



Article Influence of Boronizing on Steel Performance under Erosion-Abrasion-Corrosion Conditions Simulating Downhole Oil Production

Eugene Medvedovski *, Gerardo Leal Mendoza and Giovanny Vargas

Endurance Technologies Inc., 71-4511 Glenmore Trail SE, Calgary, AB T2C 2R9, Canada; gerardom@endurancetechnologies.com (G.L.M.); giovannyv@endurancetechnologies.com (G.V.) * Correspondence: emedvedovski@shaw.ca or eugenem@endurancetechnologies.com; Tel.: +1-403-724-6104

Abstract: Downhole heavy oil production and oil sand processing are associated with severe damage and failures of production equipment components, e.g., production tubing and pumping systems, due to erosion-corrosion resulting in processing losses, production downtime, high maintenance and replacement cost. Protective coatings (layers) on the production components mostly fabricated from low-alloy steels can be applied to minimize these problems. In the present work, the performance of hard boronized coating on carbon steel obtained through the thermal diffusion process and consisted of two iron boride layers (FeB and Fe₂B) was studied in synergistic erosion-abrasion-corrosion conditions simulating oil production environment in comparison with bare steel. Special wear testing equipment was designed and fabricated. In this testing, the inner surface of tubular sections was subjected to high velocity erosive flows of water-oil slurries containing silica sand and salts combined with rotating and oscillating motions of steel pony rods. Structural examination of the studied materials' surfaces and their profilometry after wear testing were conducted. The iron boride coating demonstrated significantly higher performance in abrasion and erosion-abrasion-corrosion conditions compared to bare carbon steel due to its high hardness, high chemical inertness, dual-layer architecture and diffusion-induced bonding with the substrate. The boronized steel tubing and casing with inner surface protection can be effectively employed in the most critical operation conditions.

Keywords: erosion; abrasion; corrosion; iron boride; steel; oil production; structure

1. Introduction

Wear and corrosion are serious problems in mineral and materials production and power generation. In particular, erosion-corrosion and abrasion issues widely occurring in downhole production and processing of heavy oil and oil sand result in sufficient losses in the industry. These problems lead to equipment failures and to related unpredictable shutdowns, necessary replacement of the damaged components, product contamination and possible environmental impact. Regarding the downhole oil production, the equipment components are subjected to high velocity flows of concentrated suspensions containing solid abrasive particles (e.g., silica sand, calcite, dolomite, clay-sand mud and others) with different sizes combining with mineral salts dissolved in corrosive media, often with a presence of corrosive gases. The depth of production wells of 2–3 km or more, which may be vertical or horizontal, is very common, and, in deep wells, oil and gas production requires elevated pressures and temperatures, which increase the above mentioned wear and corrosion problems. The problems associated with wear, especially erosion and abrasion, in the oil and gas industry have been reviewed and summarized in a few papers [1-3]. Thus, considering oil production problems, long production tubing strings and downhole tools are employed in every oil well utilizing the artificial lift systems, e.g., sucker rod pumps or progressing cavity pumps, with failures occurring because of wear and corrosion as the most-costly issues. In the artificial lift systems (see Figure 1) [4,5], wear occurs due to



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Copyright: © 2021 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (https:// creativecommons.org/licenses/by/ 4.0/). the reciprocation, up and down, motion of the rod and couplings against the tubing inner surface in pump completion. When the coupling-to-tubing slap occurs under some parts deviation or due to directionally drilled angles within the well bore, more severe wear problems occur. The rotational movement of the sucker rod and harmonic oscillation also lead to wear of the rod, couplings and tubing. When hard and abrasive sand particles occur between the moving counterparts, the lift components' wear accelerates dramatically. These severe service conditions result in hole-in-tubing failures, and, although different technical solutions were considered [2,5–7], no practical economical solutions could resolve this problem until now. All these situations require high integrity of materials for production equipment. The reliable materials for equipment components are also required for recovery and transporting of crude oil with oil sands, i.e., when oil contains high percentages of abrasive sand and ores. The presence of salts (chlorides, sulphates, bicarbonates) and bacteria in processing fluids, as well as some chemicals often introduced for the well stimulation, significantly accelerates wear problems due to the corrosivity of these fluids. The synergistic effect of wear (e.g., erosion, abrasion, fretting, etc.) and corrosion, which are the common service situations, creates more severe degradation of the materials compared to the "individual" actions (i.e., when erosion or corrosion acts independently). The total loss W, in this case, can be expressed as $W = E + C + \Delta$, where E—erosion loss, C—corrosion loss, and Δ —synergistic ingredient [1,8–15]. The outlined severe service conditions dictate a necessity of reliable production components to withstand erosion, abrasion, friction and corrosion.





Figure 1. Oil production pumpjack (**top**) and schematic of production tubing with artificial lift system [4,5] (**bottom**) (Reprinted with permission from Ref. [4,5] Copyright 2015 Elsevier).

The components of downhole production equipment (piping, tubing, casing, centralizers, different valves, chokes, nozzles and many others), as well as the transporting systems in the oil sand tailings slurry handling (e.g., piping, elbows, reducers, cyclones), are commonly made of carbon- and low-alloy steels. Due to severe service conditions, they experience a high level of degradation and failure. In order to reduce the failure problems of steel components, either more advance materials need to be employed, or low-grade steels should be protected. The use of expensive steels and alloys instead of carbon steels significantly reduces the production benefit, and, even in this case, the abrasionand erosion-related failures cannot be eliminated because these expensive alloys have lower hardness than processing materials (e.g., silica sand). Advanced ceramics with high hardness, high wear and corrosion resistance may be successfully employed for these and some other wear-related application conditions [16,17]; however, only components with rather small sizes can be produced because of the manufacturing capability limitation, but not long, several meter tubing. The lower fracture toughness of some ceramic materials limit their wide application. Polymers and elastomers experience significant degradation under the mentioned conditions, especially at thermal cycles, and their application is usually limited by service temperatures of below 200 °C. Special inorganic coatings on steels can be an effective route to protect steel components against wear and corrosion and to provide service at elevated temperatures (which presently became a common situation in downhole applications). In this case, particularly for tubular components with big lengths (up to 10 m) and high length-to-diameter ratios, the coating technology should provide protection of inner or inner and outer surfaces. The coatings should have high hardness, which needs to be significantly greater than the processing materials (e.g., sand), high chemical inertness, dense structure and sufficient thickness, i.e., they should retain their integrity after service in the mentioned conditions for certain time. In this regard, the coating technologies, such as CVD or based on the CVD principles, when hard and chemically inert materials are formed, may be very prospective [13,18]. These CVD-based technological routes, as opposed to PVD, thermal spray and many other processes, are applicable for the protective layer formation on various surfaces, e.g., the inner surface of tubing and intricate shape components.

Among different surface engineering processing options, the boronizing technology has high potential. This is a thermal diffusion process based on the CVD principles providing the formation of hard and chemically inert iron borides on surfaces of various steels and ferrous alloys. In this process, at high temperatures, gaseous boron species are formed within specially formulated mixes of certain inorganic powders, which include B-containing ingredients. Further, these B-rich species deposit onto the steel substrates, and finally boron atoms diffuse into the steel structure with consequent formation and growth of iron borides Fe₂B and FeB on the steel surface [19–25]. Due to the diffusion nature, the boronizing process and, therefore, the structure and thickness of the formed iron boride coating (protective layer) can be managed by varying temperature and time, substrate surface preparation, by creation of the conditions favorable of the B formation in the gas phase and some other factors [21,22,24,25]. Iron borides have a high level of hardness, high chemical inertness due to covalent nature and strong Fe–B bonds in the crystalline lattice and rather high thermodynamic properties (enthalpy) and melting point [25–29]. This advanced coating becomes the "integral" part of the components with no mechanical interface between the iron boride layers and the steel substrate with minimal probability of delamination. Because of this and owing to the technology's versatility, boronizing is successfully employed for the protection of steel components against wear and corrosion in different industrial applications, including in oil and gas and mineral processing [24,25].

The resistance of the employed materials (e.g., metals, ceramics, composites, polymers, coatings) to wear, corrosion and wear-corrosion, and their degradation mechanisms are defined, according to vast literature studies [8,9,12,16,17,30–37], by:

- (1) Their structure, composition and properties;
- (2) Design and geometry of production components;

(3) Process conditions, which include nature of the processing media (abrasive and corrosive) and service/application conditions, including pressure, temperature, character and hydrodynamics of flows, velocity, angle of impact, etc.

Data on wear and wear-corrosion resistance reported in literature depend on the testing method, testing conditions and employed apparatus. For erosion and erosioncorrosion studies, various testing methods and related equipment, such as a blasting unit (the simplest method for dry testing conditions) and different "pot" testers [38–41], slurry jet erosion testing devices [40–43], Coriolis erosion testing equipment [31,42,44,45], slurry whirling arm ring tester [32,46] and some other equipment [47–52], could be utilized. There are no standardized methods for testing and evaluation of erosion resistance. Although the methods based on ASTM G76-13 [53], originally designed only for dry erosion testing, and ASTM G73-10 [54] may be considered for slurry erosion only after their modifications, they have serious limitations related to the size and shape of the test samples. Thus, these methods cannot be employed for testing and evaluation of tubular samples where the inner surface of tubes is subjected to erosive impacts. Some testing methods simulate, in certain extents, specific application conditions. For example, the methods utilized special centrifugal devices where the erodents (dry sand or sand-containing slurries) impinge flat or curved samples or even the inner surface of the rings were designed and successfully employed for simulation of the processes in cyclones and hydrocyclones in mining and mineral processing and oil sand tailing [52]. However, there are no methods and related equipment simulating the processes occurring in tubing and pumping of artificial lifting systems in downhole oil production. As mentioned above, in these systems, the inner surface of tubular components is subjected to the simultaneous action of the rods and couplings or rotor (by reciprocation, rotation and vibration motions) and high velocity flows of slurries containing abrasive sand, particularly when sand particles occur between the rod and the inner surface of tubulars. These specific conditions are strongly distinguished from other known testing methods' conditions, and, therefore, the behavior of materials in these conditions and their erosion resistance could not be well extrapolated using the data obtained from other known testing methods. As a sequence, the appropriate testing apparatus and testing conditions simulating the erosion-abrasion-corrosion of tubular components should be developed for the right materials' evaluation, for prediction of the components' service and for proper materials' selection for this application.

Wear resistance of boronized steels was widely evaluated earlier, e.g., resistance to abrasion [24,55–58], adhesive wear (specifically rod wear in oil production) [59–61] and to combination with corrosion [61,62]. However, the data on behavior of boronized steels in erosion and erosion-corrosion conditions is very limited; only a few earlier publications [58,63–66] and our recent publication [52] can be listed. Although the behavior of different steels in erosion-corrosion environments was extensively studied (for example, in [10,32,36,46,49,67–69]), this data may be considered only in general for comparison since properties of bare steels and boronized steels are distinguished significantly. Moreover, the data related to behavior of boronized steels in the environments simulating downhole oil production situations and some processes of oil sand handling where the materials are subjected to the actions of slurries containing abrasive particles combining with rubbing by solid metallic components could not be found in the literature. The factors affecting performance of bare and boronized steels, related to the oil production conditions need to be analyzed. It is particularly related to the situations where sufficient volumes of corrosive slurries containing high percentages of hard abrasive particles, such as sand, at high velocities impinge the inner surface of tubular components, and a solid rod is also rubbing the tube inner surface at the same time. The equipment that is capable to test inner surface of tubes where slurry erosion is combined with abrasion motion of solid coupling inside the tube does not exist. The aim of the present work is to evaluate the erosion-abrasion-corrosion performance of the advanced iron boride coatings (protective layers) obtained through the thermal diffusion process in the simulating oil production conditions, even creating the "extreme" testing situations, and to compare the behavior

of this coating with "traditionally" employed bare steels. For example, in the majority of downhole oil production and oil sand transporting situations, the erosive flows (in fact, erosive-corrosive fluid flows) impinge the surface of tubing and related components at rather low angles (usually from below 5 to 15-20 degrees). In this work, in order to simulate the described service conditions, special testing equipment, particularly applicable for tubular components, was designed and fabricated. Thus, the testing method, testing device and application/testing conditions are totally different compared to other studies, including the equipment and testing processes recently considered [52]. In order to explore possible degradation mechanism during erosion and erosion-corrosion, the inner surface of the tubulars should be evaluated through the profilometry and surface examinations under microscope. These tests and studies have a significant importance for industry, and they will be utilized for selection and design of the downhole oil production components.

2. Materials and Methods

2.1. Starting Materials and Processing

Carbon steel grade J55 that is widely used for the tubing and piping systems in oil production was selected for our experimental works designated for erosion-abrasioncorrosion studies. The tubular samples had a length of 2 ft. (~610 mm) and a diameter of actual standard tubes of 3.5" OD (~90 mm OD and ~6.5 mm wall thickness). Additionally, flat samples with dimensions of \sim 12.5 \times 50 \times 12.5 mm³ (0.5" \times 2" \times 0.5") (i.e., width \times length \times thickness) were cut from bars made of carbon steel A36/44W, which is very similar by composition and physical properties to J55 steel. These flat samples were designated for abrasion testing. Chemical compositions of steels, according to MTRs, are presented in Table 1. The steel tubular and flat pieces were also used as the substrate for the boronizing.

Table 1. Chemical composition (wt %) of steel used in the study.

Steel	С	Si	Mn	Cu	Cr	Ni	Мо	Al	S + P	Ti/Sn
J55	0.26	0.18	1.30	0.21	0.15	0.13	0.012	0.03	0.017	0.008
A36/44	0.20	0.18	0.77	0.22	0.14	0.13	0.02	0.002	0.041	0.012
410SS	0.12	0.30	0.70	0.077	12.6	0.75	0.037	0.004	0.035	-
Fe—balance.										

The boronizing was conducted at Endurance Technologies Inc. (ETI) using the proprietary thermal diffusion technology based on CVD principles. In this process, steel components preliminary cleaned by sand blasting and then with acetone were immersed into the special powder mix contained the B-rich ingredient, activator and inert powder, which all together were placed into the retort, and then the materials were subjected to heat treatment for the formation of the iron boride protective layers on the steel surface. For steel tubing protection, the boronizing mix was packed into the tubes, i.e., the tubes worked as the retort. During the heat treatment, the B-rich gases occurred at high temperature reactions within the powder mix; the B species deposited onto the steel surface with diffusion of the B atoms to the steel. Due to high temperature interaction of B with Fe, iron borides formation, growth and consolidation occurred providing protection layer on the steel surface. The employed boronizing technology (the EndurAlloy[®] process) was described in detail elsewhere [24,25,61]. The boronizing mix (pack) composition and processing, e.g., temperature, heating-cooling profile and heat treatment conditions, were selected according to the features of the substrate material (carbon steel) and to required coating thickness (case depth) and demanding properties with respect to applicability for oil production. Thus, the boronizing process is conducted in the temperature range of 900–1000 °C for the predetermined time. The obtained flat samples had the coating for the entire surface, and the tubular components were protected only for the inner surface. Tubular samples of ~0.6 m (2-ft.) length required for slurry erosion-abrasion testing were cut from actual long tubes (bare or boronized).

Some other materials commonly employing for tubing and components in downhole oil production were also prepared for wear abrasion testing. They included bare steel grade 410 containing ~13 wt % Cr (Table 1) (stainless steel with composition and properties similar to L80-13Cr particularly used for tubing designated for corrosion-wear applications in oil and gas industry), carbon steel (A36/44W) with electroless nickel coating (ENC), carbon steel (A36/44W) with tungsten carbide-based composite coating (WC-Co-Cr). The samples with ENC coating were prepared according to the ETI procedure. The process included mechanical cleaning (sand blasting and washing with acetone) of the steel substrate, then activation of steel using special corrosive reagents with consequent washing and finally applying actual Ni-based coating onto the entire surface of the samples. The ENC formation occurs according to autocatalytic reaction of Ni-salt with P-containing reducing agents (e.g., metal hypophosphite and some others) and consequent Ni-P compound deposition. This type of coatings is widely described in the literature [70–72]. The ENC samples were heat treated at elevated temperature ranging of 350–450 °C for the nickel phosphide crystallization and consolidation in order to increase the coating's hardness and wear resistance. The WC-Co-Cr coating was applied onto the steel substrate by the thermal spray HVOF technology in Western Hard-Chrome (Edmonton, AB, Canada). This coating was applied only onto one surface designated for abrasion testing.

2.2. Abrasion and Slurry Erosion-Abrasion-Corrosion Testing Units and Testing Process

Abrasion testing was conducted according to ASTM G65-16 (Dry Sand Rubber Wheel Test) [73]. The apparatus consists of the rubber lined steel wheel rotating and touching the flat sample, which is tightly hold in the contact position with the weight. The abrasive medium (AFS 50–70 mesh sand) was continuously supplied between the fixed sample and the rotating wheel. According to the employed Procedure B designated for coatings' evaluation, the test duration was 2000 revolutions. The abrasion testing schematic is displayed in Figure 2. Upon the testing, the samples were weighed; the weight loss (in mg) and weight loss percentage were calculated, and, using the materials' density, the volume loss was also calculated.



Figure 2. Schematic of the dry sand rubber wheel abrasion test (ASTM G65-16).

The slurry erosion-abrasion-corrosion testing was conducted using the specially designed and fabricated ETI flow loop device (Figure 3) because, as noted above, there is no testing equipment that is capable to test the inner surface of the tubular pieces under the erosion-abrasion-corrosion conditions simulating processes in artificial lift systems and similar equipment. The total device dimensions are ~2.14 m (L) × 0.92 m (W) (~7 ft. × 3 ft.). This device provides the possibility to test, at the same time, up to three tubular sections mounted in the horizontal position at a small angle. The water-based slurry containing abrasive particles is circulated, using a diaphragm pump, through the test tubes; specifically, the slurry enters each tube simultaneously through the plastic hose system. Based on the device preliminary testing, it was found that the slurry line hoses need to be at least of 3/8'' (~9.5 mm) diameter because smaller hoses became clogged after short time and did not provide adequate slurry circulation. Simultaneously with the slurry flow, the installed steel pony rods create the rotating-oscillating motions inside the test tubes where the pony rod steel couplings (with 2" or ~50 mm diameter) constantly impact the inner surface of the test tubes. In this work, standard pony rods D78 SH T (~1.22 m or 4 ft. L) were used. In order to increase accuracy in wear of different tubes and couplings tested at the same time, the latter were additionally straightened and machined to exactly the same couplings' diameters minimizing their dimensional deviations. The selected load transfers the pressure to each pony rod, i.e., the contact of the pony rod's coupling with the tube inner surface can be managed. Before starting the erosion-abrasion-corrosion test, the sand containing slurry was prepared in the mixing tank using the three-blade impeller. Then the slurry was circulated through the whole system (from the tank through the tubing and returning back) for 30 min with no pony rods motion. When the slurry became homogenized, the test was started, turning on the pony rods motion. The fluid with abrasive particles penetrates between the tube inner surface and the pony rod coupling, and, as a result, the coupling's continual motion forced "embedding" the abrasive particles to the tube's inner surface. The following parameters can be set up, adjusted to the desired testing conditions and controlled during the test: the load applied onto the pony rods, pony rod rotation speed, slurry flow velocity and test duration. Additionally, the abrasive medium, e.g., type and particle size, solid concentration in the slurry, liquid phase composition can be varied creating more or less aggressive testing conditions. Thus, for the current testing, silica sand was selected as the abrasive medium, and water with machine oil addition was selected as a fluid. In order to increase a fluid's corrosivity, salts and/or some other additives (e.g., organic substances) can be added. As a result, the tubular samples become under continual action of rotating and impacting steel coupling combined with flow of the slurry with hard abrasive particles, i.e., under severe erosion-abrasion-corrosion conditions. The testing equipment units described in the literature generally consider only erosion flows of either dry powders or slurries containing hard particles, but with no involvement of mechanical actions of hard solid bodies, which creates a serious impact on the tubular materials' performance. The testing device design, testing method and selected conditions are based on the analysis of different artificial lift systems' designs and service conditions occurring in downhole oil production. In this testing, the slurry flow creates turbulence and low impingement angles, i.e., conditions occurring in production, which are the most critical for steel components [3,30–32,41].

In this work, the slurry compositions contained water with machine oil in a ratio of 75:25 (this ratio was selected according to industrial data from oil production). For some tests, starch (2 wt %) or sodium chloride NaCl salt (2 wt %) were also added. Silica sand was used as the abrasive medium; its content varied from 5 to 3 wt %, and the particle sizes were either between 325 to 200 mesh with major fraction of ~40–75 μ m (coarse sand) or below 325 mesh (finer grade sand). Specifically, when coarser sand was used for testing, a starch solution with increased binding capability was added to the slurry to minimize the sand settling. The applied loads varied from 250 lbs (~113.5 kg) to 50 lbs (~22.7 kg), the pony rods rotation velocity varied from 523 rpm to 130 rpm. After preliminary testing, the slurry flow velocity was selected as 3.5 L/min. This flow velocity provided a high slurry homogeneity without settling of sand (even the coarser grade) during the test cycle. The test duration varied depending on the test conditions.



Figure 3. General view and schematic of the ETI device for the erosion-abrasion-corrosion testing of tubular sections. (**Top**)—general view (the mixing tank is not shown), (**Bottom**)—tube with pony rod.

The intensive rubbing of the rods against tubes' inner surface combined with continuous erosive flows and associated friction issues and wear during rather long testing cycles can lead to a temperature increase in the slurry and to associated water evaporation. These, in turn, can lead to reduced slurry homogeneity, gradual sand settling, motor overheating due to higher slurry viscosity, and, hence, to inconsistent results. Thus, initial testing of the wear device demonstrated a significant temperature increase in the slurry after a couple of hours of the device running (e.g., from ~20 °C to 50–55 °C). To minimize this issue, a chiller was submerged into the slurry tank, that prevented the slurry heating (the slurry temperature did not rise above 25 °C). The cooling fan focused on the motor was also installed to prevent the motor overheating.

2.3. Materials Examination

The boronized coating thickness (case depth), structure and morphology were determined using an optical microscope MEIJI Techno IM7200 (Saitama, Japan), and the surface of the studied materials was examined with a scanning electron microscope (SEM) JEOL JSM-IT300LV (Tokyo, Japan). This was determined for the "witness" samples, which were prepared and passed through the whole processing cycle with the samples, which were tested and examined according to the present study. Small sections cut from tubular pieces designated for slurry-abrasion-corrosion testing were also examined. The determination of certain elements (e.g., boron) on the surface of selected samples was conducted by X-ray energy dispersive spectrum (EDS) analysis using JEOL JED-2300 DRY SDD EDS detector. The samples for the microscopic studies were cut, mounted, ground and polished according to the established ETI procedure using the Buehler equipment (Lake Bluff, IL, USA).

The coatings, as well as bare steel, were characterized by the Knoop hardness at 100 g indentation load (HK0.1) in accordance with ASTM E384-17 [74] (Clark CM 400AT Micro-Hardness Tester, Lenexa, KS, USA). The samples for micro-hardness testing were ground and polished using the Buehler equipment. Commonly 10 indentations were applied to each sample (according to ASTM E384-17 [74]), and average values were calculated. The Knoop micro-hardness determination method was selected, among other methods, based on earlier conducted ETI studies, due to specific configuration of the Knoop indentor and the iron boride coating thickness. In this method, the larger diagonal of the indentor and the replica is oriented parallel to the surface (alongside the coating), and with this orientation, the replica does not overlap the substrate, or the replicas from the iron boride sub-layers are also not overlapped at the 100 g indentation load. This provides the highest accuracy in micro-hardness determination compared to other methods.

After wear-corrosion testing, the samples (particularly tubular sections) were rinsed with water to remove the residual particles and then dried under hot air. Further, the samples were examined according to the particular needs.

The wall thickness of the tubular samples during and after the slurry erosion-abrasion testing was determined using the Ultrasonic Transducer (UT) tester T-MIKE ETM (StressTel, State College, PA, USA). For the reading, a thin layer of a gel was applied onto the tube outer surface corresponding to the point, where the wall thickness should be determined. Although this technique provides only approximate data, this information is important to monitor and compare the behavior of the testing tubes with different surface treatments and to prevent the situation when the tube perforation may occur.

In addition to visual inspection of the samples after dry abrasion or slurry erosionabrasion testing, their surfaces were examined by profilometry. Thus, the surface profile (wear scars) after sliding abrasion testing was conducted using a 3D optical profilometer Veeco WYKO NT 1100 (USA) with a vertical measurement range from 0.1 nm to 1 mm. The surface profile after slurry erosion-abrasion testing of tubular sections was examined using a special Creaform Go! Scan 20 3D Scanner (Bowen, PN, USA) for general configuration and a Zeta-20 3D Optical Profilometer for determination of microstructural features and roughness *R* from the University of Calgary. The scanner is capable to examine the scanning area of 143 mm × 108 mm with resolution of 100 μ m. The optical profilometer's capability in the Z-range is 200 μ m with Z-resolution of ~1 nm and in the XY-range is ~1 mm × 1 mm (without stitching) providing magnification of 20×. Because it is not possible to directly examine the worn surface (i.e., the grooves created during erosion-corrosion testing) of the tubular samples with the dimensions indicated above, the polymeric replicas of the occurred grooves were obtained. In order to make this replica, special silicone resin was mixed and poured inside the tube, and when the silicone rubber solidified, the replica was removed from the tube and used for the profile examination. The instrument displays the section in the middle of the samples and picks the protrusion points and draws lines vertically and measures the heights. The data from the Creaform scanner are saved as STL files transferring them into SolidWorks.

3. Results and Discussion

3.1. Protective Layer Characterization

The boronized surface on carbon steel had the "saw-tooth" structure consisting of two layers (Figure 4). The inner "lighter" layer (the zone covering the substrate) mostly consists of the Fe₂B phase, and the outer "darker" layer mostly consists of the FeB phase. Both layers have similar "saw-tooth" morphology despite their different crystalline lattice parameters, i.e., the FeB phase grows on the Fe₂B repeating the morphology of the Fe₂B phase. The formation of the double-layer architecture occurs simultaneously through a single heat treatment boronizing step. It is related to B atoms' diffusion to steel, interaction of B with Fe with formation of iron borides and their further consolidation. Specifically, during the process, B reacts with Fe₂B forming FeB, and the FeB layer grows over the Fe₂B layer. The features of the iron boride layers formation were described earlier [24,25]. No mechanical interface between the boride coating and the substrate, no delamination and micro-cracks between the phases and no Kirkendall porosity are observed despite the difference in crystalline structures and values of coefficients of thermal expansion between the two iron boride phases, i.e., good bonding between the "Fe-B layers" is reached due to optimization of the pack composition and process parameters. The total case depths were $\sim 200 \ \mu m \ (\sim 0.008'')$ for the flat samples designated for dry sand abrasion test and in the range of ~275 μ m (~0.011")–325 μ m (~0.013") for tubular samples for slurry erosionabrasion. With regards to case depth determination, it was measured from the top of the coating surface to the middle of the "teeth". The columnar grain structure adherent to both layers reinforces the coating in general. The thermal diffusion process provides rather uniform coating for the entire treated surface with no delamination issues. It is related to the B-rich vapor formation (using CVD principles) uniformly within the "chamber" surrounding the steel body completely. The obtained structure fully correlates with earlier reported data [20-25,61,62].



Figure 4. Microstructure of the boronized steel (typical), magnification 200×.

The micro-hardness of the iron boride coating is significantly higher than uncoated carbon steels. The inner zone of the coatings, which mostly consists of the Fe₂B phase, has slightly lower hardness values than the outer area of the coatings mostly consisted of the FeB phase (HK0.1 1500–1650 and 1700–1850 kgf/mm², respectively). In comparison, the carbon steel substrate has micro-hardness values of only 170–190 HK0.1, i.e., about 8–10 times lower than the iron boride coating. The obtained results correlate well with other published data related to micro-hardness of the boronized coatings [24,25,59–62].

The ENC samples had the coating thickness of ~65–75 μ m uniformly adhered to the carbon steel substrate (Figure 5). The coating's heat treatment provided precipitation and crystallization of nickel phosphide Ni₃P with expectable increased micro-hardness up to ~515 kgf/mm² (HK0.1). The thickness of the WC-Co-Cr composite coating was a ~175 μ m (Figure 5). Its micro-hardness HK0.1 was ~1800–1900 kgf/mm² that is comparable with the iron borides' micro-hardness; however, these data are related to the situations when the sharp diamond indenter was applied to the WC grains. When the indenter contacted the Co-based matrix, micro-hardness values were significantly lower. Because of a small amount of the metallic bonding phase in this coating, it was not possible to determine micro-hardness of the individual phases.



Figure 5. Microstructure of ENC (left) and WC-based coating (right) on carbon steel, magnification 200×.

3.2. Dry Sand Abrasion Testing Results

The results of abrasion testing conducted according to ASTM G65-16 [73] are presented in Figures 6 and 7. This test was selected as a preliminary test to demonstrate the difference between the materials used for downhole oil production being in contact with abrasive particles and therefore to make a quick materials "screening" for the further slurry erosionabrasion testing. As can be seen from the weight and volume loss diagrams (Figure 6), carbon steel and stainless steel samples, as well as hardened ENC, demonstrated identical performance. Thus, weight and volume loss of these materials after 2000 revolutions became 0.47–0.5% and (115–120) \times 10⁻³ cm³, respectively, despite their different chemistry and hardness. These indicate that significantly more expensive stainless steel 410SS, which is identical to L80-13Cr steel designated for tubing in oil production for special needs when components should serve in contact with abrasive and corrosive environments, and ENC coating also with elevated hardness and corrosion resistance demonstrated the same performance as low-cost carbon steel. The occurred wear scars (grooves) with significant depths are well observed in these samples (Figures 7 and 8). The deep groove for the ENC sample indicates that this coating was completely removed from the surface. In fact, this test result demonstrates that the use of more expensive stainless steel and ENC is not reasonable for the abrasion related application conditions. In contrast, the samples of boronized carbon steel and steel with the thermal spray WC-Co composite protective

layer experienced significantly lower wear loss, which was only <0.05 wt % (weight loss) and ~5 × 10^{-3} cm³ (volume loss). The wear scars for these materials are not deep; they look like the surface polishing. Although both these materials demonstrated similar and low wear losses in dry erosion conditions, the thermal spray technology employed for the WC-Co-Cr coating is not applicable for protection of the inner surface of long tubing required for downhole oil production (there is no information, so far, about this possibility in industry), while boronizing technology (the EndurAlloy[®] process) is well suitable for long tubular products' protection.



Figure 6. Abrasion testing results conducted according to ASTM G65-16 [73], Procedure B (2000 revolutions). (**Left**)—weight loss, (%); (**Right**)—volume loss (cm³).



Figure 7. Appearance of the samples after the ASTM G65—16 abrasion test. From left to right: carbon steel (CS), 410 SS = L80-13Cr, ENC-CS, WC-Co-CS, B-CS (EndurAlloy[®]).









mm

Figure 8. Cont.



Figure 8. Comparison of the profilometer data for bare carbon steel (**top-a**) and boronized carbon steel (**bottom-b**) after abrasion ASTM G65 testing. The wear scar depth is ~600 μ m for bare carbon steel and ~30–35 μ m for boronized steel.

One of the key factors defining the difference in abrasion test results for the studied materials is their hardness. Thus, the micro-hardness data HK0.1 of bare steels, e.g., carbon steel and stainless steel 410, are rather similar (~200 and 250 kgf/mm²). ENC has elevated micro-hardness (~500 kgf/mm²), but the coating thickness is rather small. However, all these materials are not as hard as silica sand (according to the Moh's scale), and, because of this, they are destroyed quite rapidly under abrasion with sand. As opposed to these materials, boronized steel's micro-hardness HK0.1 is significantly greater (reaching ~1850 kgf/mm²) where the well consolidated iron boride protective layers have a total thickness of ~200 μ m. As a result, this iron boride coating provides significantly better protection of steel than ENC. The WC-based coating also has a high hardness level. The structure where the WC phase with high hardness bonded by the metallic matrix having lower hardness but higher toughness provided a high level of abrasion resistance. This material should be well suitable for erosion related applications as well, especially due to the presence of a tough bonding phase.

The comparison of the materials' wear scars examined by optical profilometry confirms that boronized steel significantly outperformed bare carbon steel (Figure 8). According to the calculation from the profilometry, the wear scar depth and volume loss for bare steel are ~600 μ m and ~130 mm³, respectively, while these data for boronized steel are ~30–35 μ m and ~8 mm³. It should be noted that the wear scar of the iron boride coating is not very smooth as the steels' wear scars. It is related to the micro-cracking wear mechanism for hard but brittle iron borides. Because the wear scar for boronized steel is significantly smaller than the original case depth (~200 μ m), it is easily concluded that the iron boride layer remained after the abrasion test, i.e., only a minor portion of the top FeB sub-layer was removed, and the Fe₂B layer was not affected.

The materials surfaces, according to SEM examination (Figure 9), became significantly smoother after the testing that associates with their abrasion wear. However, the materials' wear occurred in different extents. Comparison with the ENC samples confirms complete removal of the ENC (the surface after testing became identical to the surface of the bare carbon steel sample). In the case of the composite WC-Co coating, it is assumed that the Co-rich rather soft matrix is worn out first by abrasion with hard silica particles. The presence of observed caverns on the surface of this sample, may be explained by the WC grains pull-out from softer Co-based matrix due to its wear and weakened bonding between the grains and the matrix. This is in a good correlation with other data [34,48,75]. For the boronized samples, mostly surface asperities were removed after 2000 revolutions; however, no cracks, large caverns and other defects were observed.







307

Figure 9. Cont.









Figure 9. Surface morphology of the samples before (**left column**) and after (**right column**) abrasion testing (ASTM G65), $500 \times$ magnification. Samples from top to bottom: carbon steel (**a**), stainless steel 410SS (**b**), ENC on carbon steel (**c**), boronized carbon steel (**d**), WC-Co on carbon steel (**e**).

3.3. Slurry Erosion-Abrasion-Corrosion Testing Results

According to the dry sand rubber wheel abrasion test (ASTM G65) results, considered as preliminary, it was decided to compare the behavior of bare carbon steel and boronized steel tubular sections in slurry erosion conditions. Because carbon steel, stainless steel and steel with ENC demonstrated identical performance under dry sand abrasion conditions, it may be expected that these materials will also have the similar (comparable) performance under slurry erosion-abrasion testing. It should be taken into consideration that carbon steel and stainless steel 410 have similar micro-hardnesses, which are also similar to the steel coupling, and not as hard as silica sand. Besides, in the slurry erosion testing conducted in this work, the fluid's flow will create sliding erosion-abrasion with impingement angles, which are not favorable for steels [32,37,41,42,58]. Additionally, the data presented in the earlier publication [61] demonstrated identical low performance of bare steel and ENC but high performance of boronized carbon steel when the samples were subjected to the combined action of sliding steel rod and corrosive fluid contained silica sand. The inclusion of the tube with the WC-Co-Cr coating should be very attractive for this testing, but, unfortunately, it was impossible to apply this coating by thermal spray and similar technologies onto the inner surface of tubes with the selected and other dimensions. So, the slurry erosion testing was conducted with one carbon steel and one or two boronized steel tubular sections.

The slurry erosion-abrasion tests were conducted, as mentioned above, varied the testing conditions, e.g., loads applied onto the pony rods, rotation frequency, slurry composition and test duration. It is obvious that, at more severe conditions, the tubes' wear occurred in greater extents regardless the material, e.g., bare steel and boronized steel. As expected, the wear occurred mostly in the areas where pony rod coupling (cylindrical area and "shoulders") was in contact with the tubing inner surface, particularly when silica sand containing in slurry penetrated between the tube and the pony rod (see Figures 3 and 10). The occurred wear scars had specific shapes of an uneven groove with sizes corresponding to the cylindrical part of a pony rod and two small grooves corresponding to the pony rod "shoulders". The wear scars for the bare tubes were significantly deeper or the remaining walls were significantly smaller than for boronized tubes after all tests (Table 2). Thus, for the most severe conditions significantly exceeded actual service conditions), the test was conducted for 93 h, i.e., until the perforation of the bare steel tubes (Figure 10). It was clearly observed that high loads and rotation speeds created intensive bouncing and vibration

~113.5

resulted in continuous "micro-impacts" on the inner surfaces of tubes. The boronized tubular section tested at the same conditions and time lost only up to 15% in depth, i.e., the iron boride protective layer was completely lost under these conditions. However, the boronized protective layer significantly delayed erosion-related wear of the tube. Then it was decided to conduct testing in less severe conditions better simulating service conditions. In the softest test conditions, which had significantly better correspondence to the service conditions in the artificial lift systems, the wear losses for bare steel tubes reached ~18% (tube wall thickness loss) after 19 h, while the wall loss for the boronized tube was negligible (significantly less than 1%) after the same test duration and twice as long test duration. In this condition, the iron boride layer remained with only minor losses.



Figure 10. Appearance of the tubular sections after the wear test at the most severe conditions (see Table 2). (**Left**)—general view; (**Right**)—boronized and bare steel tubes (the bare steel tube has a perforation, while both boronized tubes did not).

Test Conditions	Bare Ste Wall Thick	el Tube mess Loss	Boronized Steel Tube (EndurAlloy [®]) Wall Thickness Loss		
	mm	%	mm	%	
kg, 523 rpm, 3.5 L/min					

Table 2. Slurry erosion test results (tube wall thickness loss determined with UT instrument).

water-oil-starch slurry with 5% sand (325–200 mesh)				
93 h	3.8-6.81	up to 100	Up to 1.05	up to 15
~68 kg, 265 rpm, 3.5 L/min water-oil-slurry with 5% sand (325–200 mesh)				
48 h	2.33–3.51	33–50	0.44–0.64	~8
~22.7 kg, 130 rpm, 3.5 L/min water-oil slurry with sand 3% (325 mesh)				
19 h	0.46-1.22	7–18	0.01	~0.15
38 h	1.06-2.52	15.5–37	0.02	~0.3
~22.7 kg, 130 rpm, 3.5 L/min water-oil-1% NaCl slurry with sand 5% (325 mesh)				
19 h	0.6–1.3	8.5–19	0.01	~0.15

The presence of NaCl salt in the slurry did not make a significant impact on the wear loss of the tubular sections. Although it is well known that this salt increases corrosion impact on steels, the severe conditions related to erosion-abrasion are, probably, created the greatest effect on tubes' destruction. It seems that the presence of oil in the slurry also reduced salt penetration into the surface defects in tubes and diminished the corrosion effect. More studies related to different slurry compositions and wear test conditions affecting wear-corrosion are further required.

According to the conducted test results, it seems that testing in rather mild conditions (e.g., at relatively low mechanical loads and rotations speeds) could be the most appropriate as these conditions have better simulations to application situations, and they allow more options to vary slurry compositions and time in the testing.

Some results obtained for the polymeric replicas made from boronized and bare carbon steel tubular samples using the Creaform Scanner and optical profilometer Zeta-20 are presented in Figures 11–14. The grooves' depths after wear testing are indicated in the brackets. The data for the wear scar depths obtained with the UT tester and Creaform Scan have some difference, which is related to the accuracy in these instruments and the testing and instruments' features. Additionally, the differences in data may be explained that these measurements were conducted not at the same points. However, these data clearly demonstrate that boronized steel samples had significantly shallow grooves (smaller wear scars' depths) and a smaller range of the roughness inside the wear scars indicating significantly higher performance of boronized steel over bare steels. The observed scars with high roughness' ranges within the same groove indicates a complexity of the materials' destruction process at the conducted testing and selected conditions.

The SEM images of surface morphology for bare carbon steel and for boronized carbon steel tubing after slurry erosion-abrasion testing are demonstrated in Figure 15. The wear scars (grooves) of bare J55 steel tubes, which were in continual contacts with a steel pony rod coupling and a slurry-containing sand, had uneven surfaces where rather shallow smoother "valleys" and significantly deeper rougher "valleys" could be observed (the images are related to 19 h of testing in rather mild conditions). The significantly rougher and deeper scars (grooves) with numerous caverns and pits were observed after longer testing (48 h) in more severe conditions. In these testing conditions, the steel texture became less dense. The EDS analysis of this surface revealed, as expected, the presence of Fe and O that indicates oxidation and corrosion of steel. In some areas, notable amounts of Si were detected that is related to the embedding of silica sand grains or their fractures to the steel surface. The steel tube surface, which was subjected only by the slurry and was not in contact with a coupling, also had many caverns and pits occurred due to severe erosive flows with turbulence. When NaCl was added to the slurry, small amounts of Cl and Na were also detected on the steel surface by EDS analysis although the accuracy in Cl detection was not high. It may be speculated that some residues of salt remained in caverns on steel surface. In contrast, boronized steel tubing had significantly shallow wear scars with significantly smoother surface after the 19-hr. testing. Even after very severe testing conditions (93 h), which were significantly stronger than service conditions, the remained iron boride layer may be observed in some areas. The EDS elemental analysis of the boronized surface of the tube exposed for 93 h allowed detecting the presence of B, as well as Fe and O. The iron boride oxidation in the wear scar was expected, similarly to the earlier wear testing [52,61], i.e., an (Fe,B)_xO_y film was occurred. The B contents varied from ~8 wt % to ~2 wt % in different areas that indicates uneven wear of the iron boride layers. When the testing was conducted in the conditions 4 (with addition of NaCl), no presence of Cl was detected for the boronized steel by EDS. Moreover, the presence of O and Cl was not detected by EDS in the steel substrate under the boride layer that indicates a high level of protection through boronizing. These results correspond well to our previous study [52]. Because the sufficient wear was detected by the profilometry examination, it could be assumed that, after testing for 93 h under severe slurry erosion-abrasion-corrosion conditions (see Table 2), only oxidized F_2B layer remained in certain areas while it was completely gone in some zones. It is assumed that, at these severe conditions, the cracked and peeled-off iron boride layers' fragments were removed by high velocity slurry flows. The boronized surface that was not in contact in the steel coupling had small pits and even occasional surface micro-cracks occurred due to slurry erosive actions. The micro-



cracks were not detected in the groove areas; probably, the cumulative action of a rotating coupling and slurry flows worn the material surface where the micro-cracks became not well observed.

Figure 11. Creaform and Zeta 20 scans—bare carbon steel tube tested at condition 2 (see Table 2). (**a**) Replica; (**b**) Roughness data; (**c**) Surface scan data.

Point 5

Point 4





Figure 12. Creaform and Zeta 20 scans—boronized carbon steel tube tested at condition 1 (see Table 2). (a) Replica; (b) Roughness data; (c) Surface scan data.

Point 3



Figure 13. Creaform and Zeta 20 scans—bare carbon steel tube tested at condition 3 (see Table 2), 19 h. (a) Replica; (b) Roughness data; (c) Surface scan data.



(b)



X-axis (µm)



Figure 14. Creaform and Zeta 20 scans—boronized carbon steel tube tested at condition 2 (see Table 2). (a) Replica; (b) Roughness data; (c) Surface scan data.

(a) (b)







(e)

Figure 15. Surface morphology of steel J55 tube (**left column**) and boronized steel tube (**right column**) after slurry erosionabrasion-corrosion testing. (**a**,**b**)—wear scars (groove)—condition 3, after 19 h (SEM, magnifications $200 \times$ and $1000 \times$). Rows (**c**,**d**)—wear scars (groove)—condition 2 after 48 h. (J55) and condition 1 after 93 h. (boronized steel) (SEM, magnifications $200 \times$ and $1000 \times$). (**e**)—out of wear groove—condition 2 after 48 h (J55) and condition 1 after 93 h (boronized steel) (SEM, magnification $1000 \times$).

> The images obtained under optical microscope from the cross-sections of materials after 19 h testing (conditions 3-4 Table 2) indicated insignificant wear of boronized steel and surface destruction of bare carbon steel (Figure 16). Thus, the boronized tube surface that was in contact with a pony rod coupling and a slurry contained silica sand lost only very insignificant portion of the iron boride layer; the remained case depth was ~300–340 μm. Some cracking within the iron boride protective layer parallel to the surface of the coating (or steel substrate) occurred, likely due to continual motion (particularly, impacts) of the pony rod coupling. The tube area, which was subjected only to the slurry with abrasive particles but not subjected to the pony rod coupling, had a case depth of 340–345 µm. The "wavier" surface could be observed; however, no significant crack propagation was detected. In contrast, the bare steel tube had a deep groove after 19 h of testing. Because of the size limitations of the optical microscope, the full groove could not be demonstrated. However, it can clearly be seen that the steel surface was destroyed under the slurry erosionabrasion where large steel flakes with thicknesses of more than 30 μ m were removed. The boronized tubes that were tested at significantly more severe conditions (~90 h duration) lost the protective iron boride layer in the area contacted with a pony rod coupling and

"shoulders", as was mentioned above; however, the iron boride layer still remained in the areas close to the deepest groove zone (Figure 17). This image shows the reduction of the case depth and the crack propagation through the boride layer. As opposed to this area, the boronized surface remained practically intact in the places where the pony rod coupling did not touch the tube surface; the thickness of the protective layer remained practically without changes (over $300 \ \mu\text{m}$), and no cracks were observed (Figure 17). The iron boride layer micro-hardness remained on the original level after testing; its HK0.1 was in the range of $1600-1800 \ \text{kgf/mm}^2$. Only the micro-hardness values had more discrepancies for certain indentations. This high level of micro-hardness indicates the high integrity of the iron boride coating structure, with only insignificant micro-cracks formation and propagation at selected testing conditions.



Figure 16. Surface structure for tubes J55 steel (**top row**) and boronized J55 steel (**bottom row**) after slurry (wirh NaCl) erosion-abrasion-corrosion testing for 19 h. (**Left column**)—area from the wear groove (affected by combined action of pony rod coupling and slurry containing silica sand). (**Right column**)—area out of the visible wear groove affected only by slurry containing silica sand.



Figure 17. Surface structure for boronized J55 steel tube after slurry erosion-abrasion-corrosion testing for ~90 h. (**Left**)—area from the side of the wear groove "partially" affected by combined action of pony rod coupling and slurry containing silica sand. (**Right**)—area out of the visible wear groove affected only by slurry containing silica sand.

Due to the significant difference in structure and properties of the bare steels and the iron boride protective layers (coatings), the damage mechanisms and degradation during mechanical and corrosive actions, as well as the wear scar depths and materials removal volumes should be different. Thus, steels are not hard but ductile, while Fe–B coatings have significantly higher hardness (higher than steels and higher than the processing materials, like sand and others) but lower ductility and toughness. The chemical nature and structural bonds between the major elements in the considering materials (Fe with additives for steels or Fe–B for the crystalline iron boride coating) are also totally different. Because of this, the mechanisms of formation of micro- and macro-defects, and their nature and propagation, which are responsible for the material removal, will strongly affect their performance at the continual slurry erosion-abrasion and erosion-abrasion-corrosion.

The wear of bare steels in the considered testing and application conditions occurs through the plastic deformation and micro-ploughing mechanisms, as indicated in many literature sources [30,32,34,35,37,68]. The slurry erosive flows quickly remove the occurring particles of steel and oxidized steel (mostly magnetite Fe₃O₄ and iron hydroxide scale). The observation of the particles remained inside the steel tube after the wear tests completion revealed that these particles have mostly a flake shape. The fluid easily penetrates into the occurring surface defects, further facilitating corrosion and destruction of the steel. The combined action of hard steel coupling and erosive-corrosive flows combining hard abrasive particles significantly accelerates and increases the wear; thus, the coupling pressurizes the silica particles, as well as the worn steel and iron oxide flakes, and forces them to further penetrate into the occurred macro- and micro-defects in steel. This creates additional friction between the tube and the coupling further increasing wear. At the same time, a fluid washes out iron oxide and iron hydroxide flakes and worn steel particles creating "fresh" steel surface for further contacts with erosive-corrosive flows and pony rod coupling.

As opposed to the wear-corrosion mechanism related to bare carbon steel tubing, the surface of boronized steel with high hardness but lower toughness does not experience wear through micro-ploughing. The wear mechanism, particularly in slurry erosion conditions considered in this work, occurs through the micro-crack formation and propagation. The micro-cracking occurs not only due to erosion flows of a slurry containing abrasive silica sand and due to rubbing by a hard pony rod coupling, but also due to vibration, bouncing and "impacts" created by the pony rod. As mentioned above, these "impacts", which are specific issues for this testing equipment, are the particular concern at high

loads and rotation speeds. This point is critical for the iron boride protective (coating) layers with reduced toughness. However, since iron borides are significantly harder than processing silica sand and steel using in coupling, the micro-cracking does not occur very fast. Despite the created turbulence, the small impingement angle in this application does not create a direct (90-degree angle) impact of the processing silica particles, and sliding erosion coupled with sliding abrasion are more favorable for integrity of the hard iron boride layer, in contrast to the behavior of softer but ductile steels. As mentioned above, the steel coupling pressurized the silica particles when they penetrate between the tube surface and the coupling, which increases the silica particles' indentations and the wedging effect, especially when high loads to the pony rods are applied. However, the wedging effect and micro-crack propagation in the iron boride layer are delayed due to its high hardness and specific double-layer coating architecture. Thus, the hard but brittle top FeB layer, being in contact with silica particles and steel coupling with significantly lower hardness, starts experiencing micro-cracking, but this FeB layer is supported by the bottom layer Fe₂B with higher toughness (fracture toughnesses of the FeB and Fe₂B are approximately 1.5 and 5.4 MPa.m^{1/2}, respectively [65,76]), which significantly delays micro-crack evolution and propagation. In this case, micro-cracks bridging and deflection could occur, as discussed earlier [52]. Furthermore, the steel substrate with significantly higher ductility and toughness compared to the borides supports the coating serving like a "cushion". Only when the micro-cracks grow and transfer to macro-cracks, which leads to gradual chipping of the iron boride layer and to partial pulling out of this layer fragments, the erosion wear accelerates. The saw-tooth structure of the iron boride layers promotes the stress relaxation, since the "teeth" provide the steel substrate reinforcement, and it should delay the chipping of the protective layer. Corrosion of the iron boride layers is insignificant, as their chemical inertness is defined by strong Fe–B bonds and by a high level of crystalline lattice energy, as shown above, as well as by the insignificant presence of internal defects in the coating structure. As a sequence, the formation of soft iron oxide and iron hydroxide scale with poor protection is not expected, but the oxidized (FeB)_xO_y film inhibits further deep oxidation of the FeB layer promoting material protection.

The erosion wear mechanism for the boronized steel, including crack initiation and propagation, and its comparison to bare steel erosion were considered in detail in our previous work [52], although the testing and application conditions in the previous and present works are totally different. The earlier described micro-crack propagation and erosion wear features can be transferred, in a high extent, to the wear mechanism related to the present study, although the synergistic action of slurry erosion, abrasion, and continual "impacts" and vibration, especially at high pressures to pony rods, accelerates wedging of the erodent in the occurred micro-cracks, crack propagation and, as a result, wear. The presence of NaCl in the slurry does not create significant effect on destruction of boronized steel compared to "mechanically"-induced wear. The positive features related to enhanced resistance of the boronized steels to slurry erosion-abrasion and erosion-abrasion-corrosion can be pointed as:

- High hardness of iron borides (significantly higher than processing particles and steel);
- Double-layer coating architecture consisting of both well-consolidated microcrystallined hard sub-layers, e.g., the Fe₂B sub-layer covering the steel substrate and the top FeB sub-layer that is harder than Fe₂B and which is in contact with processing environments;
- Saw-tooth structure of both iron boride layers and their substantial thickness (a total thickness of at least ~200 µm or greater) promote the coating integrity owing to reduced micro-crack propagation, which is related to micro-cracks' bridging and deflection at the testing/application conditions;
- Micro-cracks healing, which also delays their propagation, due to oxidation of the iron boride in the micro-crack surface in contact with water-based slurries; the oxidized iron boride nano-size layer on the surface of iron borides serves as a "tribo-film" reducing friction between the counterparts;

- Diffusion induced bonding between the iron boride layers and the steel substrate with no delamination issues, as well as the softer and significantly more ductile steel substrate, supporting a hard iron boride layer (serving like a "cushion" and a stressrelaxator), also reduce the crack propagation positively affecting wear resistance;
- High chemical inertness of iron borides defined by short and strong covalent Fe–B bonds and high the crystalline lattice energy (enthalpy) and their micro-crystalline structure.

Because, in the case of boronized steel, the components' performance is defined by the structure and properties of the protective iron boride layers, different steel options can be successfully utilized for industrial equipment. In this case, the wear mechanism and the protection level will be identical regardless the steel grade. The thermal diffusion process can be employed for boronizing of steel components with various sizes and shapes, e.g., for pup joints, tubing and casing (with high L/D ratios) up to 10 m length, components in artificial lift systems, centralizers, elbows, cyclones and many others. All these lead to the possibility of using low cost carbon steels and low-alloy steels for production equipment in severe sliding erosion, abrasion and corrosion environments, including for downhole oil production, if these steels are boronized. The boronized steel tubing and casing with inner surface protection can be effectively employed in the most critical operation conditions.

4. Conclusions

Wear-corrosion resistance of tubular components made of bare carbon steel and steel with advanced iron boride coating was compared under slurry erosion-abrasion-corrosion testing conditions simulating downhole oil production, particularly in artificial lift systems.

For these studies, a special wear flow loop device was designed and fabricated, and the testing conditions were optimized. In this new device and testing method, a slurry is circulated through tubes, while a rotating pony is simultaneously rubbing the tube inner surface. The slurries may be prepared with various corrosivities and with different contents of abrasive particles (e.g., silica sand). It allows testing of three tubular sections at the same time.

Boronized carbon steel significantly outperformed bare steels in the considered testing conditions (dry abrasion and slurry erosion-abrasion-corrosion) providing delay of destruction and wear of tubular components. It was demonstrated that, in the dry abrasion testing conditions, bare steels (e.g., carbon steel and more expensive stainless steel) perform identically since their hardness differentiates insignificantly and the steels are not as hard as processing sand. The enhanced performance of the boronized steel tubing is defined by a combination of a double-layer coating architecture consisting of iron borides with high hardness (~8–10 times greater than bare steels) and chemical inertness, specific "saw tooth" well-consolidated structure of the protective layers with sufficient thicknesses, diffusion induced bonding between the iron boride layers and the steel substrate, and the support of hard iron boride layers with ductile steel. These factors, all together, afford the reducing of micro-crack propagation during harsh service conditions. The obtained slurry erosion-abrasion test results obtained for boronized and bare steels correspond well to the dry sand abrasion test results.

The proposed thermal diffusion process (EndurAlloy[®]) is well applicable to protect the inner surface of long tubing and casing (with a high L/D ratio) and various components with intricate shapes, and it can be used for boronizing of components in artificial lift systems and for other oil and gas production equipment.

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