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COMPARATIVE STUDY OF THE WEAR OF SPECIMENS LUBRICATED WITH MINERAL OIL OR BIOFLUIDS*

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Abstract

This study compares the wear resistance of specimens lubricated with biofluids or mineral oil. Tribological studies were performed by ball-cratering test. In these tests were used as lubricants epoxidized soybean oil, castor oil and mineral oil. The specimens used were untreated and nitrided AISI 4140, while a steel ball AISI 52100 was used as a counterbody. In order to analyze the results obtained, the worn volume was calculated and the characterization of the crater, after the test, was made using optical microscopy and SEM. Wettability was also measured for the different surface condition and lubricants. As expected both specimens showed a smaller volume worn when lubricated with mineral oil due to inherent properties of this type of lubricant. Among the vegetable oils studied, the castor oil showed a better performance in the lubrication of the specimens untreated while on the nitrided specimen have shown less wear when lubricated with epoxidized soybean oil, indicating a difference in the efficiency of oils, as lubricants, depending on the surface conditions of the mechanical components.

Keywords: Tribology; Wear; Nitrided; Biofluids.

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1 INTRODUCTION

Component failures (bearings, gears, valves, hydraulic pistons, etc.) and stops during production can lead to significant financial losses. Two factors are essential for proper preservation of these components: the wear resistance of the parts used and lubrication. The growing of the environment concerns has provoked technical solutions which causes minimal environmental damage.
Among them nitriding is a process used for treating metal surfaces, increasing the hardness, corrosion, fatigue and wear resistance and reducing friction [1]. Mashreghi et al. [2] investigated nitrided tool steels and they found significant increase both in wear resistance as greater corrosion resistance in the nitrided components.
The typical microstructure of nitrided surface is formed by compound layer (white layer) and diffusion zone. The compound layer consists of intermixed iron nitrides, γ' Fe4N and ε Fe2-3N phases, while the diffusion zone consists of a nitrogen rich solid solution zone with fine and coherent nitride precipitates [3] According to Pye [4] the phase γ 'is more ductile and impact resistant while the ε phase exhibits good wear resistance.
Biofluids have been extensively studied in recent years with the objective of increase its range of application and so gradually to replace mineral oils which are derived from non-renewable sources cause enormous damage to the environment. These vegetable oils are extracted mostly from plants and are mainly triacylglycerols. Beyond the environmental issues (biodegradable, less toxic and renewable) other factors cause the biofluids appear as a good alternative for use as lubricants: excellent lubricity, high viscosity index, high flash point [5]. However the replacement of mineral oils by biofluids still presents some problems, for example high cost of production. Moreover, vegetable oils in their natural form have low oxidative stability due to the unsaturation bonds present in the chain, which limits its use as a lubricant [6].
This work has the main objective to compare wear resistance between specimens untreated and nitrided when lubricated with mineral oils and biofluids using vegetable oils as base. Tribological tests were performed using the ball cratering method and the volume worn in each specimen was calculated. Worn area analysis were made using optical microscopy and SEM

2 MATERIALAND METHODS

AISI 4140 steel were used in the samples which composition is described in Table 1. Ball bearings (AISI 52100), with 1 inch diameter, were used how counterbody.

Table 1. AISI 4140 steel chemical composition (wt.%).

C	Mn	Si	P	S	Cr	Ni	Mo
0,39	0,84	0,33	0,02	0,01	0,94	0,03	0,18

Before nitriding process the specimens were quenched and tempered. Quenching were performed using austenitizing temperature of 850 ° C for 30 minutes and quenched in oil. Tempering temperature was 460 °C for 2 hours. After these processes surface hardness achieved 33 HRC. Microstructures of AISI 4140 steel before and after heat treatment are shown in Figure 1a and 1b. Untreated sample (fig 1a) presents a microstructure consisting of ferrite (white area) and perlite, while in the quenched and tempered condition (figure 1b) microstructure is compound of tempered martensite as waited.

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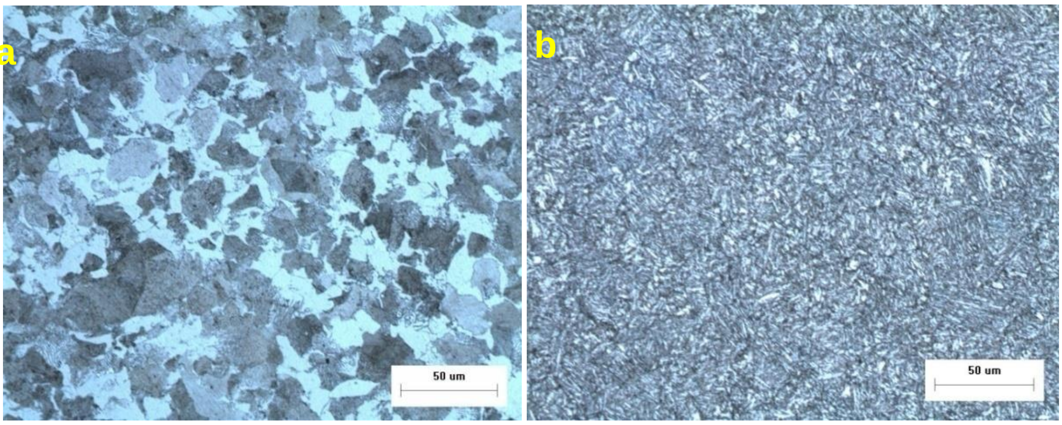

Figure 1. Microstructure untreated AISI 4140 (a),quenched and tempered AISI 4140 (b).

The ion nitriding process was performed according to the parameters described in Table 2.

Table 2. Parameters used in the nitriding process.

Gas composition		Temperature [ºC]	Time [h]
N_2[l/min]	H_2[l/min]		
3,12	0,78	520	12

Metallographic analysis were performed before and after nitriding process as well as XRD analysis using equipment Shimadzu LabX XRD model - 6000 with Cu Ka radiation (λ = 1.5418 Ǻ).

The lubricating oils used in this study were: mineral oil without additives (MO), castor oil (CO)and epoxidized soybean oil (ESO) both also without additives. The properties of the oils used are described in Table 3.

Table 3 – Lubricants oils properties.

	Mineral oil	Castor oil	Epoxidized soybean oil
Viscosity (40ºC) (cSt)	143,71	250,17	162,25
Viscosity (100ºC) (cSt)	10,01	19,44	19,85
Density 20/4ºC	0,916	0,955	0,960 (60 ºC)
Acid index (mg KOH/g)	1,65	2,80	1,48
Saponification index (mg KOH/g)	0,0	175,86	177,95
Iodine index	-	52,8	0,41
Peroxide index	0,4	5,0	32,08
Flash point ºC	200	310	306
Pour pointºC	-	-	53

The high peroxide index of epoxidized soybean oil (32,08), indicates that the epoxidation process of this oil was made by reaction of the oil with peroxide as epoxidizing agent, another explanation for the high value of the peroxide can be

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explained by the presence of peroxide resulting from the reaction with stoichiometry calculated for the excess not removed in the neutralization process.

The low iodine index (0.41 g I2/100g) indicates the efficiency of the epoxidation process used in soybean oil.

Measurements of contact angles were analyzed by dripping. A goniometer used was Krüss OF 100 with integrated camera. This device has a syringe storing the liquid, which is then deposited on the surface under analysis. The wettability tests were performed measuring the contact angle of various liquids on surfaces analyzed. These measurements were performed at 25 °C and the values of contact angles were stored on the computer through a camera with a graphical interface.

The hardness measurements were carried out on the surface and on the cross-section of samples using PANTEC MV 2000A microhardness tester at a load of 50 gf in surface and 10gf in cross section, a loading time of 10 s. In the cross-section, five hardness tests were performed for each depth of the samples, and the average values were reported.

The wear tests were performed using the method ball-cratering with the addition of lubricant in the contact region. A schematic of the machine test is shown in Figure 2. The specimens used were 2 cm height and 3 cm wide and 1 cm thickness and spheres (counterbody) 1-inch diameter (Figure 3). The rotation speed and contact pressure were 600 rpm and 4.2 GPa, respectively.Total time of each test was 2 hours.

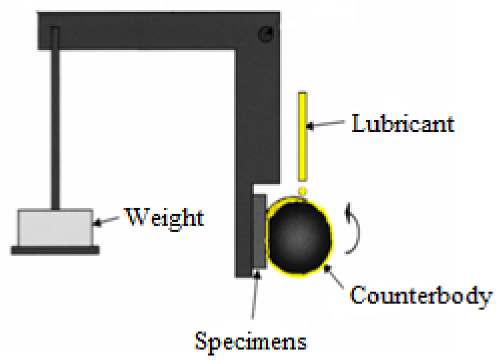

Figure 2 .Schematic drawing of machine test.

The lubricant used was introduced in the contact region between the specimens and the ball, using an ordinary syringe. At the beginning of each test 0.5 ml of lubricant was placed and each half hour, 0.1 ml was added. Before each test the specimens were ground in # 100, # 240, # 400, # 600 and # 1200 and then polished with a solution of alumina (1 µm). Both tablets and spheres were washed in ultrasonic bath for 10 minutes with isopropyl alcohol before each test.

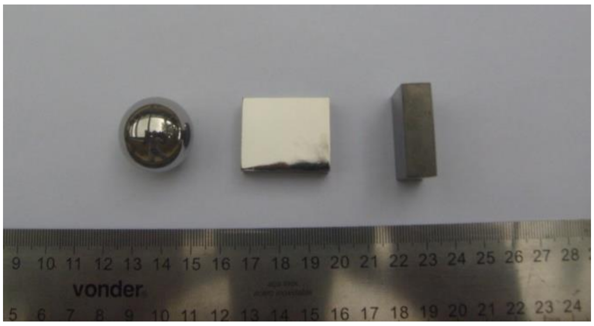

Figure 3. Specimens.

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After testing, similar cleanness were made, also in ultrasonic bath with isopropyl alcohol for 10 minutes, and examined under an optical microscope in order to make measurements of scars.

Wear was evaluated using the worn volume (v) of each scar and this value was calculated using the following equation 1[7].

$$v \approx \frac{\pi \times d^4}{64 \times R}\ , \text{ for } d \ll R \qquad (1)$$

Where d and R are the crater diameter and the radius of the sphere, respectively.

3 RESULTS AND DISCUSSION

3.1 Microstructures, Phases and Hardness

Metallographic analysis was performed on the cross section of the samples after nitriding, Figure 4. It can be noticed the formation of the white layer in the surface following by the diffusion zone formed by a martensitic structure. The white layer has an average thickness of 7.5 µm.

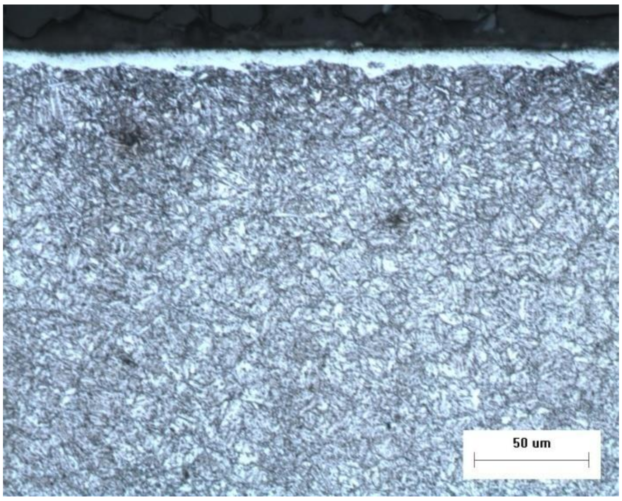

Figure 4 – Microstructure nitrided AISI 4140, nital 2%.

Figures 5 and 6 show relative intensities captured by the X-ray detector as a function of angle analysis for the tempered and quenched samples and nitrided samples respectively. Quenched and tempered samples Figure 5, shows peaks related to martensitic phase (α'), as expected and shown.

Figure 6 shows results from XRD for the nitrided samples. It can be observed peaks related to phase α' and ε, γ' phases, which have different mechanical properties.

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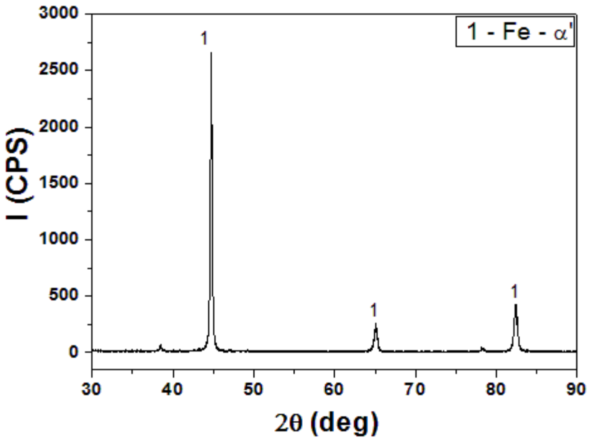

Figure 5. XRD patterns from tempered and quenched specimens.

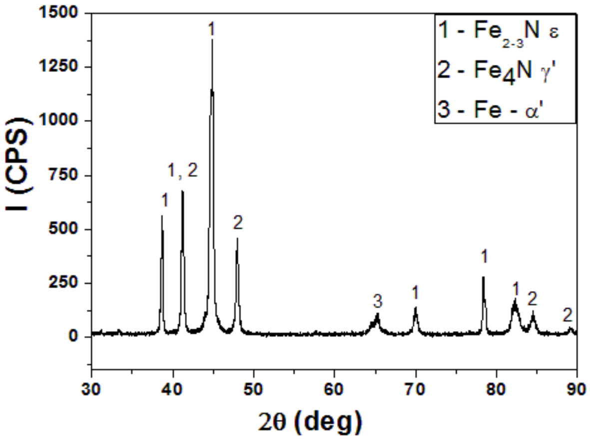

Figure 6. XRD patterns from nitrided specimens

The surface hardness of the samples was performed before (untreated samples) and after nitriding treatments (treated samples), Figure 7. Untreated samples as expected presented a lower average surface hardness. The treated samples had a significant increase in their surface harnesses increasing 3 times, showing the efficiency of the nitriding treatment. Hardness dispersion values presented in the nitrided samples are due ε and γ’ phases which have distinct mechanical properties.

Figure 8 shows the hardness profiles of untreated and nitrided samples. As expected, nitrided produced the highest surface hardness, decreasing with the distance from the surface.

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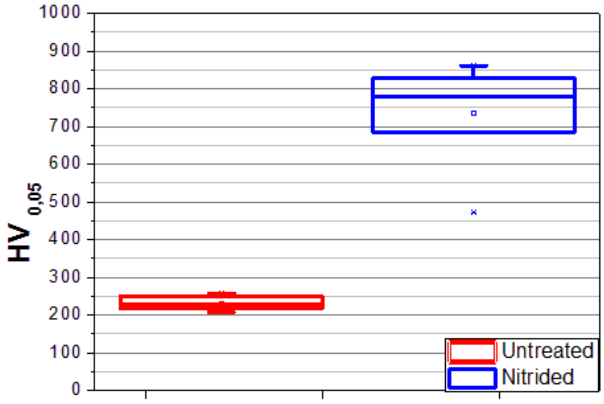

Figure 7. Superficial hardness.

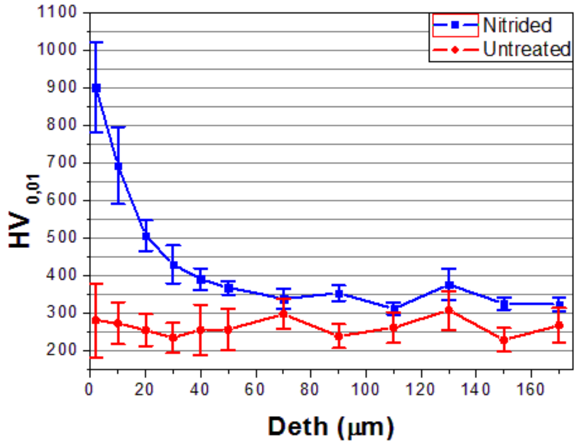

Figure 8. Hardness profiles of the samples.

3.2 Wettability

Wettability studies were performed in order to evaluate the compatibility of treated or untreated surfaces with lubricating oils tested in this study.
Analyzing wettability results, Figure 9, it can be noticed that all the oils tested showed good adhesion to all samples, with an contact angle of less than 48 °.

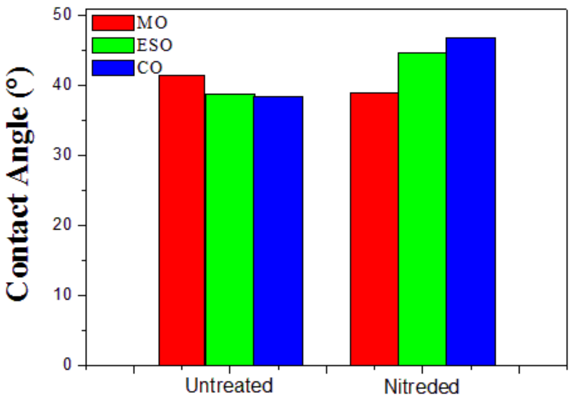

Figure 9. Wettability

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3.3 Wear Behavior

Worn volume of the specimens subjected to "ball-cratering" tests performed at room temperature, was calculated through empirical algorithm developed by Rutherford [7], described in equation 1.

Untreated specimens presented better performance when lubricated with mineral oil (figure 10), showing a smaller average volume worn, with a lower dispersion in the results. When biofluids were used as a lubricant the results showing lower performance than MO performance for same surface condition, and an average value worn about 5 times higher when ESO was used as lubricants and about 2.5 times higher samples were lubricated with CO.

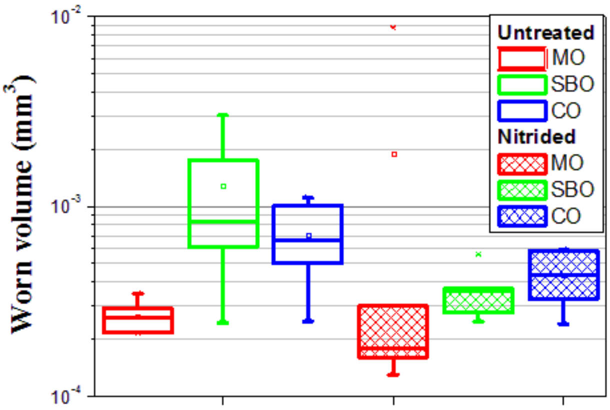

Figure 10. Worn volume

The nitrided specimens had performed better when lubricated with mineral oil (figure10), despite the amount of average volume worn to be more than the values obtained for other lubrication conditions, due to the wide dispersion between the results of the last quartiles. However, the majority (60 %) of the specimens in this condition showed lower values to those obtained with biofluids

Tests using biofluids as lubricants, there was an inversion results. The specimens untreated showed better results when lubricated with CO and nitrided specimens suffered less wear when lubricated with ESO. For all test conditions of nitrided specimens had a lower average wear.

Analyzing the wear scars of untreated specimens, Figure 11, it can be noticed that in despite of lower performance of the castor oil compared with mineral oil, the first one has a very different behavior compared to ESO. The size of the scars and their shape have a greater similarity to the craters obtained in the specimens tested with MO. This has an indication that castor oil could serve as a basis for biofluid lubricant formulations, in those applications which has same surface condition.

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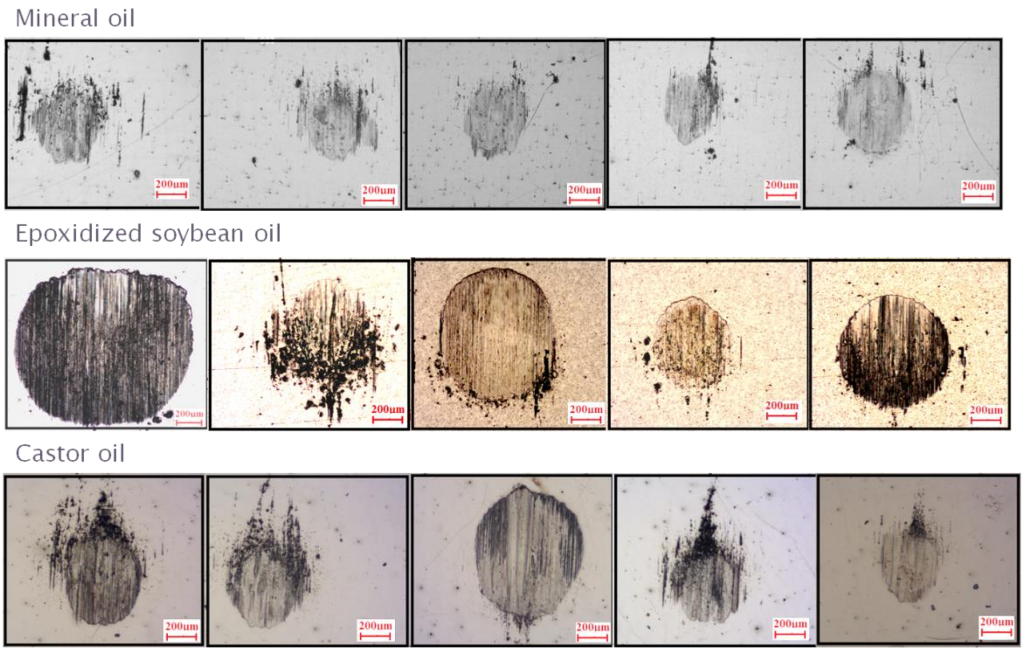

Figure 11. Scar micrographs resulting from tribological test "ball-cratering" for untreated specimens.

The difference in the scar sizes of nitrided specimens decreases when compared with the untreated samples. The figure 12 indicates similarly in the worn volumes independently of the used lubricant. It is also possible to see that in the mineral oil tests there was one which did not follow the trend of the others, causing a high average wear as shown in Figure 9.

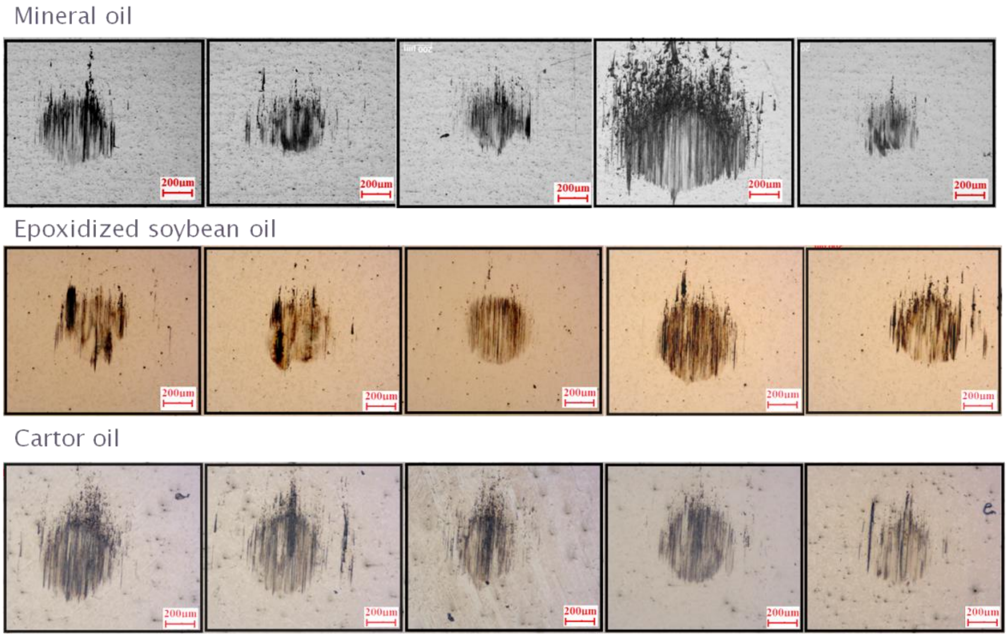

Figure 12. Micrographs of scars resulting from tribological test "ball-cratering" for nitrided specimens

The following images Figure 13 and 14 related to the untreated and treated specimens were obtained by SEM using the three types of lubricants. The scar wear regions were examined for signs of secondary electrons - (topographic) to characterize the mechanism / morphology of wear.

Figure 13 shows images obtained by analysis of specimens untreated. Figures 13a and 13b are related to the wear scars formed on the specimen surfaces lubricated

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with MO. Ploughing were observed, which presenting different values of deep and thicknesses as well as prow formation and delamination.

Figures 13c and 13d correspond to the wear scars formed on the surface of the specimens lubricated with epoxidized soybean oil. Scars present not uniform scratchs all along the scar. There was also the prow formation of prow on the top of the image (Figure 13c). The specimens lubricated with castor oil, figures 13e and 13f, as well as other specimens in the same surface condition, showed scratch which are characteristics of abrasive wear and plastic deformation in the upper region of the worn area (Figure 13e).

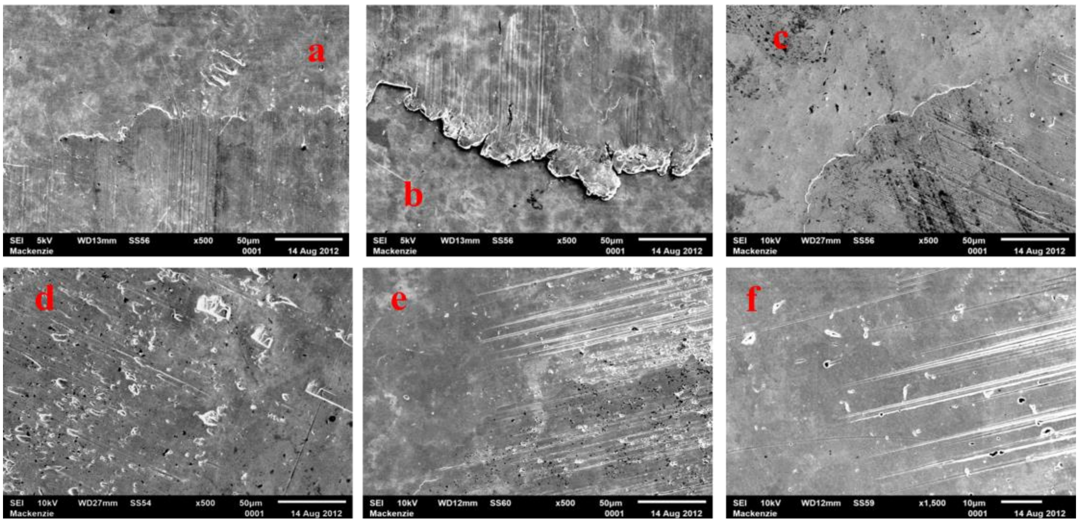

Figure 13.SEM images, scars wear untreated specimens lubricated: MO(a,b), ESO(c,d) and CO (e, f).

Illustrations of Figure 14 show scars resulting from ball-cratering test from nitrided specimens. The scars formed in tests lubricated with mineral oil, Figures 14a and 14b, did not show a well-defined geometry, predominantly abrasive wear. Figure 14c and 14d correspond to the scars resulting from lubricated test using ESO. It can be noticed that the scar presented scratchs varying in length and depth. There is also the prow formation. It can be notice that in the center of the scar there was tearing of material, figure14c, which indicates failure in the lubricant film, generating a more severe wear.

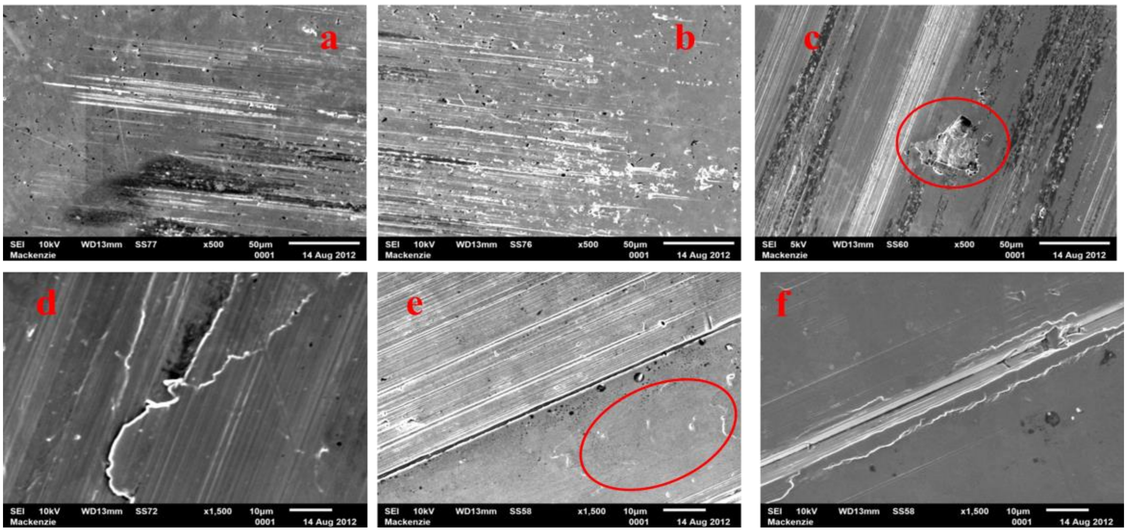

Figure 14.SEM images, scars wear nitrided specimens lubricated: MO (a,b), ESO (c,d) and CO (e, f).

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4 CONCLUSION

- As expected, the untreated and treated surfaces showed less wear when lubricated with mineral oil;
- For all tested conditions of lubrication, the nitrided specimens showed less wear;
- The biofluids tested showed a different performance between the surfaces studied. The specimens without treatment showed less wear when lubricated with epoxidized soybean oil. Already nitrided specimens showed less wear when lubricated castor oil;
- The effective wettability between all pairs tested, confirmed good adhesion between them.

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