INFLUENCE OF PLASMA NITROCARBURIZING ON FATIGUE AND CREEP PROPERTIES IN A 4340 STEEL WITH DIFFERENT MICROSTRUCTURE

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Abstract. *Three different routes of heat treatments were applied in samples of 4340 steel in order to modify the microstructures and mechanical properties. After this initial treatment was applied a plasma nitrocarburizing thermochemical treatment in a part of the samples, forming a layer of nitride (Fe_4N and Fe_3N_2) of about 10μm, with high hardness, in order to improve the surface characteristics. The layer and microstructure were characterized with X-ray analysis, optical microscopy, confocal laser scanning and hardness test by microindentation. Tensile, fatigue and creep tests show the influence of microstructure formed on the mechanical properties, the microstructure predominantly bainitic phase showed a better combination of ductility, toughness and fatigue life. After treatment nitrocarburizing was observed the effect of tempering, with a reduction in hardness of substrate and tensile strength. In steel with martensitic microstructure the fatigue life decreases. We observed also a variation in creep behavior due to different microstructures formed. After the plasma treatment, there was a considerable reduction in the rate of creep and an increase in the time required for fracture.*

Keywords*: 4340 steel, microstructural characterization, mechanical properties, plasma nitrocarburizing.*

1. INTRODUCTION

The 4340 steels are used in the aerospace industry for specific applications as in Brazilian satellite launch vehicle and in aircraft landing gear. This steel also is widely used to manufacture others industrial components that require high strength. In order to attend this special uses, a hard work has been carried out to improve properties as strength, toughness, wear, fatigue and corrosion (Bhshan and Gupta, 1991, Ranieri et al, 2009). In most of the cases it is necessary an agreement of heat and superficial treatments.

An improvement on mechanical properties as fatigue life and tensile strength in the AISI 4340 steels has been obtained by heat treatment with phase transformation. Prior researches using the phase transformation process gave rise to the termed as a “dual-phase steel” (Hayami at al, 1975) and subsequently the “multiphase steels” (Souza at al, 2008). These structures were obtained through heat treatment in an intercritical temperature, isothermal or thermomechanical treatment generally used in industry (Abdalla at al, 2007).

The multiphase steel has microstructures with different morphologies and volumetric fractions of its phases, these structures change significantly the materials properties. In this way, different heat treatment gives rise to different properties in agreement to the project (Sakuma at al, 1991). For instance, in order to improve the steel toughness, total or partial changes of the martensitic phase to bainitic or multiphasic structures can be used. Thus, high levels of strength without significant loss of ductility can be kept (Abdalla at al, 2002).

Some works show that martensite, bainite, ferrite and retained austenite played important role (Matsumura at al, 1987) on the material toughness. Due to the TRIP effect (Transformation Induced Plasticity), the austenite phase becomes to martensite during the plastic deformation, contributing with the enhance of ductility and strength stress. Isothermal treatment on a region of bainitic transformation can induce the retained austenite formation on the final steel microstructure (Sakuma at al, 1987).

In recent years, various surface treatment methods have been applied in order to improve the surface characteristics as: severe conditions of load, wear, and the chemical corrosion of the structure surface. Termochemical treatment, as nitriding, nitrocarburizing and carburizing has been used to improve of the superficial properties as wear, friction, and corrosion resistance (Sirin at al, 2008; Podgornik, and Vizintin, 2001). In this process is formed a layer with thin nitride dispersion. This compound increases the superficial hardening and superficial compressive stress, reducing wear and increasing fatigue life (Nicoletto at al, 1996). According to Bell (1991), the fatigue strength of the material can be improved due the nitrocarburing treatmento and it is known that the improvement of fatigue strength mainly benefits from the surface compress residual stress. Recent studies also show that the creep properties can be improved with treatments nitrocarburizing (Abdalla, 2010)

So far, researches have been carried out to give rise to properties improvement by heat treatment and superficial properties by termochemical treatment. There is not any information about superficial treatment influence on mechanical and microstructural properties obtained by heat treatments. In this work results about the nitrocarburizing influence on AISI 4340 steel microstructure under three different heat treatment conditions are shown: austenitized, quenched in oil and tempered; austenitized, quenched in salt bath and cooled in water (isothermic) and annealed. There are analyzed also the influence of the layer formed on the fatigue life, creep, tensile and wear properties and the relationship with each type of microstruture formed. Moreover, compound layers properties formed on the three superficial structures are shown and analyzed.

2. EXPERIMENTAL PROCEDURE

2.1. Material

The material used in this work was the AISI 4340 steel, the chemical composition was show in Table 1.

Table 1: Chemical composition of the elements

Elements	**C**	**S**	**P**	**Si**	**Mn**	**Cr**	**Ni**	**Mo**
4340 (% peso)	0,39	0,0010	0,017	0,26	0,64	0,80	1,82	0,22

The steel was received in hot rolled plates which 3 mm thick. The tensile, fatigue and creep specimens was machined in according with the ASTM technical standards.

2.2. Heat treatment

Three kinds of samples were respectively submitted to a heat treatment: normalized, quenched/tempered and isothermal transformation in controlled salt bath. The details of this treatment are shown in Table-2.

Table 2: Heat treatments

Heat Treatment	*Description*
Normalized	Annealed at 900ºC – (3.6 ks). Cooled in air.
Quenched and Tempered	Austenitized at 900ºC – (1.8 ks). Quenching in oil and tempering at 400ºC – (7.2 ks).
Isothermal Transformation	Austenitized at 900ºC – (1.8 ks). Isothermally Transformed in a salt bath at 320ºC – (0.9 ks) and Cooled in water.

2.3. Nitrocarburization Thermochemical Treatment

The samples were polished with 150, 220, 280, 320, 400, 600 and 1000, 1200 grades emery papers and then ion nitriding treatment was applied at industrial ion nitriding furnace from Metal Plasma Company, which use pulsating direct current glow discharge technology. Specimens were ion nitriding for 3 hours at 500ºC with 75% N_2 – 23.5%H_2 – 1.5% CH_4 as treatment gas.

2.4. Microstructure, microhardness and XRD test

Microhardness by a Vickers microhardness tester were measured by using a weight equal to 50 gr. The surface layer was characterized by means of an X-ray diffractometer using CuKα, X-ray tube (λ=1.5405Aº). The samples were mechanically polished by using alumina slurry and its microstructure was analyzed by optical microscopy and the fracture surface was observed by SEM (Scanning Electron Microscope) in a LEO 1460 VP equipment.

2.5 Mechanical Properties

The mechanical properties were available by three different tests: tensile, axial fatigue and creep. In this work was verified the influence of heat treatment and the compound layer formed by the thermochemical treatment. The tensile tests were made in a EMIC DL 2000 machine, three samples were used for each treatment parameter. The fatigue tests were carried out in a MTS hydraulic machine using a frequency of 25 Hz and a stress ratio of 0.2. The creep tests were conduced at 650 and 750 ºC in a Testing Laboratory of the Technological Institute of Aeronautics (ITA).

3. RESULTS AND DISCUSSION

The Fig.1.a shows, by optical microscopy, a microstructure of the normalized 4340 steel, can see basically a ferritic structure (white) and perlitic (dark). The microstructure of the tempered martensite obtained by quenching and tempering process is shown in the Fig.1.b., by this heat treatment the hardness and tensile strength are improved. The bainitic structure formed in isothermal transformation can be seen in Fig.1.c, is observed a refine in structure and an improvement in the aspect of acicullarity, this structure bettered the properties similarly that the structure with tempered martensite.

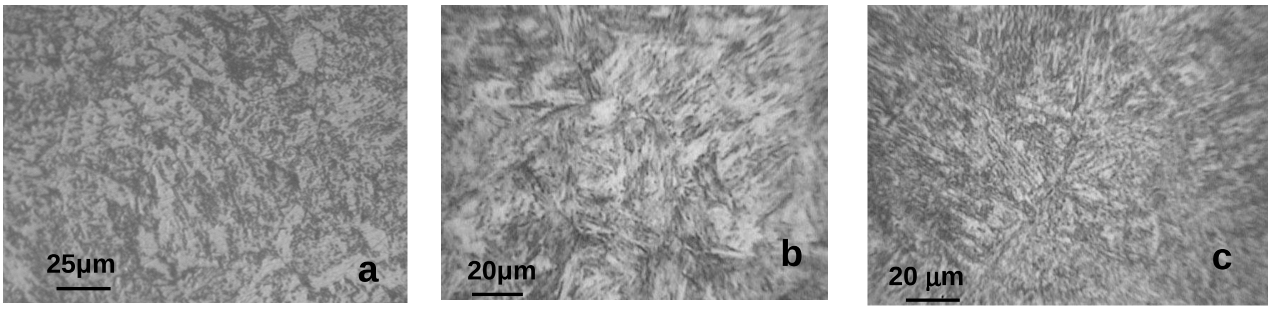

Figure 1. 4340 steel microstructure(MO): (a) Normalized; (b) Quenched and Tempered; (c) Isothermal transformation.

The Fig.2a, b and c show images obtained by confocal laser scanning microscope (LSC) layer of nitrides formed after the plasma nitriding treatment in each of the three microstructural conditions studied, the layer is about 10μm and a very high hardness (~ 900HV). In images of Fig.2, we observe the white layer of nitride on the surface, with small variations in thickness, and near of layer, in the diffuse region, there is a more intense precipitation of nitrides near the grain boundaries, this effect forms a gradient of reduced hardness in the direction of the core substrate.

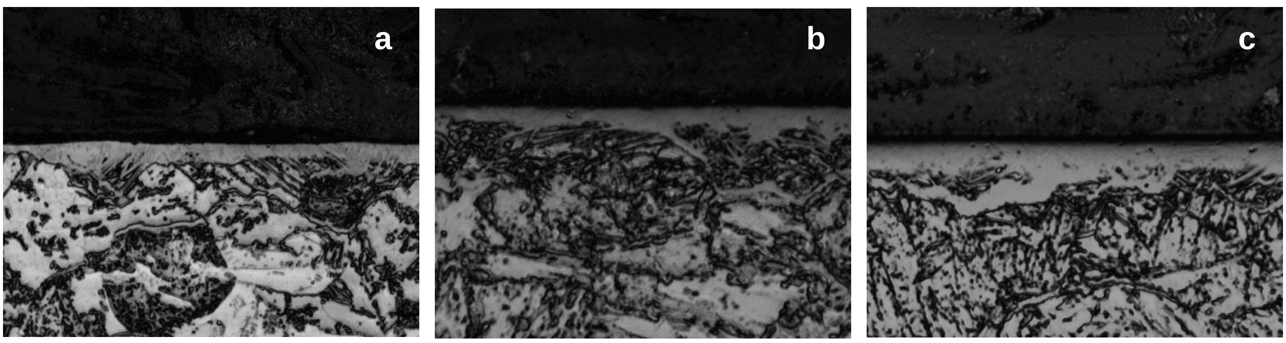

Figure 2. 4340 steel microstructure (MO): (a) Normalized and Nitrocarburized; (b) Quenched and Tempered; (c) Isothermal Transformation.

The microhardness profiles obtained after the ion nitriding on the three heat treatments conditions are shown in Fig.3. The higher hardness value was measured on the quenched and tempered surface steel (945 $HV_{0.05}$). A hardness of 875 HV was measured on the layer for the other heat treatment conditions. The diffuse case depth is around 130 µm. This layer thickness and region of atomic diffusion creates a excelente protector barrier against corrosion (Abdalla et al, 2010).

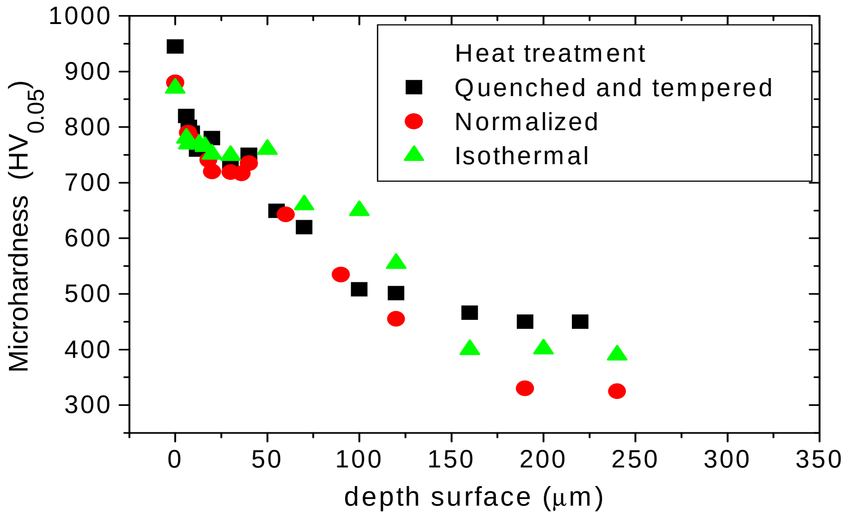

Figure 3. 4340 steel microhardness profile: Quenched/Tempered, Isothermal transformation and Normalized.

There is a substrate hardness reduction after ion nitriding. It happens during the nitrocarburizing process at 500ºC because the steel sample is under another tempering cycle, by this treatment the internal structure of the steel will recover and the phases martensite and bainite will precipitate carbides. The largest of the hardness reduction around 100 HV, occurs during isothermal treatment with bainitic structure as shown in the Table 3. Similar results in quenched and tempered material conditions were obtained by other authors (Lee at al, 1999).

Table 3**:** Substrate hardness fluctuation after ion nitriding.

Substrate Hardness ($HV_{0.05}$)			
Ion Nitriding	**Heat Treatment**		
	Normalizing	Quenching/Tempering	Isothermal
Without Treatment	341	505	498
Nitrocarburizing	330	450	400

The peaks of X-ray diffraction diagram of AISI 4340 steel were identified and their origin from α-Fe structure. The XRD diagram of the nitrocarburized surface on the three conditions of heat treatment reveal ε - [$Fe_{2\text{-}3}N$], γ'-[Fe_4N] and α-Fe phases. On the quenched and tempered condition the proportion among phases ε, γ' and α-Fe was 59%, 29% and 12% respectively. Thus the nitrocarburizing process at 500ºC (during 10.8 ks) gave rise to a compound layer where the ε-phase prevails. Similar results were obtained at the same treatment conditions by Sirin et al (2008). The XRD diagram of the nitrocarburizing specimens on the three heat treatment conditions is shown in Figure 4.

The Tab.4 shows the mechanical properties in tensile and hardness for steel in three heat treatment conditions before and after the plasma thermochemical treatment. It is observed that the permanence at a temperature of 500 ° C in the plasma chamber caused a decrease in hardness of the substrate, however, in despite of the increase in hardness of the layer surface. The yield and strength limits show a reduction too, it occurs due the recover of the structure. Is noticed that occur a reduction in ductility, this phenomenon indicate a probable precipitation in grain boundaries.

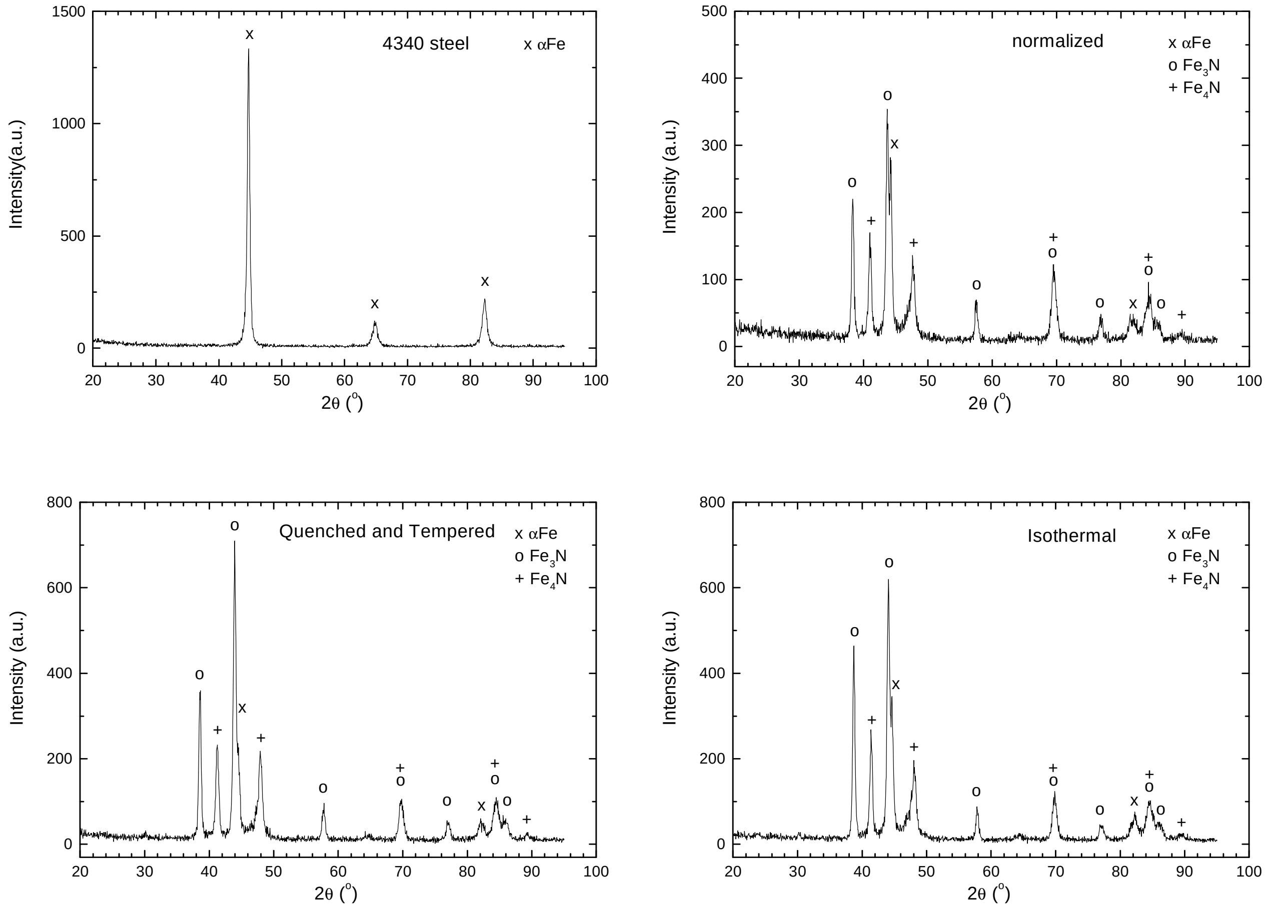

Figure 4 - AISI 4340 XR Diffratometer: Nitrocarburizing on three heat treatment conditions.

Table 4 - Hardness and Tensile Properties of 4340 Steel with Different Microstructures

Heat Treatments and nitrocarburising Plasma (Steel 4340)		Yield (MPa)	Tensile strength (MPa)	Elongation (%)	Substrate hardness ($HV_{0,05}$)
Normalizing	without plasma	*1189±18*	*1297±11*	*11,7±0,9*	*341±8*
	with plasma	*1073±10*	*1113±31*	*10,3±1,1*	*330±7*
Quenching/ Tempering	without plasma	*1597±23*	*1859±47*	*9,4±0,6*	*505±12*
	with plasma	*1128±15*	*1274±29*	*7,9±0,5*	*450±11*
Isothermal	without plasma	*1280±12*	*1448±23*	*10, 9±0,9*	*498±15*
	with plasma	*1138±17*	*1245±19*	*9,8±0,7*	*400±9*

The graphs of Fig.5 **a** and **b** show the influence of microstructure and the plasma treatment on fatigue properties. It is observed from the graph in Fig.5 that after treatment of nitrocarburizing, a reduction in the fatigue performance for the annealed steel, with an increase in fatigue limit for steel with tempered martensite structure and a significant improvement for the steel with bainitic structure. The reduce in hardness an the tensile properties of the annealed steel appears to have affected the fatigue properties. For the steel with structure of martensite, the reduction of tensile properties, affect only the region of low cycle fatigue, with improvements in fatigue limit. The compound layer formed was more beneficial to the steel with bainitic structure and the reduction in tensile properties and hardness did not affect the fatigue performance.

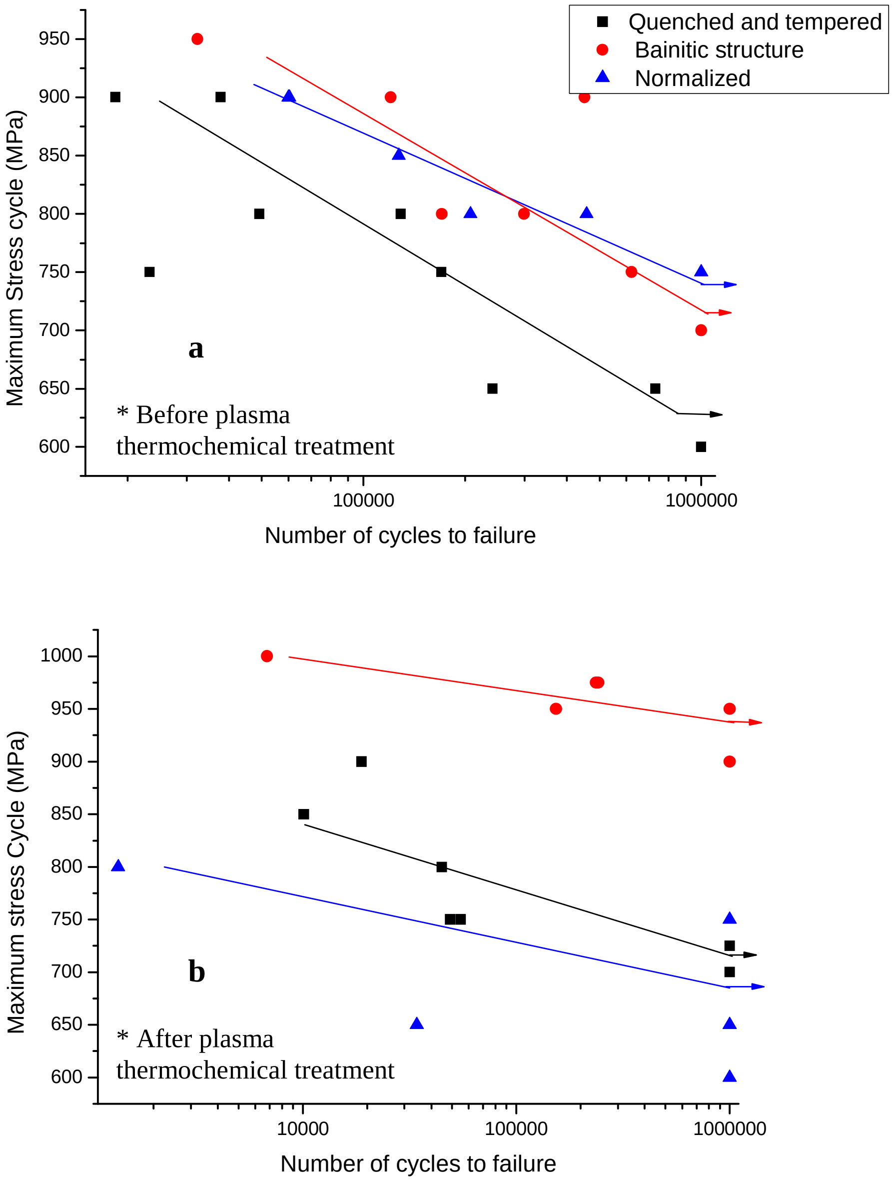

Figure 5 - SN Fatigue Curves for the microstructural conditions studied: a) before plasma thermochemical treatment and b) after application of plasma treatment of nitrocarburizing.

It is observed from the graph in Fig.5 that, after treatment of nitrocarburizing, the reduce in hardness an the tensile properties of the annealed steel appears to have affected the fatigue properties. For the steel with structure of martensite, the reduction of tensile properties, affect only the region of low cycle fatigue, with improvements in fatigue limit. The compound layer formed was more beneficial to the steel with bainitic structure and the reduction in tensile properties and hardness did not affect the fatigue performance.

The results of creep tests show that the microstructures formed show variation in properties. After plasma treatment, a reduction in creep rate and an increased time required for fracture for 4340 steel under the conditions studied. The tab.5 shows that occurrence, for the test temperature of 600ºC and the tensile strength of 200 Mpa. It is observed that for the three microstructural conditions studied, the formation of the nitride layer had a very positive effect to reduce the creep rate and increase the time required for breakage. Generally, the processes of creep fracture are initiated on the surface and the layer formed, delays the onset of fracture and increases the time required for it to occur.

Table 5.Parameters and results of creep tests

AISI 4340 steel	T [ºC]	σ [MPa]	$\dot{\varepsilon}_S$ [1/s]	t_f [s]	ε_f [mm/mm]
Bainitic structure	600	200	0,0876	8388	0,3376
Normalized condition	600	200	0,05919	9240	0,3334
Quenched and Tempered	600	200	0,1265	6721	0,725
Bainitic structure + Nitrocarburizing	600	200	0,04908	12600	0.3195
Normalized condition + Nitrocarburizing	600	200	0,03431	13439	0,3153
Quenched and Tempered + Nitrocarburizing	600	200	0,06162	9299	0,223

4. CONCLUSIONS

- Steel structures have been altered due to heat treatment, tempered martensitic and bainitic structures increased levels for yield and tensile strength , with advantage to the bainitic structure, which shows better ductility;
- The application of plasma thermochemical treatment formed a layer with high hardness (900HV) and thickness of about 10µm and a diffuse layer of around130 micrometers;
- After the plasma treatment the substrate was reduced hardness with lower levels of tensile strength for steel in quenched and tempered condition and to the bainitic condition. In the normalized structure occurs an improvement in strength, probably due to an aging process.
- The plasma treatment applied and the compound layer formed, altered the fatigue curves for the three microstructural conditions, with an interesting and promising result for the bainitic condition, showed better performance in the curve of fatigue, the fatigue limit increased to a value close to 900 MPa.
- The plasma treatment applied shows that this surface layer has influence in creep test, improving the time for fracture and reducing the creep rate.

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6. RESPONSIBILITY NOTICE

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

7. REFERENCES

Abdalla, A. J. ; Carrer, I.R. ; Barbosa, M.J.R.; Moura Neto, C. , 2010. Estudo de Fluência em aços 4340 com diferentes Microestruturas e Tratamento de Carbonitretação a Plasma. Proceedings of Congresso Brasileiro de Engenharia e Ciência dos Materiais, 2010, Campos do Jordão, Brazil.

Abdalla, A. J. ; Moura Neto, C. ; Hashimoto, T. M. ; Pereira, M. S., 2007. Tratamentos Térmicos para a Formação de Bainita em Aços Multifásico de Baixo Carbono. Proceedings of 62º Congresso Internacional Anual da ABM, 2007, Vitória ES, Brazil.

Abdalla, A.J., Hashimoto, T.M., Moura Neto, C., Monteiro, W.A., 2002. Proceedings of 8º Int.Fatigue Congress, v.4, p.2263-2270, Sweden.

Andrade, S.L.; Batista, J.F.; Taiss, J.M.; Rosa, L.K., 2002. Proceedings of 57th Congresso da Associação Brasileira de Metalurgia e Materiais, São Paulo, Brazil.

Bell T. Gaseous and Plasma Nitrocarburizing, 1991. ASM Handbook, vol.4, ASM International, Materials Park, Ohio, p. 425–36.

Bhshan, B., Gupta, B.K., 1991. Handbook of Tribology – Materials, Coatings and Surface Treatments, McGraw-Hill, New York, EUA.

Hayami, J., Furukawa, T., 1975. Microalloying, Vanitech, p.78-87.

Lee, W.S., Su, T.T., 1999. Journal of Materials Processing Technology, v. 87, p.198–206.

Matsumura, O.; Sskuma, Y; Takechi, H., 1987. Transactions ISIJ, v. 27, n. 7, p. 570 – 579.
Nicoletto, G., Tucci, A., Esposito, L., 1996. Wear 197, p. 38-44.
Podgornik, B., Vizintin, J., 2001. Material Science and Engineering A315, p. 28 – 34.
Ranieri, A., Baggio-Scheid, V.H., Suzuki, P.A., Abdalla, A.J., Plasma Nitrocarburizing Treatment Of 4340 Steel. Proceedings of COBEM - 2009 - International Congress of Mechanical Engineering, 2009, v. 01. p. 01-06. Gramado RS, Brazil.
Sakuma, Y., Matsumura, O., Takechi, H., Metallurgical Transaction, v.22A, 1991.
Sakuma, Y.; Matlock, D. K.; Rauss, G. K., Metallurgical Transactions, v.23 A, p. 1221-1232 ,1992.
Sirin, S.Y., Sirin, K., Kaluc, E., Materials Characterization 59, p. 351 - 358, 2008.
Souza, G.A., Elisei, C.C.A., Abdalla, A.J., Hashimoto, T.M., Pereira, Anais do 63ºCongresso Anual da ABM, CDROM, pg.2791-2799, 2008.