ISSN 2176-5480

22nd International Congress of Mechanical Engineering (COBEM 2013)
November 3-7, 2013, Ribeirão Preto, SP, Brazil

EFFECT OF PLASMA NITRIDING ON ABRASIVE WEAR OF FLEXIBLE PIPES ARMOR PRESSURE

Rafael Nunes Torres
University of Brasilia, UnB, Brazil
rafantorres@yahoo.com.br

Palloma Vieira Muterlle
University of Brasilia, UnB, Brazil
pmuterlle@gmail.com

Cosme Roberto Moreira Silva
University of Brasilia, UnB, Brazil
cosmeroberto@gmail.com

Ítalo de Barros Castro
University of Brasilia, UnB, Brazil
ibp_ptu@hotmail.com

Abstract. *Flexible pipes are materials used by the Oil and Gas industry for transportation of oil from subsea wells to platforms and FPSO (Floating Production Storage and Offloading). These tubes are composed by polymeric and metallic materials that may have contact with each other. One of these layers is the armor pressure, which is a helical interlocked steel tape responsible for the resistance to internal pressure. Due to the interlocking, surfaces are in contact under tension and because of the pipe flexibility the surfaces has relative motion. This movement can cause wear between the surfaces that worsens the presence of particles torn off these surfaces, characterizing abrasive wear. The micro abrasive tests by rotating ball were carried out to obtain the wear resistance of the armor pressure. Due to design features of the pipe, the base material of the armor pressure should be a ductile material, which does not have a high wear resistance. In order to obtain better performances of wear, plasma nitriding at 10 and 30 hours with atmosphere of 70%N were carried out, this treatment is commonly used for increase wear resistance in metallic alloys. As results of the abrasive wear tests, the plasma nitriding treatment at 30 hours reduced the wear coefficient around 5% and increased the microhardness near the surface region around 15%. Regarding the microstructure, the grains sizes of plasma nitriding samples are smaller and without plastic deformation from the manufacturing process when compared to the AR samples.*

Keywords: *Abrasive wear, flexible pipes, armor pressure, plasma nitriding*

1. INTRODUCTION

With the growing demand for energy and materials derived from oil, companies responsible for operating and exploration of reserves see the need to increase production. Order for this to be possible it is necessary to invest in research techniques and materials to be used in adverse conditions, as in the pre-salt. The flexible pipes are materials used by the oil and gas industry to transport these products from wells to platforms and FPSO.

The Flexible Pipes are multilayer structures composed of polymers and metals. One of these layers is the armor pressure that is "interlocked metal layer that supports the layer of internal pressure and radial loads internal pressure of the system" (API 17B). Due to the flexibility of the system, the high contact stresses and characteristics of the use of flexible pipe, the pressure armor is subject to abrasive wear in the contact regions. API and ISO standards are the main references regarding best practices for hoses.

There are several possibilities for profiles used in armor-pressure of flexible pipes. The one that was used in this study has the Z-profile, as illustrated in Figure 1.

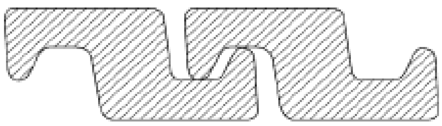


Figure 1 - Z profile of armor pressure of flexible pipes

ISSN 2176-5480

The armor pressure is manufactured from low carbon steels with some specific alloy elements, determined by the engineer department of flexible pipe to withstand the design requirements. In this case, the material used is a low carbon steel, with the base composition of AISI 1035, added with a low percentage of Si. The Si, up to 0.7%wt, has the property of reducing the grain size, acting as controlling the grain size of the austenite (ANYA e BAKER, 1989) and controlling the its formation, favoring the expansion of the ferritic field (SILVA e MEI, 2010). Furthermore, Si is dissolved in ferrite and strongly non-metallic inclusions form SiO2 (SILVA e MEI, 2010). The main role of silicon is to act as deoxidizing agent, that is, when added to liquid steel combines with oxygen, yielding solid compounds and avoiding the combination of oxygen with carbon, which would cause the detachment of CO and CO_2, causing blistering (CHIAVERINI, 2008).

At the armor pressure, the surfaces are in direct contact, with no material or lubricant responsible for the decrease of the friction in the contact region. And it's about this contact that the present paper was developed.

Wear is one of the areas of study of Tribology, according to Zum Gahr (1987) "The Tribology embraces scientific research of all kinds of friction, wear and friction, and also the technical application of tribological knowledge". The Organization for Economic Cooperation and Development - OECD (1969) defines wear as the progressive loss of substance from the operating surface of a body occurring as a result of relative motion between the surfaces. The tribology has a very large economic importance related to breaches of industrial machinery. It is believed that the economic losses due to friction and wear are up to 4% of GDP in developed countries, about £ 8.5 billion in the UK in 2001, and up to 1% of these losses can potentially be saved by the application of known techniques to reduce friction and wear in machine (JOST, 2001). In the present material, the losses are not only economic, but also environmental, since a failure in a hose can cause oil leakage.

The plasma nitriding treatment was chosen for this work due to the great success in improving the properties of various types of steel alloys. The plasma nitriding is a thermo-chemical surface treatment to improve the hardness and hence wear resistance. This is due to the formation and precipitation of nitrides and carbonitrides dispersed in the microstructure of the material distorting the lattice and increasing the surface hardness of the material (ASM METALS, 1975). Through the plasma, the nitrogen ions are accelerated in order of bombarding the steel surface, occurring nitrogen absorption and diffusion into the micro structure of the metal (ASM METALS, 1975). Regarding gas nitriding, plasma nitriding provides better control of uniformity and chemical composition of the layer, and cause less distortion in the parts (SILVA e MEI, 2010).

This paper aims to evaluate the wear of the armor pressure alloy steel, as well as the metallographic characterization and evaluation of the hardness before and after the plasma nitriding treatment. These additional characteristics will also be evaluated because their direct correlation with the wear of materials.

The previously cited treatments were chosen for this study due to the great success in improving the properties of various types of steel alloys. In their work, Baldissera and Delprete (2008) did a bibliographic review with various papers results dealing with the influence of cryogenic treatment in steels. It is a fact that the cryogenic treatment induces transformation of austenite to martensite and, in addition, carbides may form in some materials. This transformation alters mainly the properties of hardness and wear resistance of alloy steel.

2. METHODOLOGY

Table 1 presents the samples discriminations, as well as their nomenclature. The base material of the pressure armor is very similar to the AISI 1035 carbon steel.

Table 1 - Samples discriminations and treatments carried out

Sample	Nomenclature	Treatment conditions
Without Treatment (As Received)	AR	N/A
Plasma Nitriding 10 hours	PN10	500°C, 10 hours, atm. 70%N
Plasma Nitriding 30 hours	PN30	500°C, 30 hours, atm. 70%N

To evaluate the wear behavior of the material was carried out rotating ball micro abrasive wear test (Figure 2). In this method a sphere of radius R is rotated against a specimen in the presence of slurry of fine abrasive particles.

ISSN 2176-5480

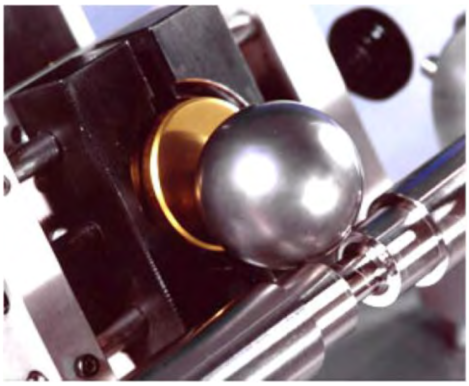


Figure 2 - Rotating ball micro abrasive wear test

It is assumed that the geometry of the wear scar on the specimen reproduces the spherical geometry of the ball, and the wear volume may then be calculated by measurement of either the crater diameter or its depth. Thus, the volume removed *V*, can be related to the sliding distance of the sphere *S* and the normal force F_N test, which is equivalent to the Archard equation for sliding wear (TREZONA e HUTCHINGS, 1999):

$$V = \kappa S F_N, \quad (1)$$

where, κ is the wear coefficient or specific wear rate with units $m^3(Nm)^{-1}$. The abrasive wear resistance is defined as κ^{-1} and has units $(Nm)m^{-3}$.

The volume of the ball crater due to wear and, consequently, the volume removed during the test can be calculated using the equation below

$$V \approx \frac{\pi b^4}{32 \emptyset_{esf}}, \quad (2)$$

where *b* is the diameter of the crater on the surface of the wear specimen.

The wear tests followed the pattern suggested by (GEE, GANT, *et al.*, 2005). This test methodology was adopted due to the lack of standards for wear testing criteria. Table 2 and Table 3 show the data and parameters for wear testing. Figura 3 shows a the equipment used in wear testing.

Table 2 - Wear testing parameters

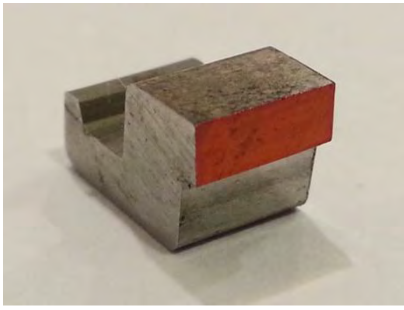
	Shaft velocity	Rotations	Testing time [s]
20 mm sphere	16 rot/10seg	508	318
		762	476
		1143	714
		1524	953
		1905	1191
		2540	1588

Table 3 - Testing parameters

Normal force [N]	0,2 ± 0,02
Velocity [m.s^{-1}]	0,1 ± 0,01
Material of the sphere	AISI 52100
Sphere diameter [mm]	20
Sphere surface finish	Conditioned using run-in procedure
Abrasive material	SiC
Fluid carrier	Water
Feed rate	Keep wet
Abrasive concentrations [% vol]	20
Test duration (approximate number of ball revolutions)	508, 762, 1143, 1524, 1905, 2540

Figura 3 - Abrasive wear test equipment (CSM Calowear)

Besides the wear tests were conducted Vickers (HV0.3) microhardness profiles for evaluating this property of the material throughout the thickness of the sample. Were prepared two types of samples for the microhardness tests and metallographic characterization. Are named Sample 1, all samples cut longitudinally along the axis of rolling of the profile (Figure 4 - (a)) and Sample 2 are all the samples of the face of the profile (Figure 4 - (b)).



(a)
Sample 1

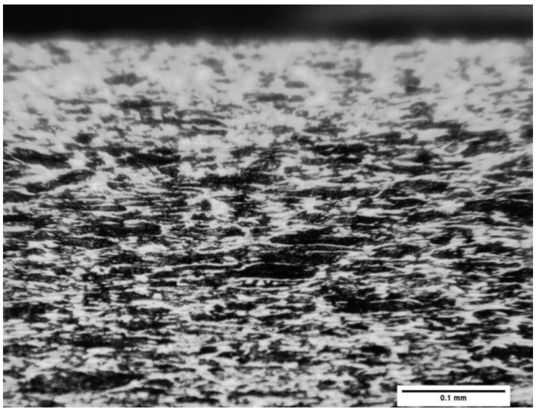
(b)
Sample 2

Figure 4 - Cutted faces of the samples

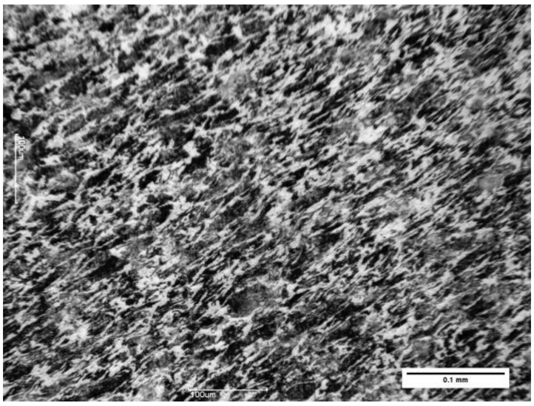
To prepare the samples for metallography, they were abraded with sandpaper with granulometry of 180, 240, 400, 600, 800, 1200, polished with abrasive paste of 15, 6, 3 and 1μm and 2% Nital etching. Metallographic images in Figure 5 were obtained by optical microscopy.

3. RESULTS AND DISCUSSION

Figure 5 (a) to (f) presents the metallographic characterization of the samples. Due to the percentage of carbon of the steel (0.35 to 0.4% C), the samples microstructure consists pearlite and ferrite phases.

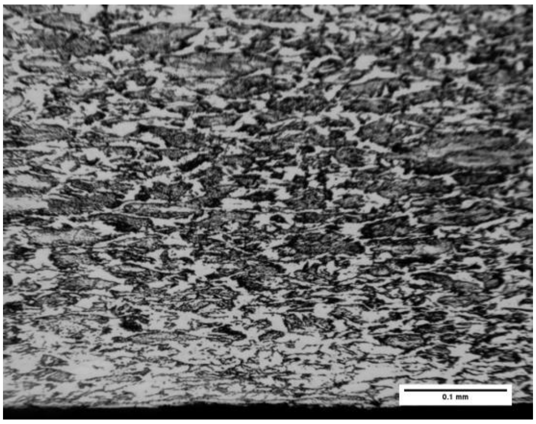


(a) - Sample 1, AR

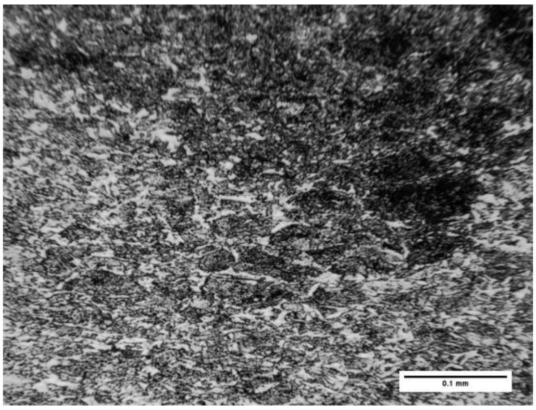


(b) - Sample 2, AR

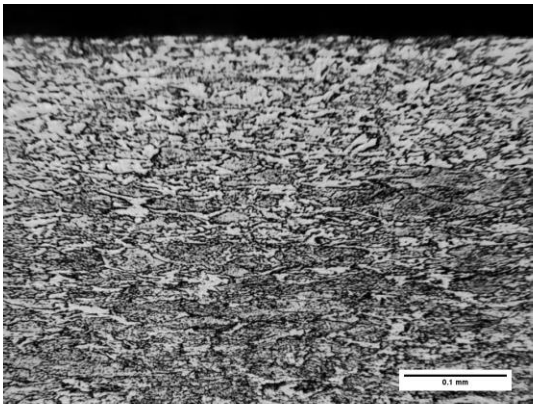
ISSN 2176-5480



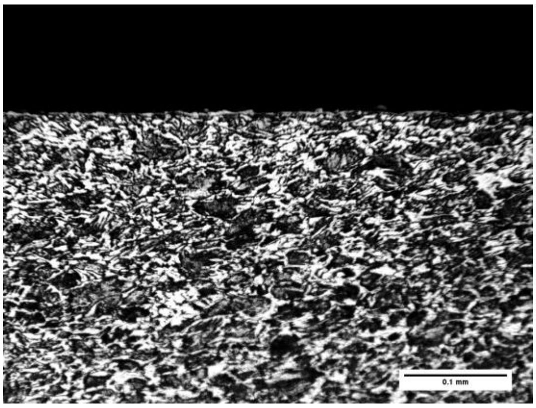
(c) - Sample 1, Plasma Nitriding 10 hours



(d) - Sample 2, Plasma Nitriding 10 hours



(e) - Sample 1, Plasma Nitriding 30 hours



(f) - Sample 2, Plasma Nitriding 30 hours

Figure 5 - Samples metallography (Magnification: 500x)

It can be observed from the AR samples the lamination direction which provides the strain hardening of the material. From Figure 5-(c) can be seen that remains a little influence of the lamination process and at Figure 5-(e) this effect is not found. The grains sizes of plasma nitriding samples are smaller and without plastic deformation from the manufacturing process when compared to the AR samples. This refinement was caused by the procedure of the plasma nitriding, in which the samples are treated in a controlled atmosphere furnace at a temperature of 500°C, approximately, causing the recrystallization of the sample. The plasma nitriding form the nitrided layer, which is formed by a zone of compounds and a diffusion zone. The compounds zone is located in the top layer being formed of nitrides and carbonitrides, primarily. This region is known as white layer. The diffusion zone starts just below the zone of compounds being formed due to the diffusion of nitrogen into the metal and may have inconsistent precipitation of nitrides (GOBBI, 2008). However, observing the images of the material after nitriding (Figure 5 (c) to (f)), there was no formation of white layer

Figure 6 and Figure 7 show the test results of Vickers microhardness profile (HV0.3), wherein the abscissa is the distance from the surface. In the regions near the surface, there is a greater hardness in the samples treated, but advancing the profile occur the hardness reduction. As wear is a surface phenomenon, the crater did not reach greater depths. In the wear tests conducted for this article, the calculated maximum depth was 0.08 mm (depending on the diameter of the crater at the surface wear).

On Table 4 can be seen values of Vickers hardness tests carried out on the samples. Near to the surface an improvement of the hardness are provided by the treatments. This increment was of 15% for the PN10 and of 11% for the PN30, when compared to the sample on AR condition (comparing Samples 1).

Table 4 - Vickers Micro Hardness (HV0.3) Near to Surface (values from Figure 6 and Figure 7)

Vickers Hardness (HV0.3)					
As Received		**Plasma Nitriding 10hr**		**Plasma Nitriding 30hr**	
S1	S2	S1	S2	S1	S2
300	322	346	312	333	370

ISSN 2176-5480

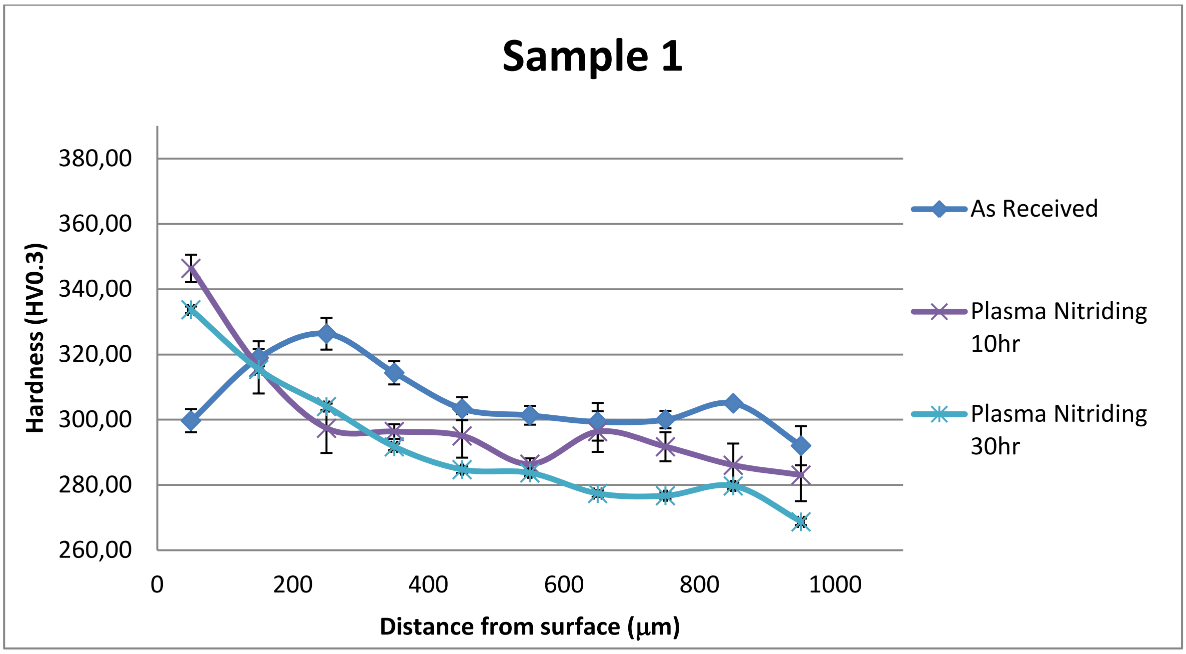


Figure 6 - Vickers (HV0.3) micro hardness profile - Sample 1

Figure 7 shows that was no improvement of hardness for the PN10 (-3.8%) and for the PN30 there was an improvement of 15% on hardness, when compared to the value of the hardness on the Sample 2 of AR condition.

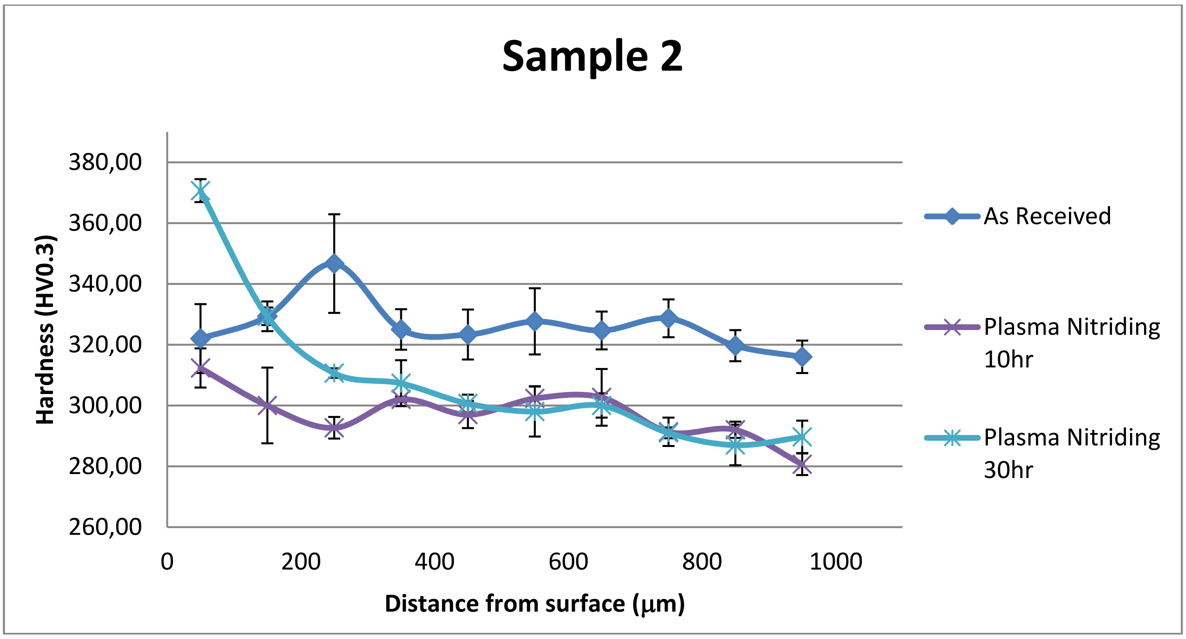


Figure 7 - Vickers (HV0.3) micro hardness profile - Sample 2

Figure 8 shows a graphic of the results of the wear tests. The area highlighted by the red ellipse shows the initial behavior (transient) wear. In this phase, the wear may not have default behavior in relation to test time or sliding distance of the sphere. The rest of the chart is said steady state wear. The steady-state wear coefficient occurs when the wear reaches a stable value over time (FRANCO, 2009).

ISSN 2176-5480

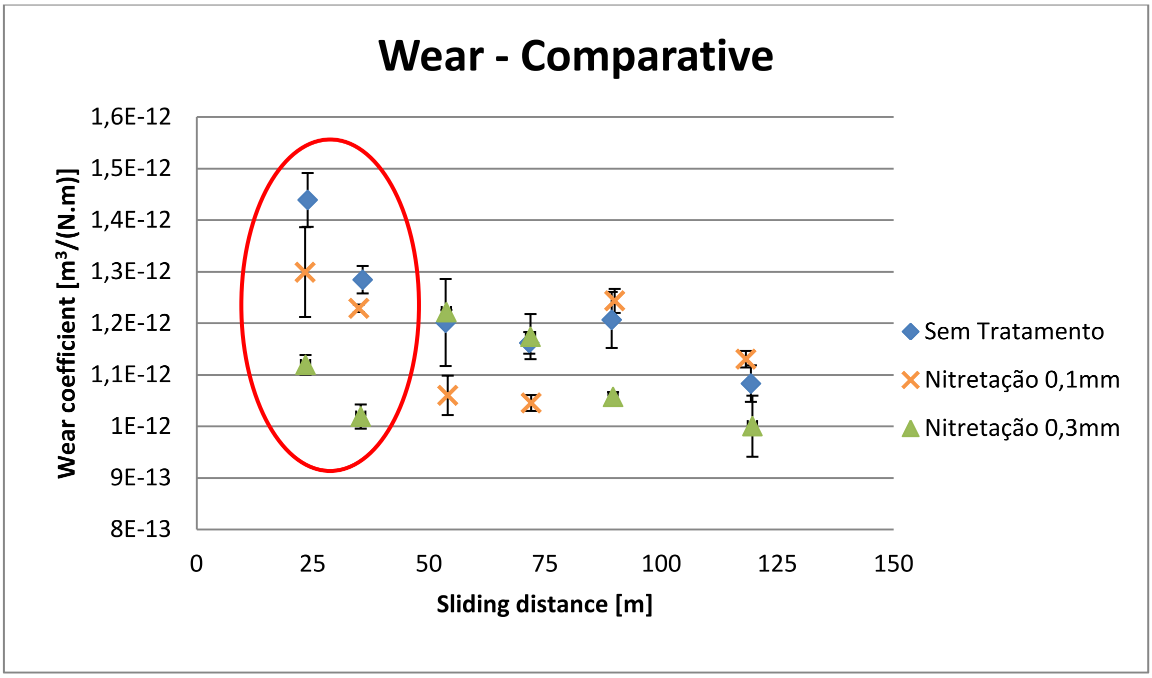


Figure 8 - Comparative chart: Wear coefficient *versus* sliding distance of the ball

It is observed that there was little improvement on the wear resistance of PN10 conditions when compared to the results of the samples AR. When analyzing the results of PN30, there was an improvement in the coefficient of wear, especially in the initial wear (transient), according to what is expected by the treatment based on bibliography review.

The hardness of the material can be indicative of their behavior with respect to wear, although this relationship may not always be proportional, as can be seen in the results presented here. The PN30 provided the increase in hardness in the region near the surface, but the advantage in terms of wear was not expected, i.e. 15% increase in hardness corresponded to a 5% reduction in wear.

4. CONCLUSION

Regarding the microhardness, the plasma nitriding treatment showed improvement in the region close to the surface. The treatments did not achieved fully satisfactory results in relation to reducing the wear coefficient of the material, providing less than 5% of decrease on wear coefficient. If the white layer had been formed on the surface, due to plasma nitriding, it could have achieved better results; both on microhardness and on wear. Even with the improvement shown by the results of plasma nitriding treatment, the costs of the procedure does not justify large-scale application based only on reported results.

Anyway, the application of these and other methods of improving component materials of flexible pipes requires a series of characterization tests of mechanical, physical and chemical properties, for many different service conditions.

5. ACKNOWLEDGEMENTS

Thanks to Petrobras for inclusion in the University of Brasilia PRH-224 and the availability of funding for research. I thank the University of Brasilia for the provision of material and technical resources to conduct this work. Thanks to the Prysmian Surflex support in this research. To my family and friends.

6. REFERENCES

ANYA, C. C.; BAKER, N. T. **The Effect of Silicon on the Grain Size and the Tensile Properties of Low Carbon Steels**. Materials and Science Engineering, 1989. 197-206.

API-17B. API 17B - **Recommended Practice for Flexible Pipes**.

ASM METALS, H. **Properties and Selection of materials**. ASM Metals Handbook, vol. 2, 1975.

ASM METALS, H. **Plasma (Ion) Nitriding of Steels**. ASM Metals Handbook, Vol. 4, 1991.

BALDISSERA, P.; DELPRETE, C. **Deep Cryogenic Treatment: A Bibliographic Review**. The Open Mechanical Engineering Journal, 2008.

CHIAVERINI, V. **Aços e Ferros Fundidos**. 7. ed. [S.l.]: ABM - Associação Brasileira de Metalurgia e Materiais, 2008.

FRANCO, A. H. **Estudo da influência da criogenia na resistência ao desgaste abrasivo do aço ASTM 743 CA6NM**. Dissertação de Mestrado, 2009.

GEE, M. G. et al. **Results from interlaboratory exercise to validate the micro-scale abrasion test**. Wear, vol. 259, 2005. 27-35.

GOBBI, V. J. **Influência da nitretação a plasma na resistência ao desgaste microabrasivo do aço ferramenta AISI D2**. Universidade de Brasília, UnB. [S.l.]. 2008.

JOST, H. P. **The task of tribology societies on a changing world**. Second World Tribology Congress, 2001.

OECD. Friction, **Wear and Lubrication: Terms and Definition**. Research Group on Wear of Engineering Materials, 1969.

SILVA, A. L. V. D. C.; MEI, P. R. **Aços e Ligas Especiais**. 3. ed. [S.l.]: Editora Blucher, 2010.

TREZONA, R. I.; HUTCHINGS, I. M. **Three-body abrasive wear testing of soft materials**. Wear, 1999.

ZUM GAHR, K. H. **Microstructue and wear of materials**. [S.l.]: Elsevier, 1987.

7. RESPONSIBILITY NOTICE

The authors are the only responsible for the printed material included in this paper.