

Wear resistance of AISI 304 stainless steel submitted to low temperature plasma carburizing

<http://dx.doi.org/10.1590/0370-44672016700094>

Marcos Antônio Barcelos

Professor

Instituto Federal de Educação, Ciência e Tecnologia do Espírito Santo - Coordenadoria de Mecânica
Vitória - Espírito Santo - Brasil
marcosbarcelos@ifes.edu.br, mbbarcelos@yahoo.com.br

Mariana Valinhos Barcelos

Professora

Instituto Federal de Educação, Ciência e Tecnologia do Espírito Santo - Coordenadoria de Metalurgia
Vitória - Espírito Santo - Brasil
mvalinho@ifes.edu.br

Juraci de Sousa Araújo Filho

Professor

Instituto Federal de Educação, Ciência e Tecnologia do Espírito Santo - Coordenadoria de Mecânica
Aracruz - Espírito Santo - Brasil
juraciaraujo@ifes.edu.br

Adonias Ribeiro Franco Jr.

Professor

Instituto Federal de Educação, Ciência e Tecnologia do Espírito Santo - Programa de Pós-Graduação em Engenharia Metalúrgica e de Materiais PROPEMM
Vitória - Espírito Santo - Brasil
adonias@ifes.edu.br

Estéfano Aparecido Vieira

Professor

Instituto Federal de Educação, Ciência e Tecnologia do Espírito Santo - Programa de Pós-Graduação em Engenharia Metalúrgica e de Materiais PROPEMM
Vitória - Espírito Santo - Brasil
estefanovieira@ifes.edu.br

1. Introduction

The technological advances in several industries and increased market demands are requiring the use of treatment techniques to optimize material properties in order to obtain high-quality products. The improvement of steel mechanics with a reduced cost is a constant goal. Low temperature plasma carburizing (LTPC) is an efficient thermochemical treatment for increasing the hardness of austenitic

Abstract

Despite the AISI 304 stainless steel has high corrosion/oxidation resistance, its tribological properties are poor, being one of the barriers for use in severe wear applications. Thus, there is a wide field for studying technologies that aim to increase the surface hardness and wear resistance of this material. In this work, hardness and wear resistance for AISI 304 stainless steel submitted to the thermochemical treatment by low temperature plasma carburizing (LTPC) in a fixed gas mixture composition of 93% H₂ and 7% CH₄ are presented. Through the evaluation of the carburizing layers, it was possible to observe a substantial improvement in tribological properties after all temperature and time of treatment. This improvement is directly related to the increase of the process variables; among them temperature has a stronger influence on the wear resistance obtained using LTPC process.

Keywords: carburizing; plasma; stainless steel AISI 304; wear.

stainless steel, combining good tribological properties, low cost and no environmental problems. This technique has been used in A316 austenitic steels and the high mechanical and corrosion performance is due to the formation of an expanded austenitic microstructure, also known as the carbon S phase. The hardening mechanism is based on supersaturation of carbon into the austenitic structure,

expanding the FCC cell and producing high levels of residual stress (Gallo et al., 2012; Pinedo and Tschiptschin, 2013; Sousa et al., 2013).

LTPC is a process controlled by diffusion, so time and temperature of the treatment are limiting parameters. However, if the temperature is higher than 450°C, the S phase may decompose, resulting in precipitation of carbides such

as Cr_{23}C_6 and Cr_7C_3 that reduce wear and corrosion resistance. The combined improvement in hardness, wear properties, and excellent corrosion resistance are responsible for making the S phase one of the most significant recent developments in stainless steels (Sun and Bell, 1993; Bell, 2002; Sun and Bell, 2002; Cheng *et al.*, 2005; Mendes *et al.*,

2014). When temperature or treatment time are increased, the S phase thickness also increases, but the possibility of forming carbides is higher. Therefore, the objective is to promote the carbon adsorption on the steel surface at sufficiently low temperatures, maximizing the S layer and avoiding the precipitation of carbides, as described in some papers

written by Souza *et al.*, Sun, Dong *et al.* and Adachi *et al.* (Sun, 2005; Dong *et al.*, 2006; Sousa *et al.*, 2009; Adachi and Ueda, 2013). In this work, a commercial AISI 304 stainless steel was chosen to be studied under LTPC because few information on the tribological behavior of this alloy is available (Sun, 2005; Dong *et al.*, 2006).

2. Material and method

The AISI 304 stainless steel used in this work was received in the form of cylindrical bars with a diameter of 29 mm. In Figure 1 is presented the experimental procedures used in this study. The first preparation phase was to cut and grind the specimens. The final thickness of

each sample was approximately 3.0 mm. The surface was prepared following the conventional metallographic grinding and polishing techniques. The samples were grinded with sandpaper and polished using 0.3 μm alumina suspension. The chemical composition was determined using an opti-

cal spectrometer *Oxford Model Foundry Master Pro* and the result was compared to the specified chemical composition according to ASTM A959 designation, and is presented in Table 1 (ASTM, 2014). The chemical composition was in accordance with nominal specifications.

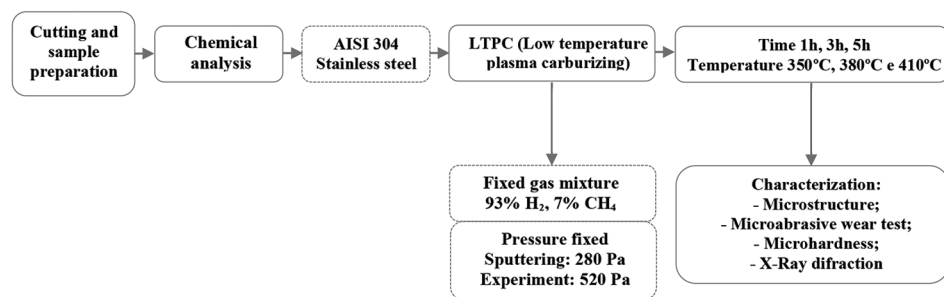


Figure 1
Flowchart of experimental procedures steps.

Type	A959-11ASTM Designation	Composition(%)						
		C	Mn	Si	Cr	Ni	P	S
AISI 304	S30400	0.07*	2.00*	1.00*	17.5-19.5	8.0-11.0	0.045*	0.030*
Measured	-	0.08	1.92	0.75	18.00	8.34	0.040	0.02

*maximum, unless range or minimum is indicated.

Table 1
Chemical composition of the studied AISI 304 steel.

2.1 Plasma Carburizing

The thermochemical treatments were performed for two samples per condition. Initially, the sputtering procedure under argon atmosphere was carried out at temperature of 300°C and pressure of 280 Pa during 30 minutes in order to

remove the oxide layer. After sputtering, the treatments were performed under a fixed gaseous mixture of 93% H_2 and 7% CH_4 at temperatures of 350°C, 380°C and 410°C and times of 1 h, 3 h and 5 h. The samples were cooled inside the chamber

until room temperature. The experimental parameters were chosen based on Gallo *et al.*, Sun and Souza *et al.* (Gallo and Dong, 2009; Sun, 2009; Sousa *et al.*, 2009) works and the main aim was to prevent carbide formation during the experiments.

2.2 Microstructural Characterization

For each experiment, one sample was cut in a perpendicular direction of the LTPC using a high precision cutter machine. The perpendicular

surface was then ground, polished and etched with a modified *aqua regia* solution, which was a mixture of 8 mL of HNO_3 and 12 mL HCl diluted

in 1 L of ethyl alcohol. The images of the microstructures were obtained by Scanning Electron Microscopy (SEM) *Leica Model Sy40i*.

2.3 Hardness

The microhardness evaluations were performed on the sample's top surface and hardness profile of the cross-section was

carried out for each condition. Microhardness tests were performed in a Hardness Tester *Shimadzu Model HMV-2*. The

load for all measurements were 10 g using a Vickers indentation, with minimum distance between measurements of 10 μm .

2.4 Phase Determination

The material phase identification, as received, was done through an X-Ray diffractometer *Bruker Model D2 Phaser*.

Measurements for each carburized sample were performed to identify new phases in function of LTPC parameters. The analysis

were carried out with acceleration of 30 kV, applied current of 10 mA, step size of 0.05° and scanning range from 20° to 90°.

2.5 Microabrasive wear test

To measure the wear resistance, the second sample of each experiment was submitted to a micro-abrasive wear test according to the methodology proposed by Rutherford *et al.* (Rutherford and Hutchings, 1996). A ball-crater was used in the abrasion test, also known as micro-scale abrasive wear test, which consists of the wear occurring by the action of a rotating ball pressed against the sample's flat surface with an addi-

tion of an abrasive slurry in the wear interface, producing a crater. The tests were performed in the free-ball micro-abrasion equipment *CSM Calowear*, using an AISI 52100 steel ball with diameter of 25.4 mm, sliding speed of 0.05 m/s, silicon carbide (SiC) abrasive slurry with an average particle size of 4.5 μm and concentration of 0.75 g/cm³. The slurry was dripped on the ball's top surface at a flow-rate of approximately 1

drop per 3 s. The normal load (F_N) was set as 0.24 N using a load cell with an accuracy of ± 0.005 N. The worn craters were measured in intervals of 5, 15, 30 and 45 min. The craters were characterized by the optical microscope attached to the micro-abrasion equipment. The wear coefficient (k), as a function of time, was calculated after the permanent wear regime was achieved, using the Equation 1:

$$k = \frac{V}{L.FN} \quad (1)$$

Where F_N is the load normal to the testing

sphere, V is the worn volume, and L is the

sliding distance (Cesconetto *et al.*, 2015).

3. Results and discussion

3.1 Layer thickness

Figs. 2, 3 and 4 present the cross section micrographs of samples after LTPC at temperatures of 350°C, 380°C and 410°C and treatment time of 1 h, 3 h, and 5 h. At 350°C, whereby the thickness increased with time, from 3.9 to 6.0 μm, while at 380°C the thicknesses were 5.9, 7.0 and 7.6 μm. However, the best results were obtained at 410°C,

where the thickness increased from 5.9 to 11.0 μm. It is observed that the carbon-S phase appears uniform, with its thickness increasing gradually with time and temperature, reaching 11.0 μm at 410°C and a carburizing time of 5 h, probably due to the stabilization of the diffusion process. The same behavior was observed by Sun, concluding that

a long treatment time is not required because the process of layer obtainment is described by Fick's second law (Sun, 2005). Other examples of AISI 304 studies are Sun (Sun, 2005), who obtained a layer of 29.0 μm at the temperature of 475°C and 20 h, and Dong *et al.*, (Dong *et al.*, 2006), who obtained 15.0 μm at 425°C and 12 h.

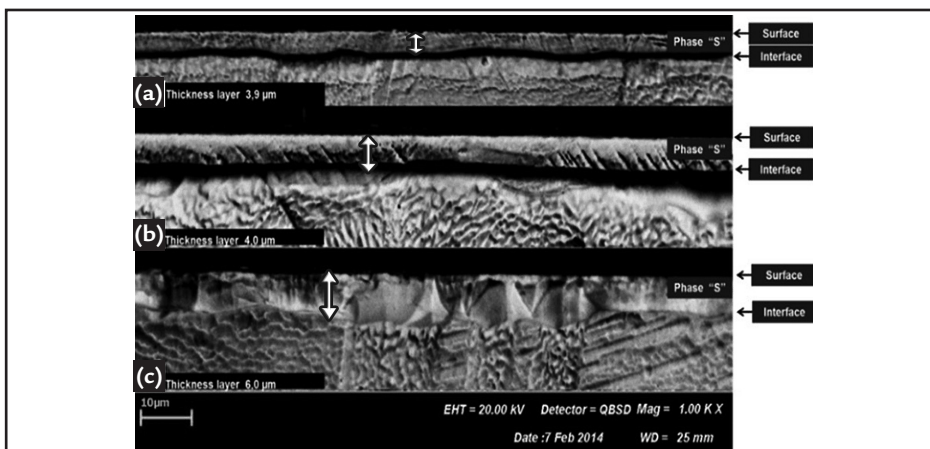


Figure 2

Cross section micrographs of samples after carburizing of AISI 304 stainless steel at the temperature of 350°C and different carburizing times: (a) 1 h, (b) 3 h and (c) 5 h.

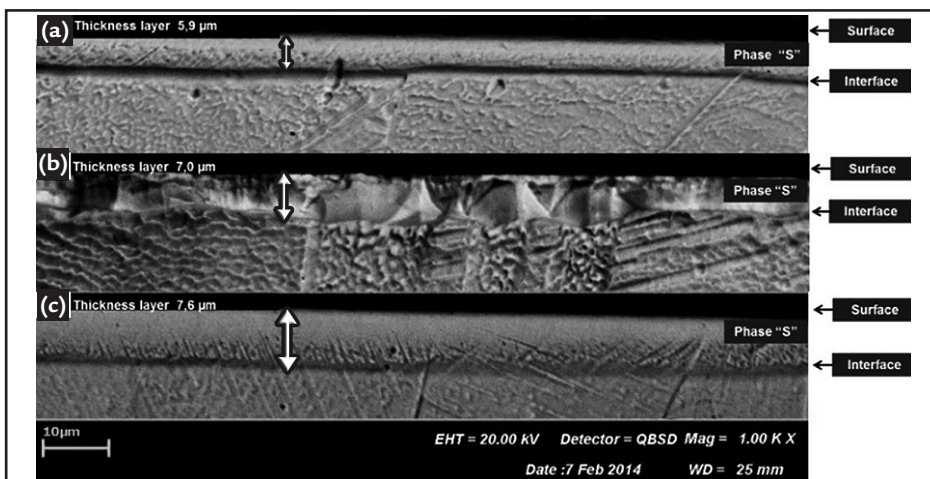


Figure 3

Cross section micrographs of samples after carburizing of AISI 304 stainless steel at the temperature of 380°C and different carburizing times: (a) 1 h, (b) 3 h and (c) 5 h.

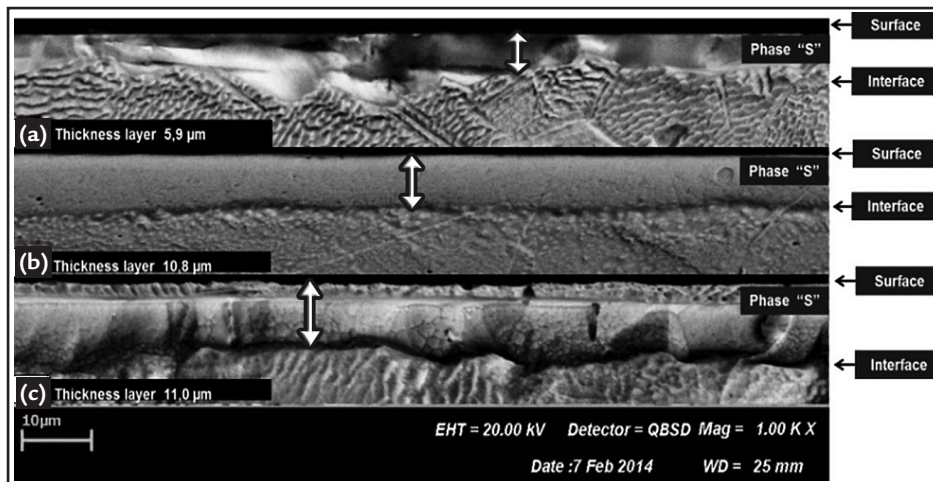


Figure 4
Cross section micrographs of samples after carburizing of AISI 304 stainless steel at the temperature of 410°C and different carburizing times: (a) 1 h, (b) 3 h and (c) 5 h.

3.2 Hardness

The hardness at the top of the samples after the LTPC heat treatment is shown in Figure 5, demonstrating that hardness increases with time. At a temperature of 410° and 5 hours of carburizing time, the highest hardness was achieved: 470 HV0.01, against the matrix hardness

of only 200 HV0.01. In Figure 6 is shown the influence of the treatment parameters on the samples hardness profiles and it can be seen that the hardness reduction is smooth, promoting an advantage when compared to the nitriding process for the same steel. Even though the plasma nitrid-

ing process is more efficient to increase the hardness, the LTPC process presents a better performance because the hardness profile from the surface to the center does not change abruptly; therefore there is a gradual decrease when the steel is exposed to abrasive environment (Bell, 2002).

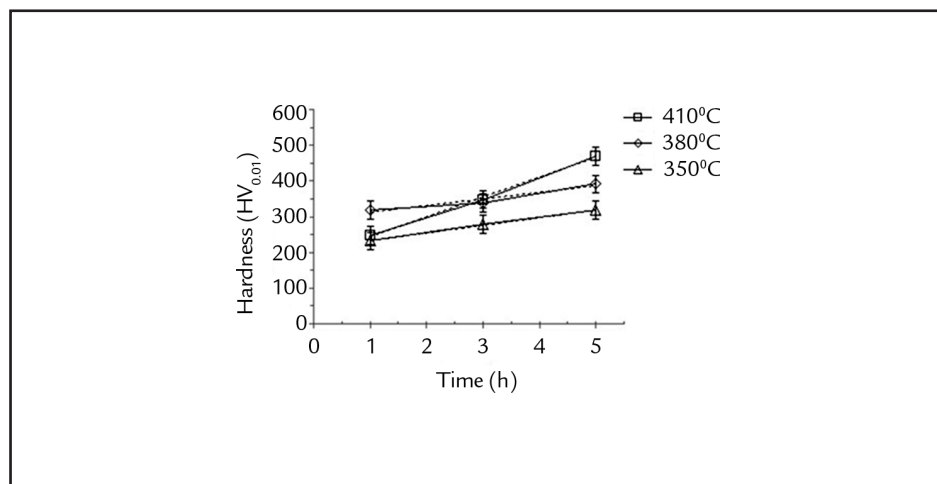


Figure 5
Hardness of the AISI 304 stainless steel on the top surface after LTPC heat treatment at different temperatures.

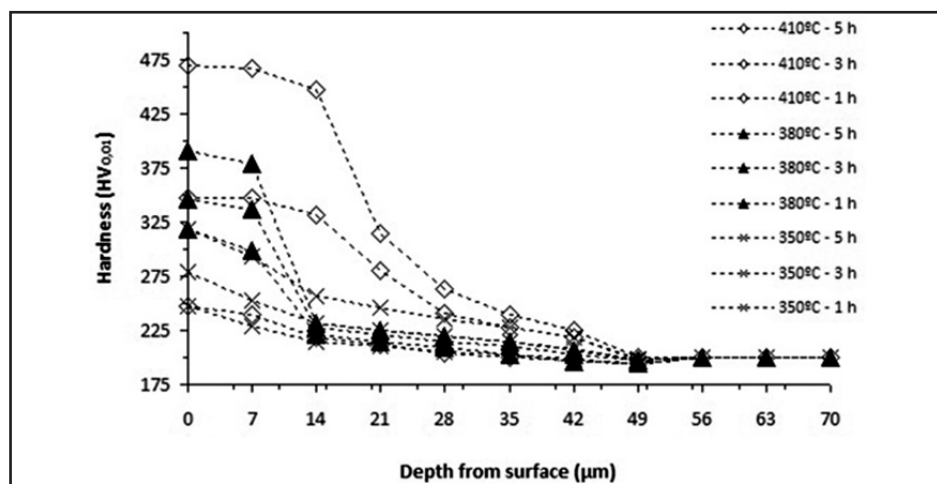


Figure 6
Hardness profile of AISI 304 stainless steel after LTPC heat treatment at different times and temperatures.

3.3 X-Ray Diffractograms

In Figure 7 are presented the X-Ray diffractograms of the samples before and after the LTPC. The AISI 304 steel, as

received, didn't show any peak related to the presence of carbides and S phase. For all treated samples, the peaks were

slightly shifted to the left, i.e. to smaller angles, which makes evident an expansion of the lattice due to the increase of carbon

content (Cheng *et al.*, 2005). The diffraction patterns didn't present any carbide

peak, evidencing that those phases are not significantly present.

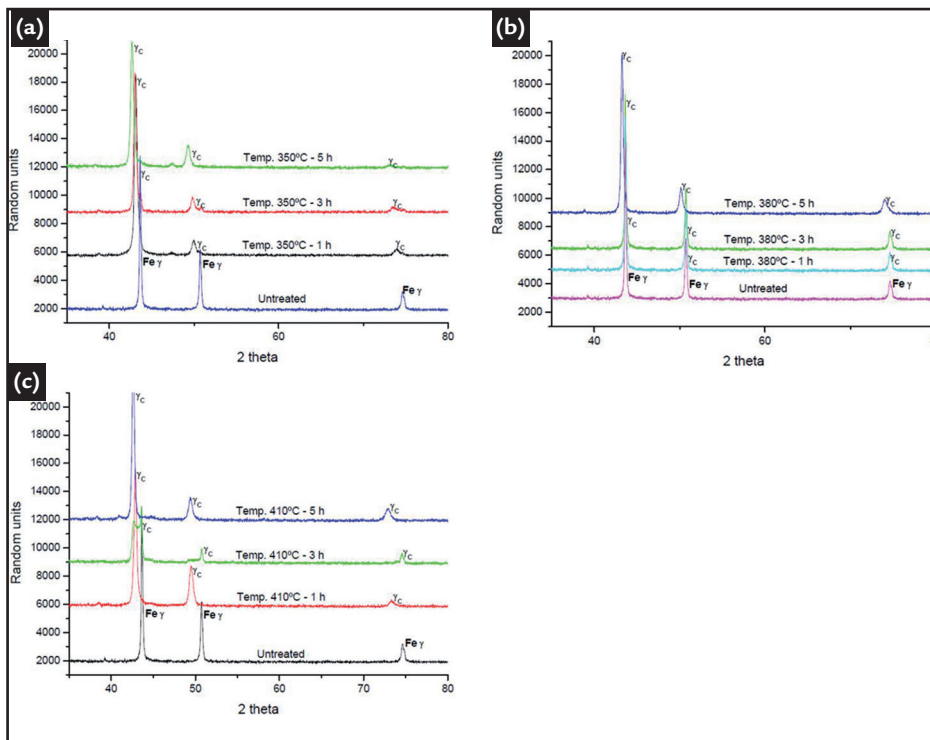


Figure 7
X-ray diffraction (XRD) patterns of the untreated AISI 304 stainless steel and the samples submitted to LTPC process at different temperatures: (a) 350°C, (b) 380°C and (c) 410°C.

3.4 Micro-abrasive wear

Figure 8 correlates the treatment time with the wear coefficient. For all conditions, wear resistance increases

when compared to the untreated sample. The best results were achieved for a temperature of 410°C and a time of 5 h. The

active mechanism of wear in carburized samples was also determined, as can be seen in Figure 9.

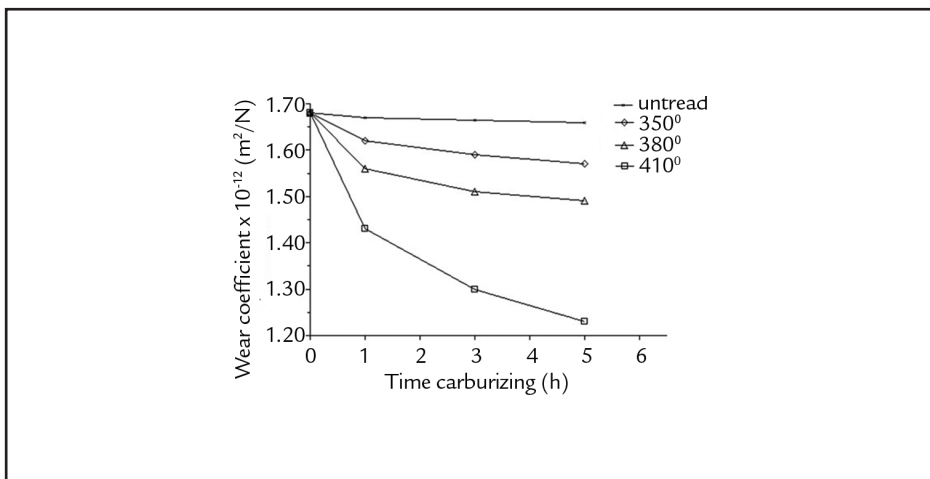


Figure 8
Micro-abrasive wear coefficient as a function of time for AISI 304 stainless steel carburized at different temperatures.

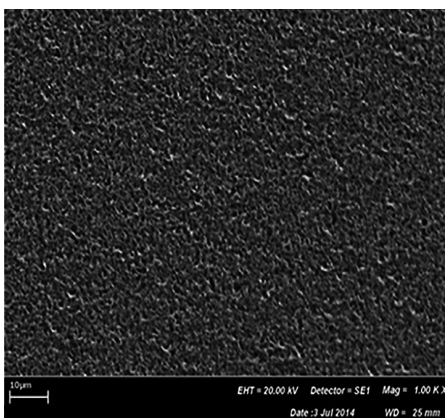


Figure 9
Worn surface after micro-abrasive wear test of AISI 304 stainless steel carburized for 5 hours at temperature of 380°C.

4. Conclusions

The use of plasma carburizing on AISI 304 stainless steel was beneficial for all studied conditions.

The depth of S layer becomes thicker when the heat treatment temperature and rest time were increased. The highest layer thickness obtained was 11.0 μm and it remained almost

constant for long period of time due to the lower diffusivity of the carbon in the formed S layer.

The increase of S phase thickness and hardness improved the wear resistance, but extended heat treatments are not recommended because no significant gains are achieved. The

wear mechanisms using silicon carbide (SiC) identified was a rolling abrasive mechanism, which is common for systems where particles with lower hardness are used.

Wear resistance improved due to the formation of expanded austenite, i.e. S layer. Carbide precipitation was avoided.

Acknowledgments

The authors would like to thank to IFES, FINEP, CNPq and FAPES - Brazilian research agencies that supported this work.

References

- ADACHI, S., UEDA, N. Surface hardness improvement of plasma-sprayed AISI 316L stainless steel coating by low-temperature plasma carburizing. *Advanced Powder Technology*, 24, p. 818-823, 2013.
- ASTM INTERNATIONAL. ASTM A959: Standard guide for specifying harmonized standard grade compositions for wrought stainless steels. USA: ASTM INTERNATIONAL, 2014.
- BELL, T. Surface engineering of austenitic stainless steel. *Surface Engineering*, v.18, n.6, p. 415-422, 2002.
- CESCONETTO, M. C. R. L, FRANCO JUNIOR, A. R., VIEIRA, E. A. Improving the abrasive wear resistance of a microalloyed steel by plasma nitriding. *Materials Research*. v.18, n.2, p. 334-340, 2015.
- CHENG, Z. J., LI, C. X., DONG, H., BELL, T. Low temperature plasma nitrocarburising of AISI 316 austenitic stainless steel. *Surface and Coatings Technology*, 191, p. 195-200, 2005.
- DONG, H., QI, P. -Y., LI, X. Y., LLEWELLYN, R. J. Improving the erosion-corrosion resistance of AISI 316 austenitic stainless steel by low-temperature plasma surface alloying with N and C. *Materials Science and Engineering A*, 43, p. 137-145, 2006.
- GALLO, S. C., DONG, H. Study of active screen plasma processing conditions for carburising and nitriding austenitic stainless steel. *Surface and Coatings Technology*, 203, p. 3669-3675, 2009.
- GALLO, S. C., LI, X., DONG, H. Dry sliding wear of active screen plasma carburised austenitic stainless steel. *Tribol let*, v. 45, n.1, p. 153-160, 2012.
- MENDES, A. F., SCHEUER, C. J., JOANIDIS, I. L., CARDODO, R. P. Low-temperature plasma nitriding of sintered PIM 316L austenitic stainless steel. *Materials Research*, v.17, n.1, p. 100-108, 2014.
- PINEDO, C. E., TSCHIPTSCHIN, A. P. Low temperature plasma carburizing of AISI 316L austenitic stainless steel and AISI F51 duplex stainless steel. *REM-Revista Escola de Minas*, v.66, n.2, p. 209-214, 2013.
- SOUZA, R. M., IGNAT, M., PINEDO, C. E., TSCHIPTSCHIN, A. P. Structure and properties of low temperature plasma carburized austenitic stainless steels. *Surface & Coatings Technology*, 204, p. 1102-1105, 2009.
- SOUZA, R. R. M., DE ARAÚJO, F. O., GONTIJO, L. C., DA COSTA, J. A. P., ALVES Jr, C. Cathodic cage plasma nitriding (CCPN) of austenitic stainless steel (AISI 316): influence of the different ratios of the (N₂/H₂) on the nitrided layers properties. *Vacuum*, v.86, n.12, p. 2048-2053, 2012.
- SUN, Y., BELL, T. The role of oxidation in the wear of plasma nitrided and PVD TiN coated steel. *Wear*, 166, p. 119-125, 1993.
- SUN, Y., BELL, T. Dry sliding wear resistance of low temperature plasma carburised austenitic stainless steel. *Wear*, 253, p. 689-693, 2002.
- SUN, Y. Kinetics of low temperature plasma carburizing of austenitic stainless steels. *Journal of Materials Processing Technology*, 168, p. 189-194, 2005.
- SUN, Y. Response of cast austenitic stainless steel to low temperature plasma carburizing. *Materials and Design*, 30, p. 1377-1380, 2009.
- RUTHERFORD, K. L., HUTCHINGS, I. M. A micro-abrasive wear test, with particular application to coated systems. *Surface and Coatings Technology*, v.79, n.1-3, p. 231-239, 1996.