik emisyon spektrometresi yardımıyla, deney numunesinin kimyasal analizi gerçekleştirilmiştir. Borlama deneyi, 950°C'de 3 saat süre ile kutu borlama yöntemi kullanılarak gerçekleştirilmiştir. Borlayıcı olarak, SiC, B_4C and KBF_4' den oluşmuş, ticari adı Ekabor 2 olan toz karışımı kullanılmıştır. Borlama işlemi sonucunda, AISI 316 paslanmaz çeliğin yüzeyinde oluşan borür tabakasının morfolojisi, tarama elektron mikroskobu (SEM-geri saçılan elektronlar), kalınlığı optik mikroskoba entegreli görüntü analiz sistemi, sertliği Vickers batıcı uç ile ve kimyasal analizi XRD (X-ışını difraksiyonu) cihazı ile gerçekleştirilmiştir. Daha sonra, borür tabakasının aşınma direncini belirlemek için, sürtünme testi gerçekleştirilmiştir. Sonuç olarak, borlanmış AISI 316 paslanmaz çeliğin yüzeyinde oluşan demir-borür tabakasının çift fazlı olduğu belirlenmiştir. Bu tabaka, kolonsal yapılı ve homojen kalınlıktadır. Ayrıca, çok sert ve yüksek aşınma direncine sahip olduğu belirlenmiştir.

Anahtar kelimeler: AISI 316 paslanmaz çelik, kutu borlama, Ekabor 2, demir-borür tabakası, aşınma direnci

[*]Corresponding author / Sorumlu yazar. Tel.: + 90 216 595 21 85; e-mail / e-posta: polat.topuz@gedik.edu.tr

1. INTRODUCTION

Boron is the transition element with an atomic weight of 10.81 and an atomic number 5 in the IIIA group. The boron atom has a radius of 0.098 nm and a melting temperature of 2092 °C. Boron element density is 2.33 ± 0.02 g.cm^{-3}, the number of valans electrons 3 and the ion radius is 0.023 nm [1,2]. Boron is not free in nature and is found as borates [3]. There are about 230 kinds of boron minerals in nature. The different properties of the compounds made with various metal or nonmetal elements allow the use of many boron compounds in the industry [4]. Boron compounds have little electrical conductivity, but pure boron is conductive. The crystalline boron is similar to the diamond in terms of its appearance and optical properties, even almost as hard as diamonds [5].

Boronizing is a thermochemical surface hardening process that establishes a diffusion bond between boron atoms and the atoms of material and forms a single or double phase boride layer on the material surface. Today, many metals or alloys are possible to boronized [6]. The borides formed by boronization, improve the tribological characteristics of the substrate steel [7]. Boronizing processes can be applied by many methods. They can be classified as solid, liquid, gas, paste, plasma and fluidized bed furnace boronizing, and can usually be applied to materials with a well-cleaned surface in the temperature range of 800-1000°C for 1 to12 hours [8]. The formation of boride layer is diffusion controlled. As the temperature increases, the thickness of the boride layer formed on boronized iron surfaces also increases. The phase formed as a result of boronization of iron-based materials, only FeB, is the permanent tension, prone to tensile, if the phase, Fe_2B, is prone to compress. Because of this situation, the phases apply the tensile-compressive force in the double-phase boride layers [9,10]. The properties of the layers obtained by the boronizing of iron based materials are examined with hardness, wear resistance and corrosion resistance. The main advantage of the boronizing process is the high hardness of the material. The hardness depends on the type of material and FeB or Fe_2B phases on the surface. FeB phase is harder and brighter than Fe_2B phase [11]. The atoms of the boronizing compound used in the boronizing process are settled between the atoms of the iron-based material by diffusion. The hardness of the boride layer changes depending on the composition of the boronized material and the structure of the boride layer [8].

In order to increase the wear resistance, the material must be harder than the abrasive and the friction coefficient should be lower. It is possible to obtain friction coefficient close to teflon on the material surfaces by boronizing [12].

In this study, it is aimed to improve the wear properties of AISI 316 stainless steel by boronizing process. In this context, boronizing was carried out at 950 °C for 3 hours using atmospheric-controlled furnace with pack-boronizing method. As a result, the un-boronized sample and the boronized sample were subjected to friction test under the same conditions and it was determined that boronizing process increased the wear resistance of the surface of the AISI 316 stainless steel.

2. MATERIAL AND METHOD

The chemical analysis of the sample to be used in the experiments was carried out with optical emission spectrometry before starting the boronizing experiments. As can be seen from the Table 1 below, it was determined that the test samples were AISI 316 stainless steel.

Table 1. Chemical analysis of AISI 316 stainless steel used in boronizing treatment

Steel Type (AISI)	Alloying Elements (% wt)								
	C	Mn	Si	P	Cr	Ni	Mo	S	N	Fe
316 (X5CrNiMo17-12-2)	0.079	2.010	0.741	0.045	17.415	11.870	2.600	0.029	0.090	Bal.

Samples similar to the projectile core were prepared for the boronizing treatment and other examinations. The flat surface of the samples is used for metallographic examinations and the lower oval end portion is used for friction tests. The pictures taken before and after boronizing of the prepared samples for boronizing test are shown in Figure 1 below.

Figure 1. The samples used in the experiments before and after boronizing

A heat treatment pot made of AISI 304 Stainless Steel was used for boronizing treatment. The sample to be boronized is placed inside the pot to be all the surfaces are covered with Ekabor 2 powder. Then, the remainder of the pot was covered with silica sand to prevent the oxygen entering from the outside and the lid of the pot was closed. Finally, the pot was placed in a heat treatment furnace set to 950 °C for 3 hours at this temperature and allowed to cooling in room temperature at the end of the treatment.

After boronizing treatment, the flat part under the sample was prepared metallographically. Metkon brand, rotary disc device is used for metallographic sample preparation. The image of the device is shown in Figure 2 below.

Figure 2. Metallographic sample preperation device

For metallographic examination, sanding with 80 to 1200 mesh sandpaper followed by polishing with 10 µm Al_2O_3 polisher, and finally etching with Royal Water (3 unit HCI + 1 unit HNO_3) were used. After the metallographic preparation, the microstructure images of the boronized sample were first taken by Bulut Makine brand optical microscope, then by Jeol 5410 LV SEM (BEI). The pictures of the microscopes are shown in Figure 3 and 4 below.

Figure 3. Optical microscope and image analysis unit

Figure 4. SEM and EDX unit

Back scattered electrons used in scanning electron microscopy; they allow the elements to color according to their atomic numbers. For coloration, they use white, gray tones and black color. The electrons that hit the surface of the material are captured by the back-scattered electron detector and then colored them by using the image rotation unit according to the atomic number. In this way, the atomic number is the smallest element in the light color, and the atomic number is the biggest element appears in darker color. However, this coloring is only valid applicable to the sample being tested at that moment. This varies in terms of color grading, depending on the content of each material. This situation is completely related to the elements contained in the samples [13].

The greatest advantage of the boronizing process is the high hardness of the steel. Hardness depends on the main steel type and FeB or Fe_2B phases formed on the surface [8]. Vickers hardness measurement method was used to detect changes in the hardness of the boronized AISI 316. Measurements, 5 µm intervals from surface to matrix, 100 g. weight is used. For hardness measurements, Duroline-M brand Micro Vickers device is used. This device is shown in Figure 5 below.

Figure 5. Micro Vickers hardness device

Prior to the friction tests, XRD analyzes were performed for the determination of the all phases in the boride layer of the boronized sample. For XRD analyzes, Philips Panalytical X-Pert Pro brand X-Ray Diffractometer is used. X-Ray Diffractometer is shown in Figure 6 below.

Figure 6. X-Ray Diffractometer (XRD)

Friction tests were carried out to examine the wear behavior of the samples, of which all measurements were completed. The tests were performed at a distance of about 1000 m at a speed of 0.785 $m.s^{-1}$ under 2.5N load, and the friction coefficients of the boronized and un-boronized samples were determined. AISI 316 L stainless steel is used as abrasive disc. The surface roughness value of the disc given in figure 7 below was measured as 189 $kg.mm^{-2}$.

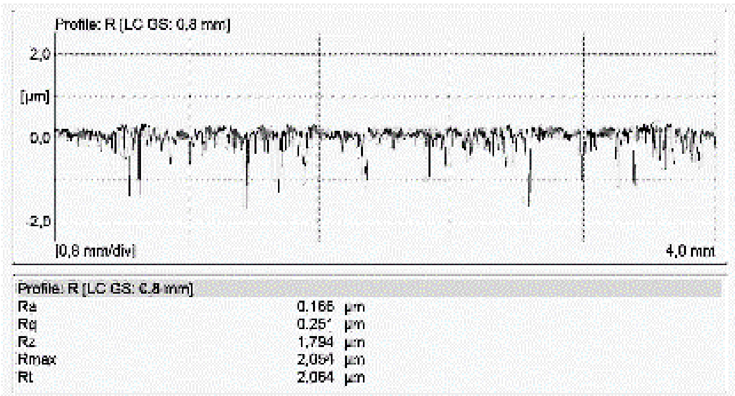


Figure 7. 316 L abrasive disc surface roughness values

The schematic drawing of the pin-on disk device used in friction tests is shown in Figure 8 below.

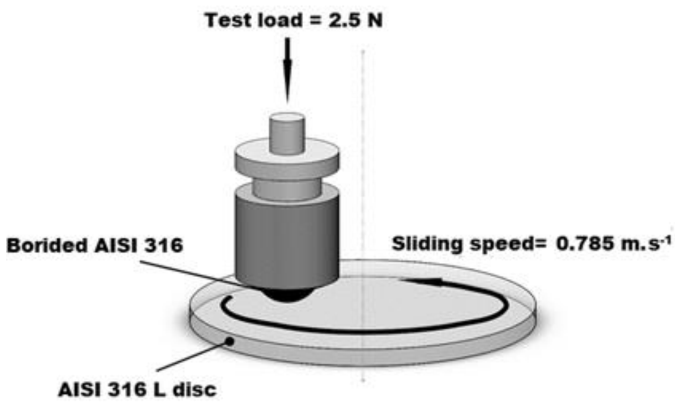


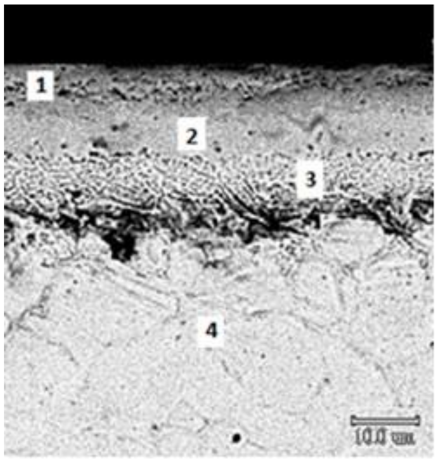
Figure 8. Schematic figure of pin-on disc friction test device

3. RESULTS AND DISCUSSION

In this study, the positive effect of wear resistance of boronized AISI 316 stainless steel was investigated. After boronizing treatment performed at 950 °C for 3 hours, columnar morphologic boride layer consisting of FeB and Fe_2B phases was obtained. The boride layer formed on the boronized steels is revealed in many studies [14,15,16] consisting of FeB and Fe_2B. This is also the case in this study. This is due to the reduction of boron atoms emitted from the surface during diffusion. Thus, the FeB phase which is rich in boron element, followed by Fe_2B phase which is containing less boron element, are formed the two-phase layer on the surface [8].

For the microstructure examinations, an optical microscope and SEM (with the help of back-scattered electrons) were used. Optical microscope and SEM (BE) images are shown in Figure 9 (a) and (b).

(a)



(b)

Figure 9. Optical microscope (a) and SEM (BE) (b) images of boronized AISI 316

In the SEM (BE) image, the outermost FeB phase appears darker than the next Fe_2B phase. This is because the amount of boron contained in these phases is different from each other. The amount of iron in FeB is less than Fe_2B, so its color is darker. On this image, the number 1 shows the outermost FeB phase, the number 2 shows Fe_2B phase, the number 3 shows transition zone, and the number 4 shows matrix structure.

For the chemical analysis of the transition zone between the boride layer and the matrix (zone 3 on the SEM image), the point analysis was performed with the help of the SEM-linked EDX unit, and as a result of this analysis, it was observed that, this region is rich by chromium. EDX analysis result shown in Figure 10.

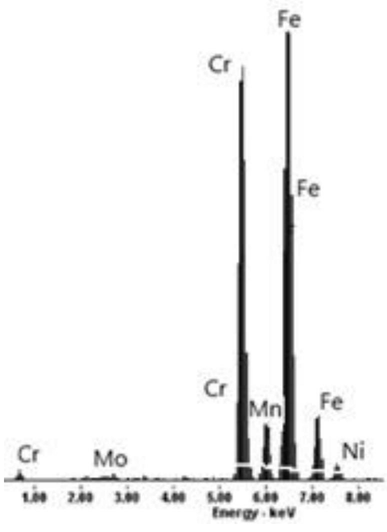


Figure 10. EDX analysis of the Cr-rich zone indicated by number 3 in the SEM image.

To determine the average thickness of the boride layer, 10 separate measurements were performed with the help of the image analyzer connected to the optical microscope. As a result of the measurements, the average boride layer thickness was determined as 21.8 ± 2.7 µm. The results of layer thickness measurements are given in Figure 11 below.

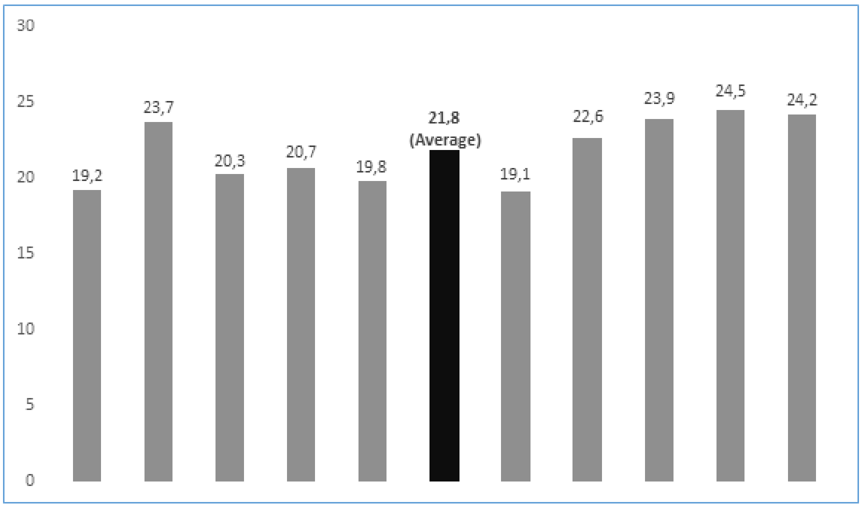


Figure 11. Average thickness of boride layer after 10 measurements

Vickers Hardness Measurement method was used to detect changes in the hardness of the boronized AISI 316. Measurements, 5 µm intervals from surface to matrix and, 100 g. weight is used. According to this, the FeB phase hardness has an average of 1947 kg.mm^{-2}, the Fe_2B phase hardness has an average of 1818 kg.mm^{-2}, the chromium-rich transition region hardness has an average of 425 kg.mm^{-2} and the matrix region hardness has an average of 319 kg.mm^{-2} were measured. The results of these measurements are given in Figure 12 below.

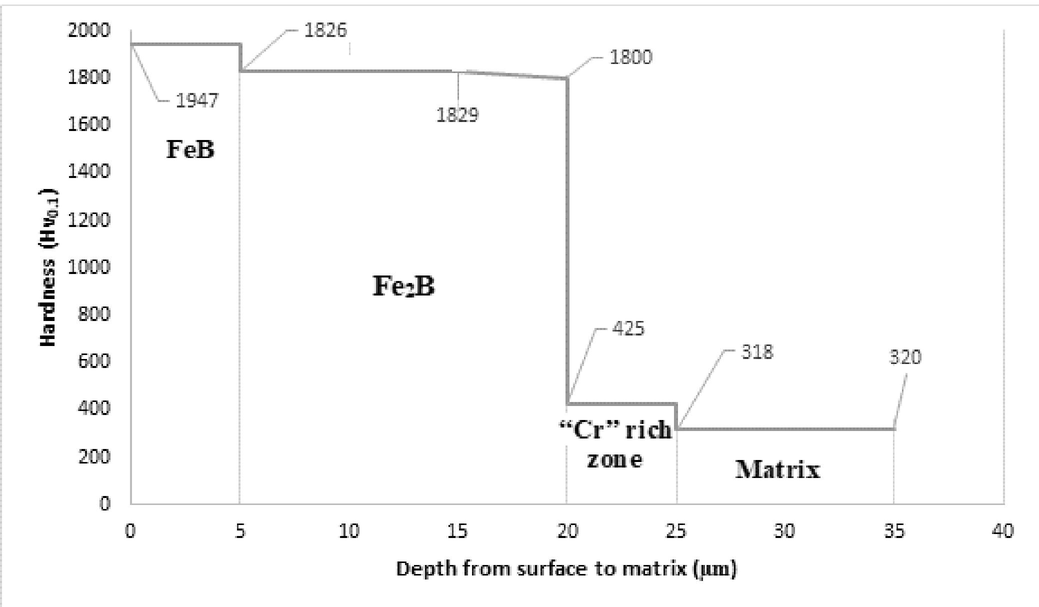


Figure 12. Hardness values of borided AISI 316 (kg.mm^{-2})

XRD analysis was performed to determine all the phases in the boride layer. According to the results of XRD analysis, all phases forming the boride layer; FeB, Fe_2B, CrB, Ni_2B and $Cr_2Ni_3B_6$. X-ray diffraction patterns of boronized AISI 316 steel is shown in Figure 13 below.

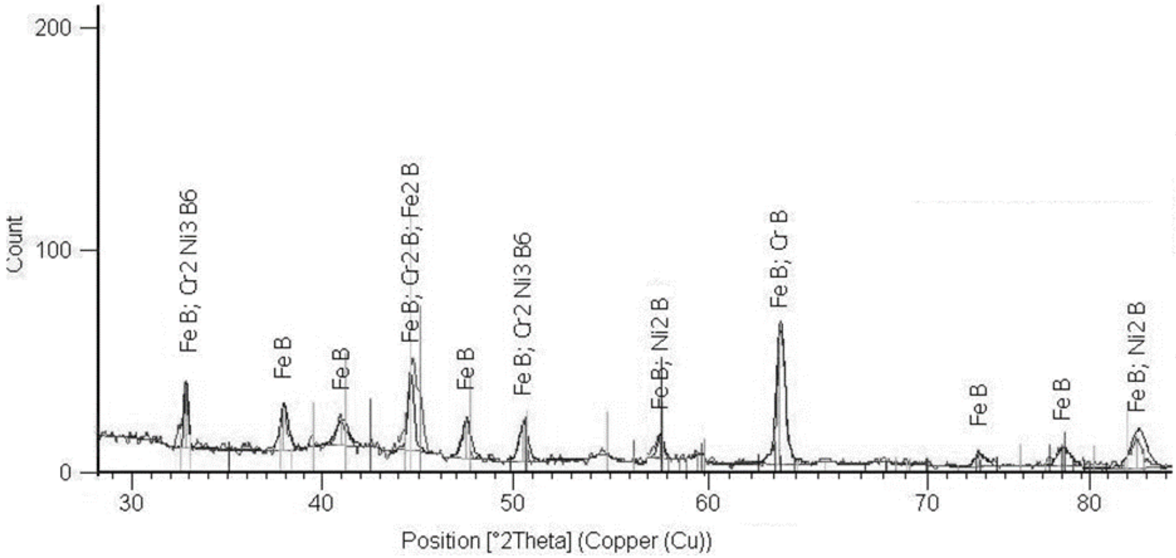


Figure 13. X-ray diffraction patterns of boronized AISI 316 steel

As a result of the friction tests performed under the same conditions where AISI 316 L stainless steel was used as abraded disc, the average friction coefficient of the boronized sample was determined to be 0.185 and the average of the un-boronized sample was 0.385. This result shows that, in terms of friction coefficient, there is approximately 2 times difference between boronized sample and boronized sample. The graph of the friction coefficients of the boronized and un-boronized AISI 316 is given in Figure 14 below. Also, in Figure 15, the graph shows the average friction coefficient datas.

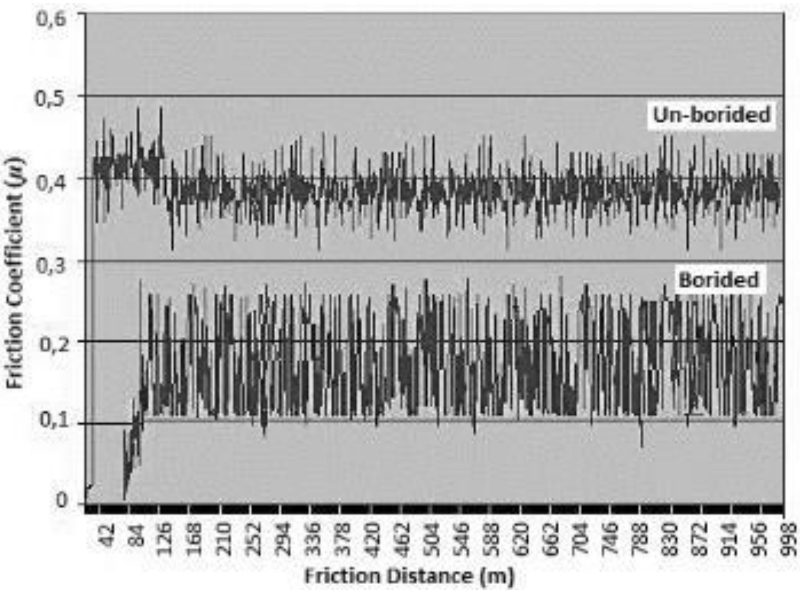


Figure 14. Friction coefficient graphs of borided and un-borided AISI 316

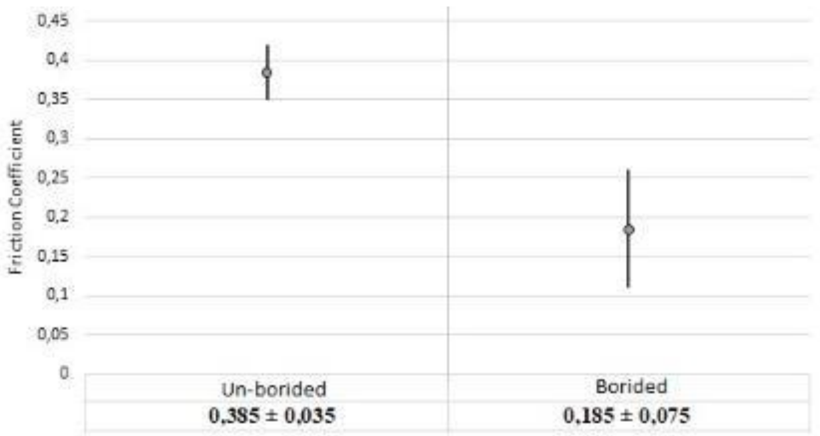
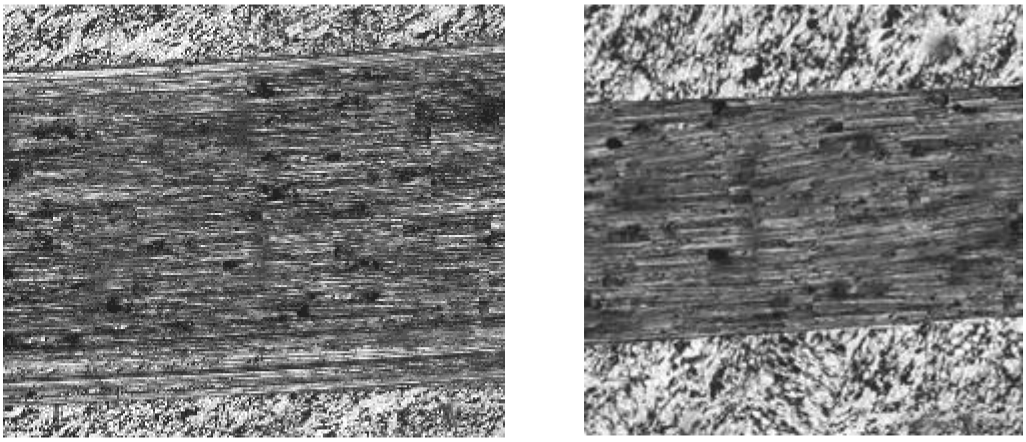


Figure 15. Average friction coefficients of boronized and un-boronized AISI 316

After the friction tests were completed, the wear rates of the 316 L disc were calculated based on the traces left by the boronized and un-boronized sample. Wear traces are shown in Figure 16 (a) and (b).



(a) (b)

Figure 16. Traces of wear on the 316 L disc; (a) by boronized steel, (b) by un-boronized steel

As can be seen from the figures, the trace formed by the boronized sample is wider than the un-boronized sample. This is due to the fact that the boride layer formed on the surface of the sample is too hard. After measuring the thicknesses of the wear traces on the disc, the wear rates of the 316 L disc were obtained using following equation [17]:

$$W = \frac{2\pi\left(R+\frac{L}{2}\right)\frac{r^2}{2}(\theta - Sin\theta)}{Sliding\ Distance} \quad (1)$$

In equation; W is the wear rate ($mm^3.Nm^{-1}$), L is the wear track thickness (mm), R is the radius of the wear scar (mm), r is the radius of ball (mm), and θ = 2 arcsin (L/2r) (radian).

As a result of the calculations; the wear rate of the 316 L disc by the boronized sample was found to be 172.7 x 10^{-6} mm^3 x Nm^{-1} and the wear rate by the un-boronized sample was found to be 62.88 x 10^{-6} mm^3 x Nm^{-1}. In a similar study, when the wear test results of AISI 316 boronized at 900 °C for 2 hours were examined. The coefficient of friction of the un-boronized sample was 0.7 and the wear rate was 90 x 10^{-6} mm^3 x Nm^{-1}, the friction coefficient of the boronized sample was 0.6 and the wear rate was 15 x 10^{-6} mm^3 x Nm^{-1} [17].

As a result, it is possible that the boronizing of AISI 316 stainless steel, using by pack-boronizing method. The boride layer formed on AISI 316 has a columnar morphology, and visually formed FeB and Fe_2B phases. Layer thicknesses show a homogeneous distribution on the sample surface. An ultra-hard boride layer is formed on the surface of boronized AISI 316, thus it is possible to use in places requiring excessive surface hardness, besides, boride layer is a structure which is very resistant to wear compared to the material which is formed on and it can be preferred in places where wear resistance is required.

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