Surface modification method of duplex type stainless steels by the pack boriding process

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Abstract
This work presents the investigation of a boriding process on two grades of stainless steel namely UNS32750 super duplex stainless steel and UNSS31803 duplex stainless steel in order to improve material properties and possibly to reduce catastrophic failure of industrial components. Usage of duplex stainless steels has become customary in the fields of oil and refinery, marine and pipeline applications due to increased corrosion resistance; however, these materials exhibit low wear characteristics. To overcome this problem, in this work the pack boriding process was employed. Evaluation of effects of the boriding process on the microstructure and mechanical properties was performed using scanning electron and optical microscopy, Vickers hardness tests and wear tests. It was shown that the 4 h process resulted in the greatest boriding layer thickness yielding the maximum surface hardness of 1407 HV in the super duplex stainless steel UNS32750 while this value was 1201 HV in the duplex stainless steel UNSS31803. Wear resistance of borided materials were up to 6-fold greater than those of non – treated materials. Also, the borided duplex materials were shown to be more suitable for industrial applications for valve and shaft components as compared to the boronized super duplex stainless steel.

Keywords: UNS32750; UNSS31803; SEM; wear; hardness.

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1. INTRODUCTION

Tribological properties of the duplex stainless steel family limit the usage of these metallic materials in industrial applications. Although these steels exhibit good corrosion resistance and mechanical strength, the wear resistance characteristics are poor. However, in hostile environments duplex stainless steels are the only alternative for standard austenitic stainless steels such as SS 304 and SS 316. Good strength and corrosion resistance of duplex stainless steels are due to the presence of chromium, nickel and nitrogen [1]. The increased mechanical strength, toughness and corrosion resistance makes these steels suitable in chloride environments [2]. These duplex and super duplex stainless steels, having increased pitting resistance [2] are applicable in the fields of oil and gas industry, marine applications, piping construction, chemical industry, and petrochemical plants [3]. Greater weldability and better mechanical properties of these steels as compared to those of austenitic stainless steels favor their usage in oil and gas refinery and chemical plants [4]. To increase the life of the sliding or mating parts, the material wear characteristics are considered as a dominant factor [5]. From the literatures, it is apparent that the valves and flanges require good tribological properties in addition to the corrosion resistance, which is not found in most of the materials. Good method to improve the material wear characteristics is by surface treatment. Among many surface treatment methods such as nitriding, carburizing, and nitrocarburizing, the boriding process based on the chemical treatment of the surface, was shown to

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result in increased hardness [6]. This process is not widely applicable in many industries unlike the conventional processes, even though it has many advantages likeproducing materials with high wear resistance, high galling property and good corrosion resistance [7]. The materials are heat treated with the boronizing medium at high temperatures to form extremely hard borided layers on the material surface [8]. Boron particles located on the boundaries between the grains, strengthen the bond between the boundaries and decrease the fragility [9]. The dual phase layer FeB and Fe_2B are formed on the outer surface and the growth kinetics are investigated by the analysis of the penetration depth of FeB and Fe_2B [10]. For low alloy steels the formation of FeB and Fe_2B increases the thickness of the boride layer as compared to that in high alloy steels because of the affinity of boron atoms for iron [6]. The FeB layer is harder than the Fe_2B layer, which promotes brittleness and surface defects on the material [11]. To overcome the difficulties of increasing the layer thickness in high alloy materials, the process temperature and time have to be increased [12-14]. The super duplex stainless steels contain several alloying elements such as chromium, nickel, manganese and molybdenum, which also reacts with boron and reduce the thickness of the iron boride layer [15]. Effects of the boronizing method on wear characteristics and the friction behavior were investigated on the Din 20 $MoCr_4$ steel material showing the hardness of the formed boride layer in the range 1475 to 1848 $HV_{0.05}$, while FeB and Fe_2B were observed on the surface of the material [16]. Many dual process treatments were also attempted like plasma nitriding, nitroboronizing, alumina boronizing and boronitrocarburizing [17]. Campos Silva *et al.* [18] studied the scratch and adhesion properties of the nickel boride layer on Inconel 718 super alloy formed by the powder-pack boriding process carried out at 900 ^oC for 2 to 6 h. The same process was applied in another study on a TB2 alloy and a diffusion model was proposed for the growth of the boride layer thickness [19]. A boronizing process was applied on the materials AISI 420 and 5120 resulting in approximately 5-fold lower wear rate as compared to that for theun-borided materials [20]. A solid boriding thermo-chemical treatment by using two boriding agents was applied on AISI H13 steel resulting in improved wear resistance as revealed by pin on disc tests [21]. Sista *et al.* [22] suggested that electrochemical boriding is an ultra-fast technique applicable for Inconel alloys to produce hard and protective layers on the material surfaces. From the literature, it is observed that duplex stainless steels have wide applications in the construction of pumps, shafts and valves, pressure vessels for petroleum, as well as in oil refining industrial processes and paper industry; however, for these applications high wear resistance is required [2]. Therefore, in this work we have performed experimental investigations of using a pack boriding process on UNS32750 and UNSS31803 stainless steels to improve the material mechanical and metallurgical properties.

2. EXPERIMENTAL METHODS

2. 1. Materials selection

The materials selected for the proposed work are super duplex stainless steel and duplex stainless steel (UNS32750 and UNSS31803), which were procured from M/s Jagruthi metal industries, Mumbai, India with the aim to investigate possibilities for improvements of the material characteristics by applying a boriding process. The standard chemical compositions with mass fraction ranges of alloy elements for the selected materials are given in Table 1.

Table 1. Chemical composition of super duplex UNS32750 and duplex UNSS31803 stainless steels [23]

Material	Content, wt.%									
	C	Mn	Si	P	S	Cr	Mo	Ni	N	Cu
UNS32750	0.03*	1.2*	0.8*	0.035*	0.02*	24-26	3-5	6-8	0.24-0.32	0.5*
UNSS31803	0.03*	2.0	1.0	0.03*	0.02*	21-23	2.5-3.5	4.5-6.5	0.08-0.2	-

*maximum

The boronizing medium consists of 90 % B_4C powder, having 325 mesh size, added with the diluent silicon carbide at 5 % and activator KFB_4 at 5 % (all chemicals procured from Alpha Aesar- Thermo Fisher Scientific, Mumbai, India). The SEM image of the boronizing medium is show in the Figure 1.

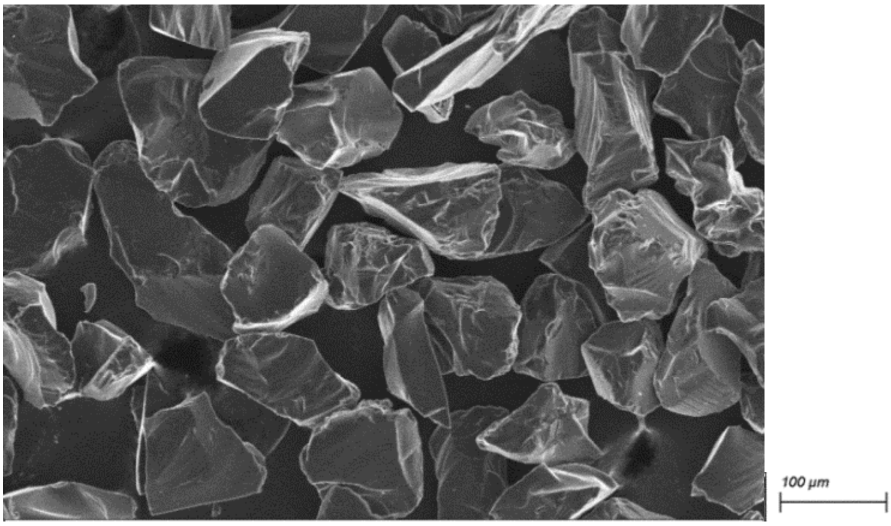

Figure 1. SEM image of the boriding medium

2. 2. Preparation of samples

Before the boriding process, surface grinding was carried out to the specification of 10 mm thickness to achieve the surface roughness of below 1.5 μm to increase the affinity for borondiffusion. The prepared cylindrical samples (ϕ 55 mm and 10 mm thick) were cleaned by acetone washing and individually kept in a SS304 container, covered with 3 mm of the boriding medium. Considering the environmental friendliness, ease of operation and low cost, the solid powder pack boriding process is used instead of toxic liquid and gas boronizing processes [24].

The workflow is briefly given as flowchart in Figure 2.

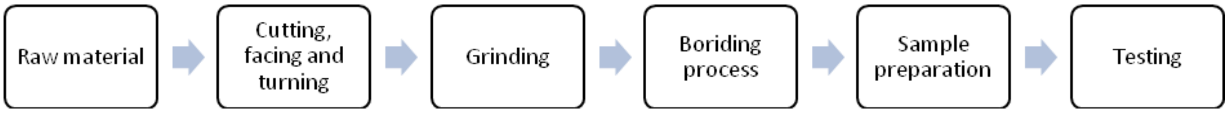

Figure 2. Work flow of the experiment

2. 3. Boronizing process

The electric furnace (Indfurr, Chennai, India) with specifications of 4.5 kW power and 1200 °C temperature was used for the boriding process. Initially a trial run was performed in order to select the process parameters. Based on the literature, the suggested temperatures for duplex stainless steel types are above 950 °C [25]. It was also inferred in the trial that at 1075 °C and the process duration of 5 h scales are formed due to formation of brittle FeB. Hence based on the industrial experts guidance the process parameters for the duplex stainless steels were fixed at the temperature of 1050 °C with varying process time of 2, 3 and 4 h. For each test three replicate experiments were performed.

2. 4. Testing methods

This investigation fulfils standards for metallographic analyses including determination of the microstructure, boride layer thickness, wear characteristics, hardness and surface roughness. In specific, scanning electron microscopy (SEM), optical microscopy, energy dispersive spectrometry, Vickers hardness tests, surface roughness tests, wear tests, and spark emission spectrography were used to determine metallurgical and mechanical characteristics of the borided samples. The surface roughness values were measured before and after boriding with the help of a surface roughness tester TR110 (Time Group, China).

An important factor considered for improving the wear resistance is the surface hardness which shows the capability to resist indentation. With the increase in hardness wear and corrosion resistances are proportionally improved. Hardness was determined by a Vickers micro hardness tester (CL/ME/MVIC40 with the FSA model, India). The samples are prepared in a bakelite mould process and the molded samples are polished to various grit sizes and finally diamond polished for achieving a suitable surface finish. By using the Vickers micro hardness tester, the hardness values are determined on the surface, outer boriding zone, transition zone and the core with the indentation force of 50N.

By following the standard of ASTM E03-01 and the Metals Handbook [26] microstructure was analyzed by using an optical microscope with image analysis, (MVMS1310 Metavis, Medimage Technologies, India) at 25.3 °C. The etchant used in the mould was the mix of picric acid (1 g; 2,4,6 –trinitrophenol, Sisco Research Laboratory, India), HCL (5 cm^3; Sisco Research Laboratory, India) and ethanol (100 cm^3; Sisco Research Laboratory, India)

A drum type wear testing machine (Profilic Engineers, India) was used to compare wear characteristics of the materials by the weight loss method. The total revolution was 84 times, and the rotational frequency was 40 ± 1 rpm as per the industrial standard. The load applied on the material was 9.807 N (*i.e*. 1 kgf) and 60 grade coarse abrasive sheets were used. Initially before the wear test the sample was prepared was cut in a circle with the specified diameter of 15 mm and the thickness of 10 mm. The weight was measured before and after the wear test for both investigated materials. The difference between the initial and final weights is considered as the abrasion loss. For each test three replicate measurements were carried out and the average values were noted.

The samples for SEM analysis were cut into 8 mm squares by a wire cut EDM machine. By using various grit sizes of emery sheets the samples were carefully mirror polished and cleaned using acetone. The etchant was then applied on the samples and the cut section morphology was analyzed by using field emission scanning electron microscopy (SIGMA with Gemini column, Carl Zeiss, USA). The elemental composition of the materials was analyzed by energy dispersive spectroscopy (EDS) by using an EDAX analyzer (Nano XFlash Detector, Bruker, Germany)

Composition of the materials wasexamined by preparing samples (ϕ10 mm) for spark emission spectrography (OBLF, Germany). The spark emission test was taken at one spot.

3. RESULTS AND DISCUSSIONS

3. 1. Chemical composition

Chemical composition of both stating materials was determined by spark emission spectroscopy (Table 2) confirming the specifications.

Table 2. Chemical composition of super duplex UNS32750and duplex UNSS31803 stainless steels used in the present study

	Content, wt.%								
Material	C	Mn	Si	P	S	Cr	Mo	Ni	Nb
UNS32750	0.018	0.70	0.52	0.02	0.009	24.36	3.86	6.34	0.269
UNSS31803	0.015	1.35	0.8	0.017	0.012	21.74	2.83	4.59	0.112

3. 2. Surface roughness

Surface roughness is an important material characteristic, and it is altered by diffusion of the boriding medium.

Therefore, the average surface roughness values (R_a) were measured before boriding to find the extent of improvement in R_a values after the process. Decreased surface roughness after boriding was also reported in literature [12]. The measured values presented in Table 3, indicate that the boriding process slightly smoothens the rough surface due to the diffusion of boron.

Table 3. Surface roughness values

Material	Boriding duration, h	R_a / µm (before boriding)	R_a / µm (after boriding)
super duplex stainless steel UNS32750	2	0.71 ± 0.08	0.59 ± 0.06
	3	0.66 ± 0.06	0.56 ± 0.04
	4	0.82 ± 0.06	0.58 ± 0.06
duplex stainless steel UNSS31803	2	0.93 ± 0.08	0.68 ± 0.03
	3	0.63 ± 0.05	0.48 ± 0.04
	4	0.56 ± 0.04	0.5 ± 0.03

3. 3. Hardness

Average hardness values based on 3 readings from each area are presented in Figure 3. Hardness values of the outer boride layer, transition zone and core are measured in the molded sample and the surface hardness is measured at the boronized surface. All of the three samples (*i.e.* boriding for 2, 3 and 4 h) for both materials were analyzed. Hardness in the core area of super duplex UNS32750 and duplex UNSS31803 stainless steels is around 290 HV and 240 HV, respectively.

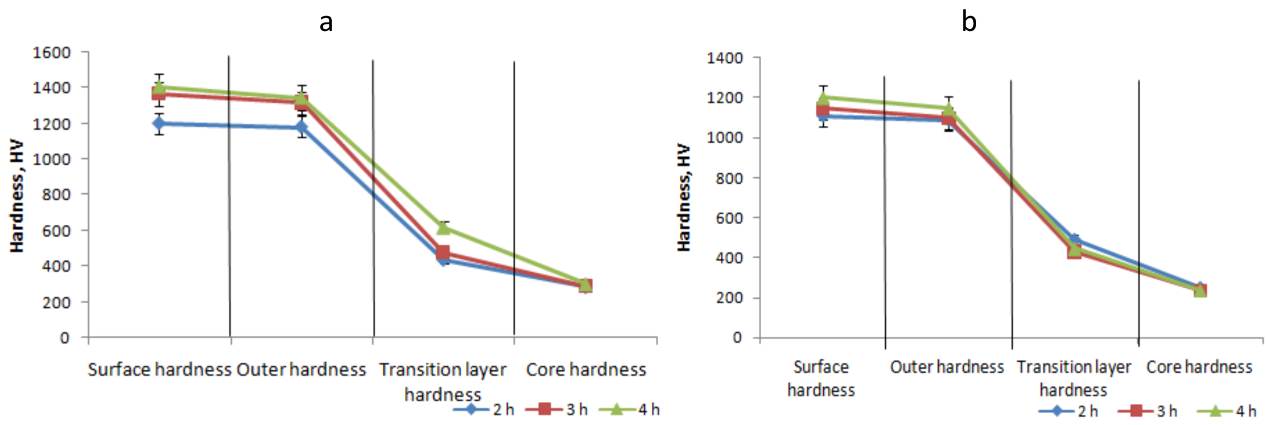

Figure 3. Surface hardness and case depth for (a) super duplex stainless steel UNS32750 and (b) duplex stainless steel UNSS31803

Hardness values for both investigated materials improved drastically. The outer surface shows the maximum hardness level, followed by the boriding layer and transition zone. The higher hardness of the core in the super duplex stainless steel UNS32750 than in the duplex stainless steel UNSS31803 may be due to the presence of more chromium in addition to nickel and nitrogen.

From the obtained results it is inferred that the hardness at the surface, outer and transition zone is increasing over time of boriding. The increase in hardness was reported with the increase in temperature and time [24]. This may happen due to longer time for a boron particle to diffuse to the base metal which increased the thickness of the boriding layer. As a result, the surface hardness increased 5-fold in the duplex stainless steel UNSS31803 and 4.5-fold in the super duplex stainless steel UNS32750 as compared to the base material hardness. This will produce effects on the wear and corrosion resistance.

The decreasing hardness trend in various zones as observed in Figure 3 may be due to the boron diffusion. Longer time for diffusion leads to formation of dual phase of FeB and Fe_2B on the surface and the boronizing zone. The FeB is harder and becomes brittle on the surface and followed with the Fe_2B phase. Diffusion is lower in the transition zone as compared to that on the surface. Boron diffusion practically does not take place in the core area which therefore exhibits the same hardness as the base material. Thus, Figure 3 clearly shows the increased surface hardness of both materials as compared to that of the base materials.

3. 5. Boride layer thickness and metallographic evaluation

Thickness of the boride and transition layers was determined by optical microscopy as shown in Figure 4(a). The interface layer is the transition zone between the boriding, and the base microstructure as shown in the micrograph in Figure 4(b). The case depth increases as the boriding time is increased. The boriding layer thickness in the duplex stainless steel UNSS31803 is slightly greater than that in the super duplex stainless steel UNS32750 due to lower amounts of alloying elements in the former case.

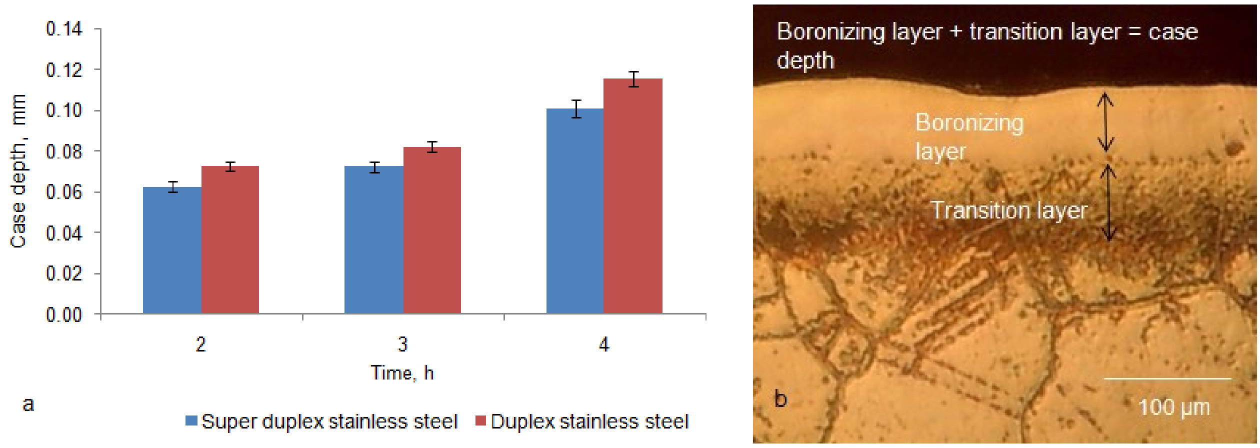

Figure 4. (a) Case depth as a function of boriding time; (b) representative optical micrograph on the right shows the boriding and interface layers on the cross-section of one sample

The microstructural analysis was carried for all samples while SEM images and EDX analysis are presented in Figure 5 for materials exposed to boriding for 4 h.

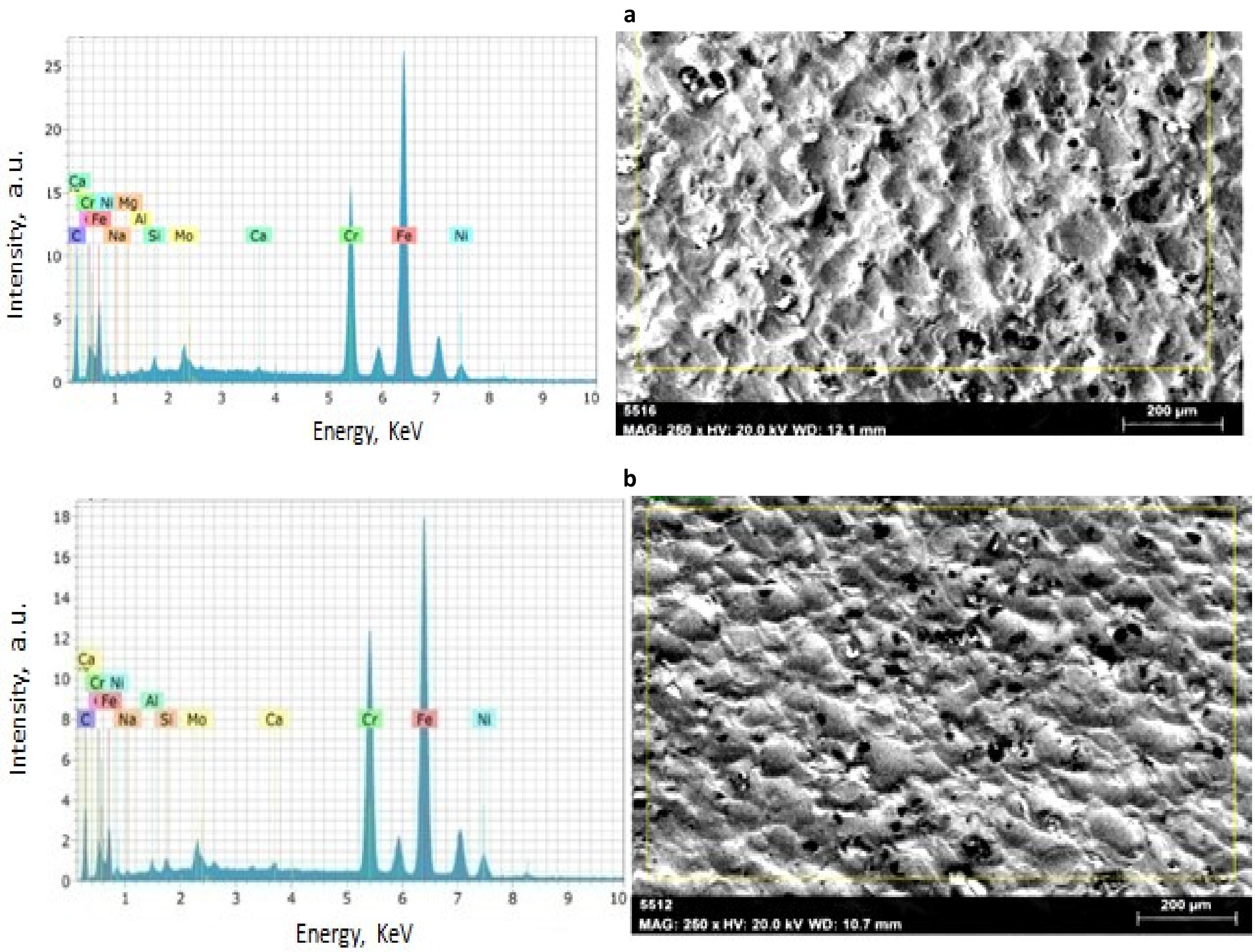

Figure 5. SEM micrographs and EDX analyses for materials exposed to the 4 h boriding process for: (a) duplex stainless steel UNSS31803, (b) super duplex stainless steel UNS32750; (black dots in SEM micrographs correspond to surface pores; scale bar: 200 µm)

It can be observed that low porosity appears on the surface of the materials as revealed by black dots on the surfaces (Figure 5). The porosity is lower in the duplex stainless steel UNSS31803 as compared to the super duplex stainless steel UNS32750 due to lower amounts of alloying elements in the former case.

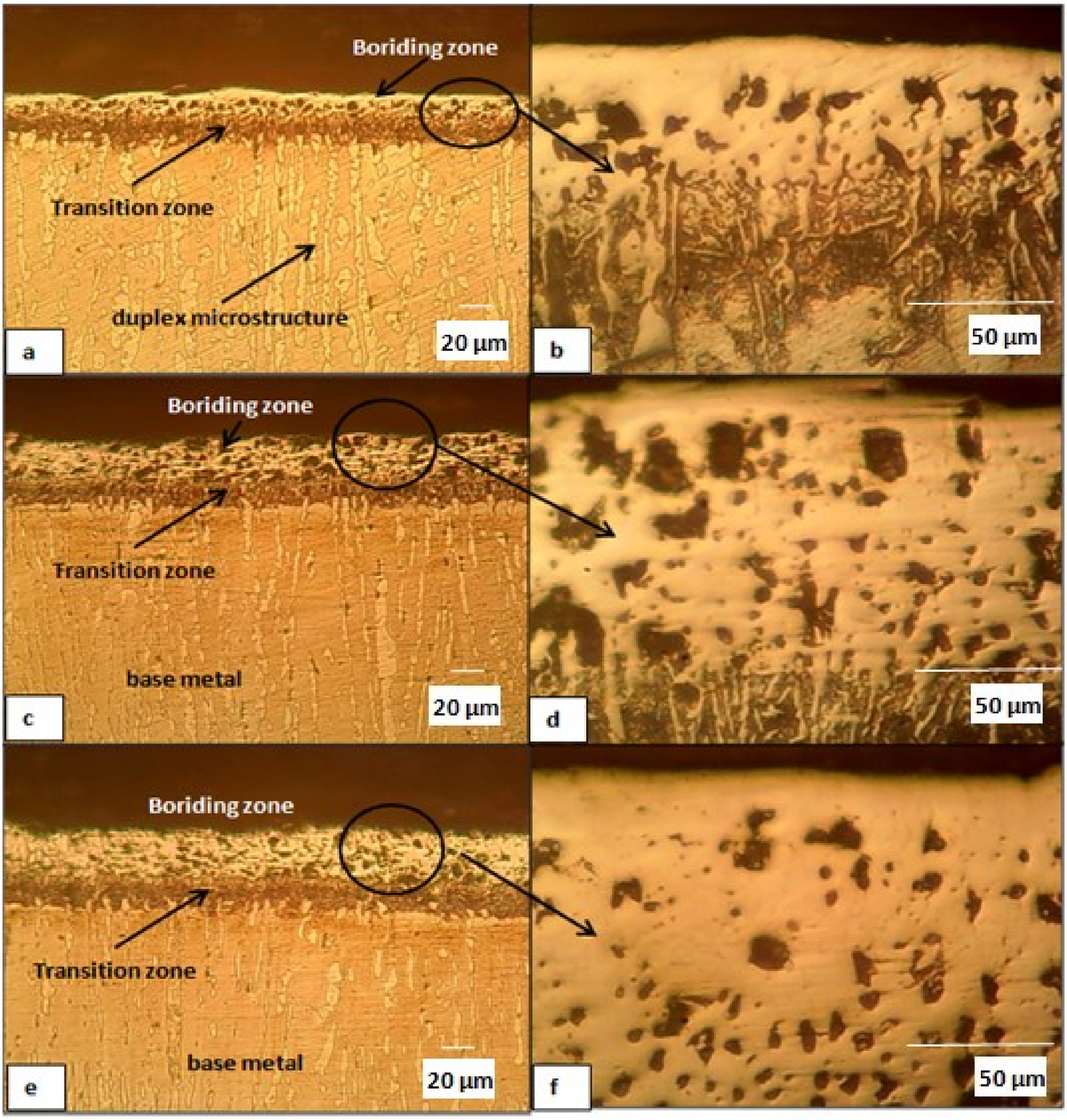

Figure 6. Optical microscopic images showing microstructure of duplex stainless steel UNSS31803 exposed to boriding for: a, b) 2 h; c), d) 3 h; e, f) 4 h

Figure 6 shows microstructures of the UNSS31803 material exposed to boriding for 2, 3 and 4 h. The microstructure of the base metal clearly shows the duplex stainless steel. The total dispersion of boron is the combined layer thickness of boriding layer and transition layer. For 2 h boriding on the surface of the base metal the total dispersion of boron layer is 73 μm, in which the boriding layer is about 36 μm thick and the transition zone is formed ~38 μm thick below the boriding layer. (Figure 6a, b) The transition layer indicates the saw tooth morphology, which is commonly seen as columnar like structure after the boriding layer. With the increase in process duration to 3 h the thickness of boriding and transition layers is measured as ~47 μm and ~35 μm, respectively as shown in Figure 6 c, d.

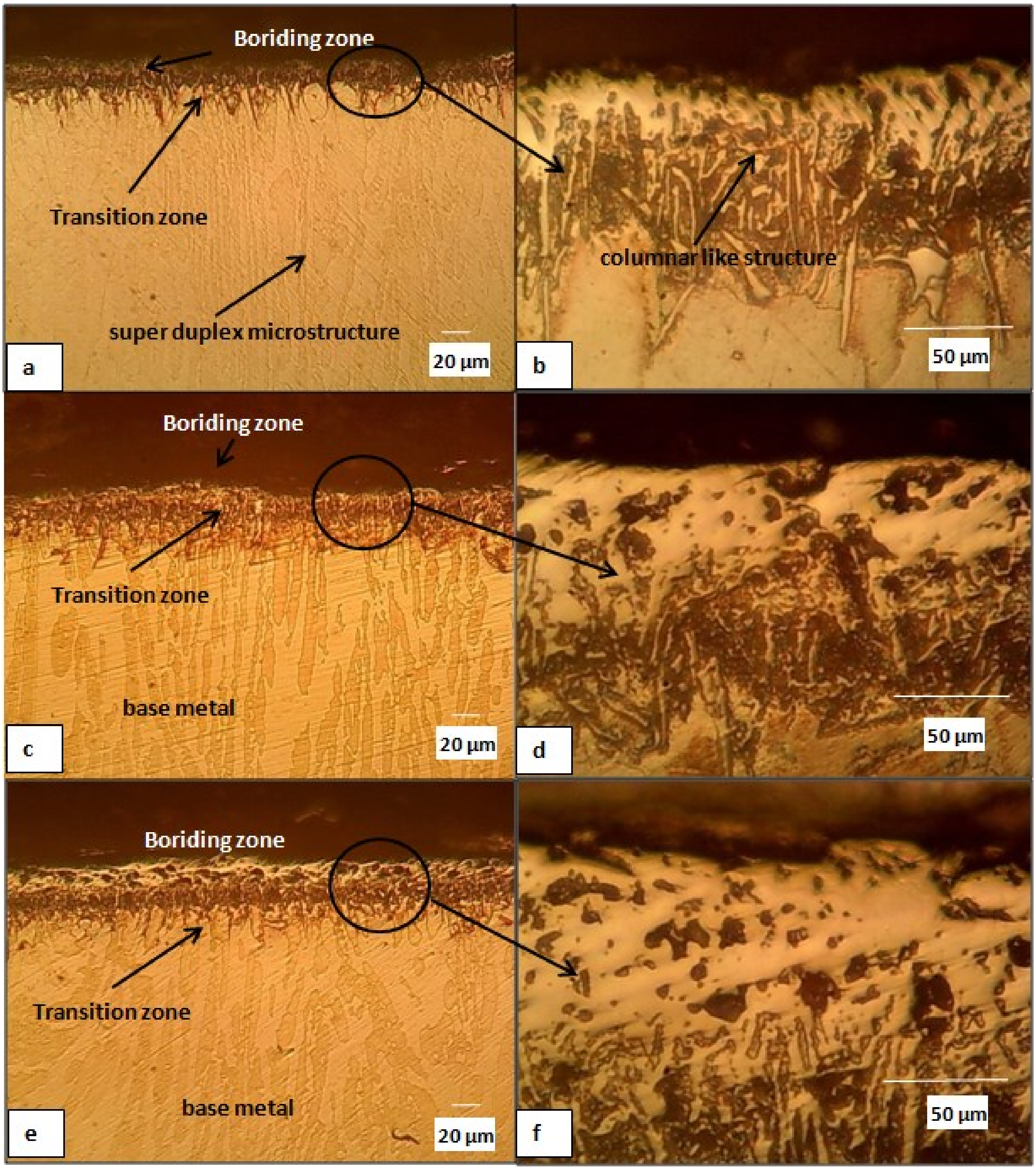

Figure 7. Optical microscopic images showing microstructure of super duplex stainless steel UNS32750 exposed to boriding for: a, b) 2 h; c, d) 3 h; e, f) 4 h

Further increase in boriding duration to 4 h results in increased thicknesses of both boriding and transition layers of around 116 μm shown in Figure 6 e, f. Due to the extension of time the boronizing medium have more time for better diffusion and the layer thickness is more compared to the 2 h and 3 h process.

Boriding of the super duplex stainless steel UNS32750 for 2 h resulted in formation of a very thin boriding layer of 5 µm and transition layer thickness of ~58 µm (Figure 7 a, b). With the increase in the boriding duration the boriding layer thickness is increased. The super duplex microstructure is clearly seen in the base material. The uneven iron boride layer formation and low thickness may be due to the restriction of bonding between the iron and boron.

The other alloying elements shared the boron atoms due to the high contents such as NiB2, CrB_2. The saw tooth microstructure was observed in the transition layer. The layer thickness formed for 3 h process is ~72 µm and for 4 h process it is ~101 µm respectively which also shows the increase of time of boriding process increases the boriding layer thickness. Even though the temperature was kept constant at 925 °C, the boride layer thickness increases with the extension of time to 4 h.

3. 6. Wear test analysis

The removal or deformation of the material at the surface corresponds to the wear property of the material. There are many wear types such as adhesive, abrasive, and fretting wear, which damage the material and decrease the life of the products. Thus, to find the wear resistance, in this work the effects of abrasive wear was investigated. The abrasion loss was calculated from the difference between the final weight after the wear test and the initial weight before the test [27] and the results are shown in Figure 8.

The weight loss of untreated samples measured as 0.25 and 0.32 % for duplex UNSS31803 and super duplex UNS32750 stainless steels. The weight loss after boriding for 4 h for the duplex stainless steel UNSS31803 was only 0.04 % while for the super duplex stainless steel UNS32750 was 0.12 %. Both materials show significantly lower abrasive wear losses after the boriding process and hence proved that the wear resistance of the boronized samples is higher and that the life of the final products would be improved. In specific, the wear loss is ~6-fold and 2.5-fold lower for the borided duplex stainless steel UNSS31803 and the super duplex stainless steel UNS32750 samples, respectively, as compared to the corresponding values of non-treated samples.

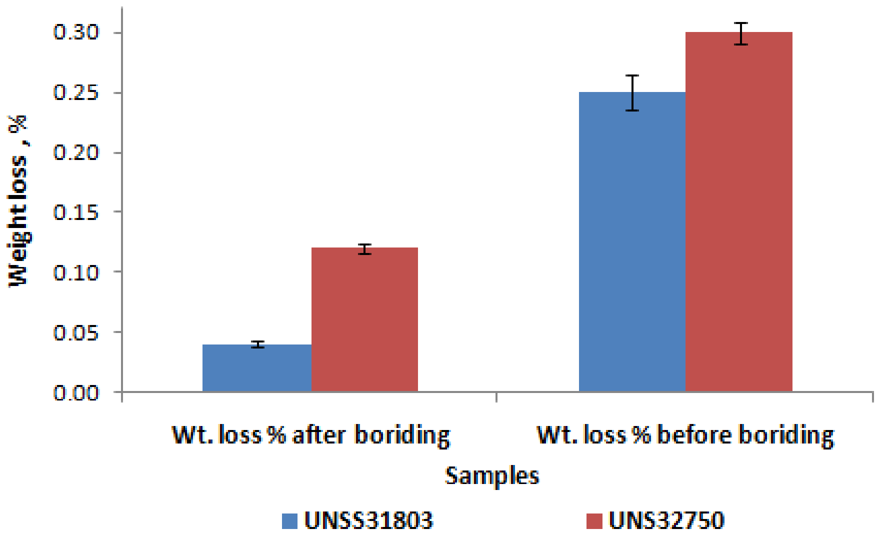

Figure 8. Weight loss comparison in the wear test

The samples submitted to the wear test were analyzed regarding the surface roughness in terms of Ra and Rz values. The average roughness R_a and R_z values for the super duplex stainless steel UNS32750 were ~6 µm and ~13 µm, respectively, while for the duplex stainless steel UNSS31803 these values were ~4 µm and 9 µm, respectively. Thus, surface roughness was lower of UNSS31803 samples as compared to that of UNS32750 samples.

The obtained results clearly indicate that the increased boride layer thickness in the duplex stainless steel UNSS31803 sample ensures lower surface roughness as compared to that in the super duplex stainless steel UNS32750 sample. The difference in average surface roughness (R_a) value was ~2 μm but R_z values differed by 4 μm between the two materials. It was suggested in literature that the variation was due to the formation of a thicker layer of FeB and differences in the composition of material substrates [28]. Due to higher diffusion of boron and a thicker boride layer in the UNSS31803 sample, the wear resistance increased, and the wear depth was reduced causing lower surface roughness than that of the UNS32750 sample.

4. CONCLUSION

Investigation of the boriding process on UNS32750 super duplex stainless steel and UNSS31803 duplex stainless steel samples was carried out with the aim to improve the wear resistance of these materials. The following observations were derived from the experimental analysis.

- In both investigated materials, the borided samples shows slight decrease of surface roughness values compared to the non treated samples. The results may be due to the diffusion of boron which smoothed the surface.
- Longer boron diffusion by extending the boriding process time increased the material hardness. The maximal value of the surface hardness of 1407 HV was achieved in the super duplex stainless steel UNS32750 sample after 4 h of boriding while this value for the duplex stainless steel UNSS31803 sample was 1201 HV. Measured hardness values decreased from the surface boride layer to the interface layer and further to the base material.
- Surface morphology of the samples after boriding showed uneven surfaces although the surface finish improved. Microstructures of both materials confirmed the duplex stainless steel type. Thicknesses of the boride and interface layers increased over time of boriding in both materials due to prolonged boron diffusion. After 4 h of boriding the total boron diffusion layer in the duplex stainless steel UNSS31803 was ~115 μm thick while it was ~100 μm thick in the super duplex stainless UNS32750. The prolonged boron diffusion increased the boride layer thickness and improved surface finish.
- Wear characteristics of boronized materials improved as compared to non-treated samples so that the improvement was ~2.5-fold for the super duplex stainless steel UNS32750 sample while even 6-fold for the duplex stainless steel UNSS31803 sample.
- From the results obtained in this experimental work it can be concluded that the duplex stainless steel UNSS31803 is more suitable for application of the boriding process with respect to the improvement in wear resistance. Still, the borided samples of both stainless steels are suitable for applications in pumps and valves in the oil and petroleum fields possibly preventing the irrecoverable losses by the improvement of surface properties. These borided materials could thus increase the life of products due to high wear resistance and good surface characteristics in addition to the higher corrosion resistance.

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SAŽETAK

Metoda modifikacije površine dupleks nerđajućih čelika postupkom boriranja

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Ovaj rad prikazije istraživanje procesa boriranja dve vrste nerđajućeg čelika: UNS 32750 super dupleks nerđajući čelik i UNSS 31803 dupleks nerđajući čelik, u cilju poboljšanja osobina materijala i smanjenja mogućnosti katastrofalnih otkaza industrijskih komponenata. Upotreba dupleks nerđajućih čelika postala je raširena u postrojanjima za vađenje nafte i rafinerijama, u pomorstvu i u cevovodima, a zbog njihove povećane otpornosti na koroziju. Međutim ovi materijali su pokazuju slabu otpornost na habanja. Da bi se prevazišao ovaj problem, u ovom radu je korišćen proces borirawa. Procena efekata boriranja na mikrostrukturu i mehanička svojstva izvršena je pomoću skenirajuće elektronske i optičke mikroskopije, testova tvrdoće po Vickersu i testova habanja. Pokazano je da je postupak boriranja u trajanju od 4 sata rezultirao najvećom debljinom boriranog sloja, dajući maksimalnu površinsku tvrdoću super dupleks nerđajućeg čelika UNS 32750 od 1407 HV, odnosno 1201 HV dupleks nerđajućeg čelika UNSS 31803. Otpornosti na habanja boriranih materijala bile su i do 6 puta veće od onih kod netrfetiranih čelika. Pokazalo se da su borirani dupleks materijali pogodniji za proces boriranja jer pokazuju veću otpornost na habanje u poređenju sa super dupleks nerđajućim čelikom, zbog povećane debljine sloja gvozdenog borida.

Ključne reči: UNS32750; UNSS31803; SEM; habanje; tvrdoća