# PLASMA NITROCARBURIZING TREATMENT OF 4340 STEEL WITH BAINITIC AND MARTENSITIC STRUCTURES

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***Abstract**. In this work a characterization of the AISI 4340 steel, treated by different heat treatments and by plasma nitrocarburizing process, has been done. Three different heat treatments were used. The steel samples were normalized; austenitized, quenched in oil and tempered; and austenitized, quenched in salt bath and cooled in water (isothermic). After the heat treatments the samples were plasma nitrocarburized at 500 °C for 3h. The samples were characterized by microhardness test, optical microscopy, and XRD. Multiphase structures predominantly bainitic and martensitic with hardness between 500 and 580 HV have been formed by the heat treatments. The nitrocarburizing process has resulted in a compound layer with 10 μm and hardness higher than 900 HV, containing predominantly the* $\varepsilon$ *- [*$Fe_{2\text{-}3}N$*] phase, as well as, in a diffusion layer with a thickness of 130 μm. The results concerning the influence of the plasma treatment on the microstructures of the samples are presented and analyzed.*

**Key words**: Plasma nitrocarburizing, heat treatment, 4340 steel, multiphase structures.

## 1. INTRODUCTION

The structural 4xxx steels series are widely used to manufacture industrial components. Due to economic questions, low alloy steels have been used in applications formerly restricted to special steels as tool steel. In order to attend this special uses, a hard work has been carried out to improve properties as toughness, wear, fatigue, corrosion (Bhshan, and Gupta, 1991) and strength stress. In most of the cases it is necessary an agreement of heat and superficial treatments.

An improvement on toughness, stress and fatigue strength in the 4xxx steels series, mainly the AISI 4140 and AISI 4340 alloy steel, 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 *et al*, 1975) and subsequently the “multiphase steels” (Souza *et al*, 2008). These structures were obtained through heat treatment in an intercritical temperature, isothermal and thermomechanical treatment generally used in industry (Andrade *et al*, 2002). 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 *et 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 *et al*, 2002). Some works show that martensite, bainite, ferrite and retained austenite played important role (Matsumura *et 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 *et al*, 1987).

Improvement of the superficial properties as wear, fatigue, corrosion, friction and load bearing capacity of dynamically loaded components, has been obtained through termochemical treatment, as nitriding, nitrocarburizing and carburizing (Sirin *et a*l, 2008; Podgornik, and Vizintin, 2001) . Atomic Nitrogen in the ion nitriding gets into the metal surface and permeates deeper in the material combining with alloying elements as Al and Cr forming then a thin nitride dispersion. This compound increases the superficial hardening and superficial compressive stress, reducing wear and increasing fatigue life (Nicoletto *et al*, 1996).

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. Moreover, compound layers properties formed on the three superficial structures are shown and analyzed

## 2. EXPERIMENTAL PROCEDURE

The material used in this work was the AISI 4340 steel (chemical composition: 0.39%C - 0.001%S - 0.017% P - 0.26%Si - 0.64%Mn - 0.80%Cr - 1.82%Ni - 0.22%Mo). The steel was received in hot rolled plates which were sectioned to rectangular geometry (10 cm x 15 cm) and 3 mm thick. Three 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-1.

**Table 1:** Heat treatments

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

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. Microhardness series 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.

## 3. RESULTS AND DISCUSSION

The microstructure of the normalized steel (a); normalized and nitorcarburizing (b) are shown in Fig. 1. Basically we can see a ferritic structure (white) and perlitic (dark) that is AISI 4340 feature. Any significant changes can be observed in this microstructure but the grains become more polygonal

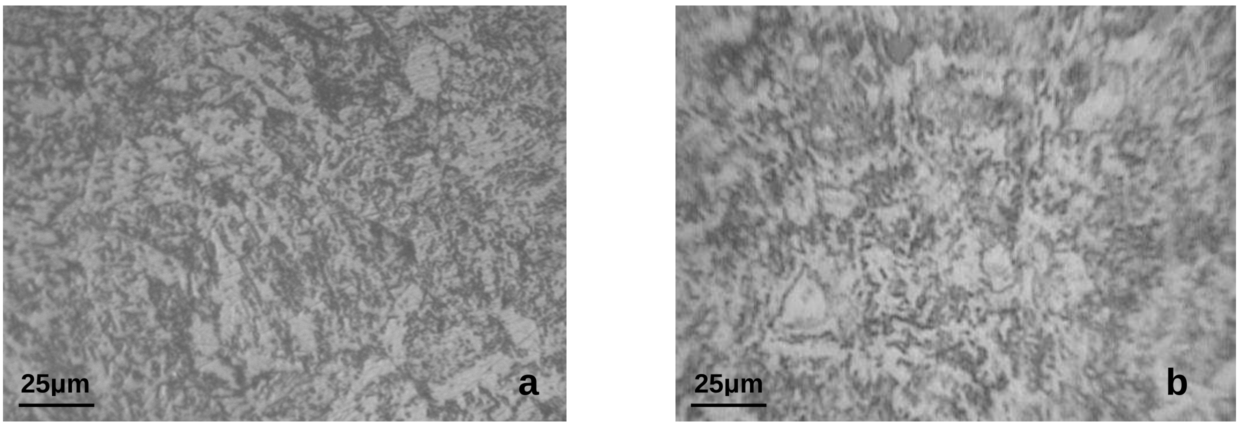


**Figure 1:** AISI 4340 steel microstructure: (a) Normalized; (b) Normalized and Nitrocarburized.

The microstruture of the tempered martensite obtained by quenching and tempering process(a) and íon nitriding(b) is shown in the Figure 2. The tretragonallity of the martensite grains is reduced and gave rise to carbide precipitation. The martensitic needle can not be observed and acicularity were reduced.

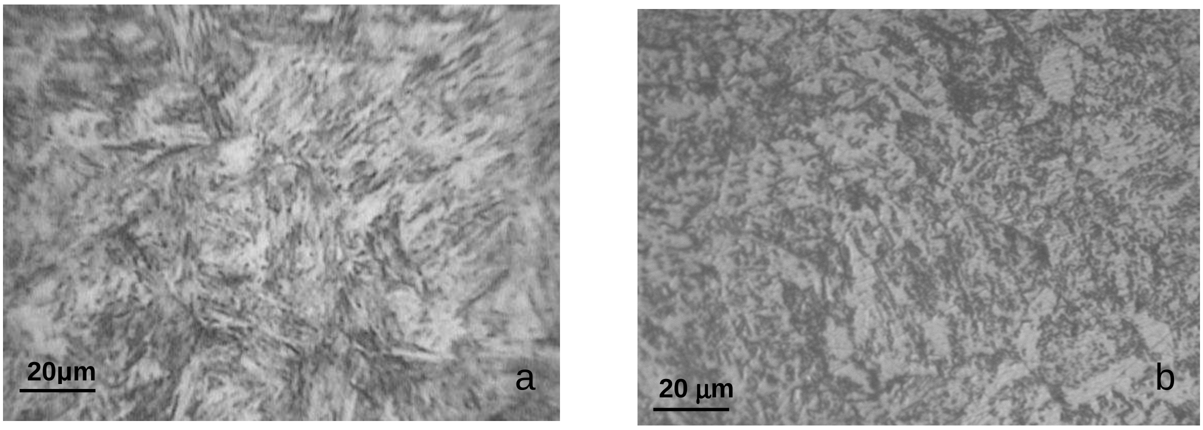


**Figure 2:** AISI 4340 steel microstructure: (a) Quenched and Tempered; (b) Quenched/Tempered and Nitrocarburized.

The bainitic structure formed in isothermal transformation can be seen in Figure 3, before (a) and after (b) the ion nitriding. The ion nitriding at 500ºC gave rise to a tempered of bainitic steel, the atomic diffusion of carbon reduced its acicullarity. The substrate becomes rounded with carbide precipitation.

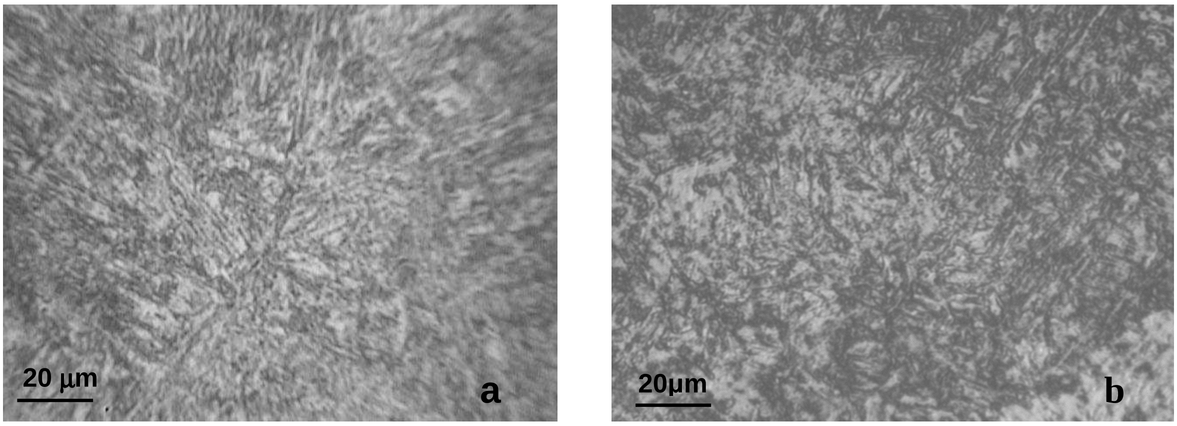


**Figure 3:** AISI 4340 steel microstructure: (a) Isothermal transformation; (b) Isothermal transformation and Nitrocarburizing.

The superficial compound layers on the normalized, isothermal and quenched/Tempered samples are shown in Figure 4. A layer with 10 μm thick is formed on the normalized steel surface. There is a lightly reduced layer thick in the other two heat treatments indicating that the surface of the ferritic-perlitic structure is able to absorb more nitrogen.

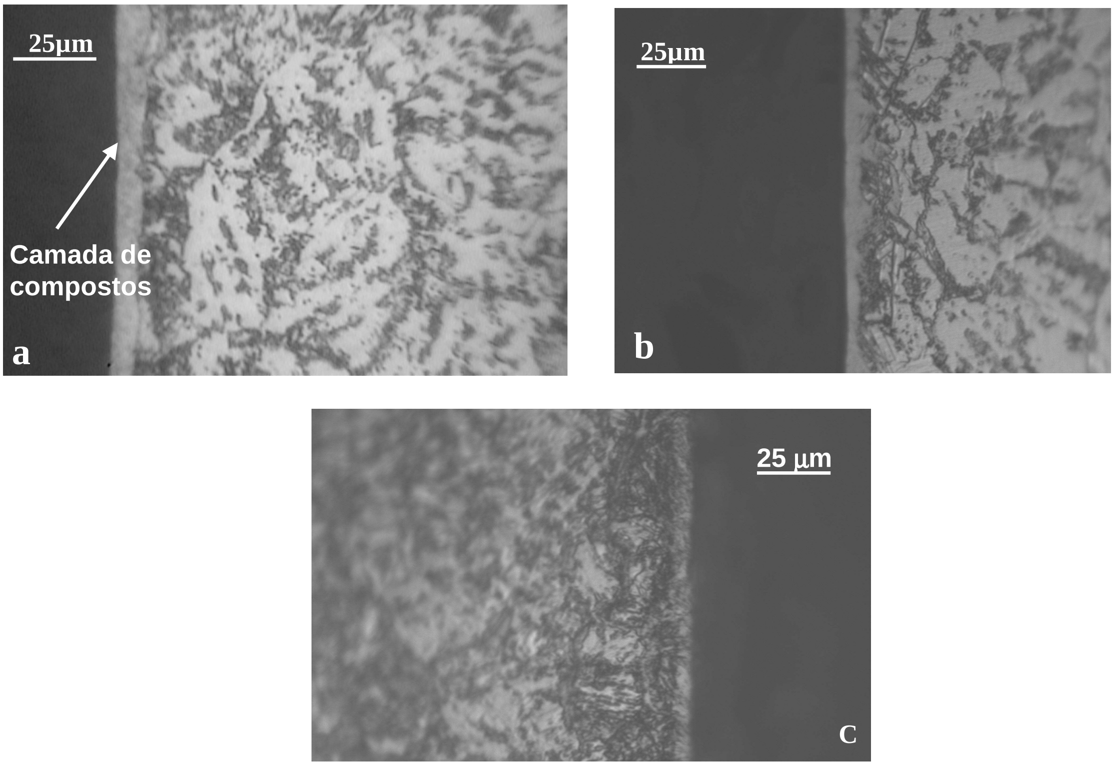


**Figure 4:** AISI 4340 steel microstructure: (a) Normalized and Nitrocarburized; (b) Quenched and Tempered; (c) Isothermal Transformation.

The microhardness profiles obtained after the ion nitriding on the three heat treatments conditions are shown in Figure 5. 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 other heat treatment conditions. The diffuse case depth is around 130 µm. The hardness fluctuation between 25 µm and 50 µm depth shows that a carbon migration on this region happened, induced by the ion nitriding treatment.

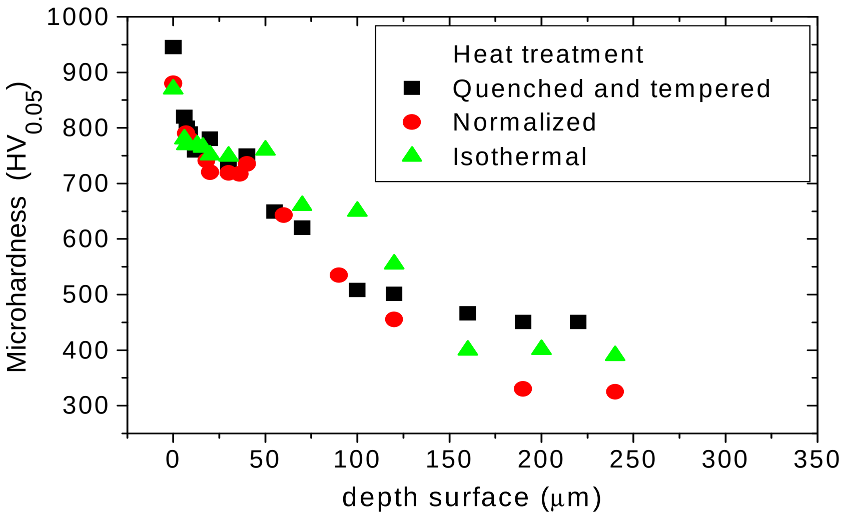


**Figure 5** AISI 4340 steel microhardness profile: Quenched/Tempered, Isothermal transformation and Normalized.

There is a substrate hardness reduction (microhardness measurement beyond 120 μm depth) after ion nitriding. It happens during the nitrocarburizing process at 500ºC because the steel sample is under another tempering cycle. Mostl of the hardness reduction around 100 HV, occurs during isothermal treatment with bainitic structure as shown in the Table 2. Similar results in a quenched and tempered conditions (reduction at 407 HV) were obtained by Lee at all (1999).

**Table 2:** 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 can be seen in Figure 6. 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 but less ε-phase (Sirin, 2008). The XRD diagram of the nitrocarburizing specimens on the three heat treatment conditions is shown in Figure 6.

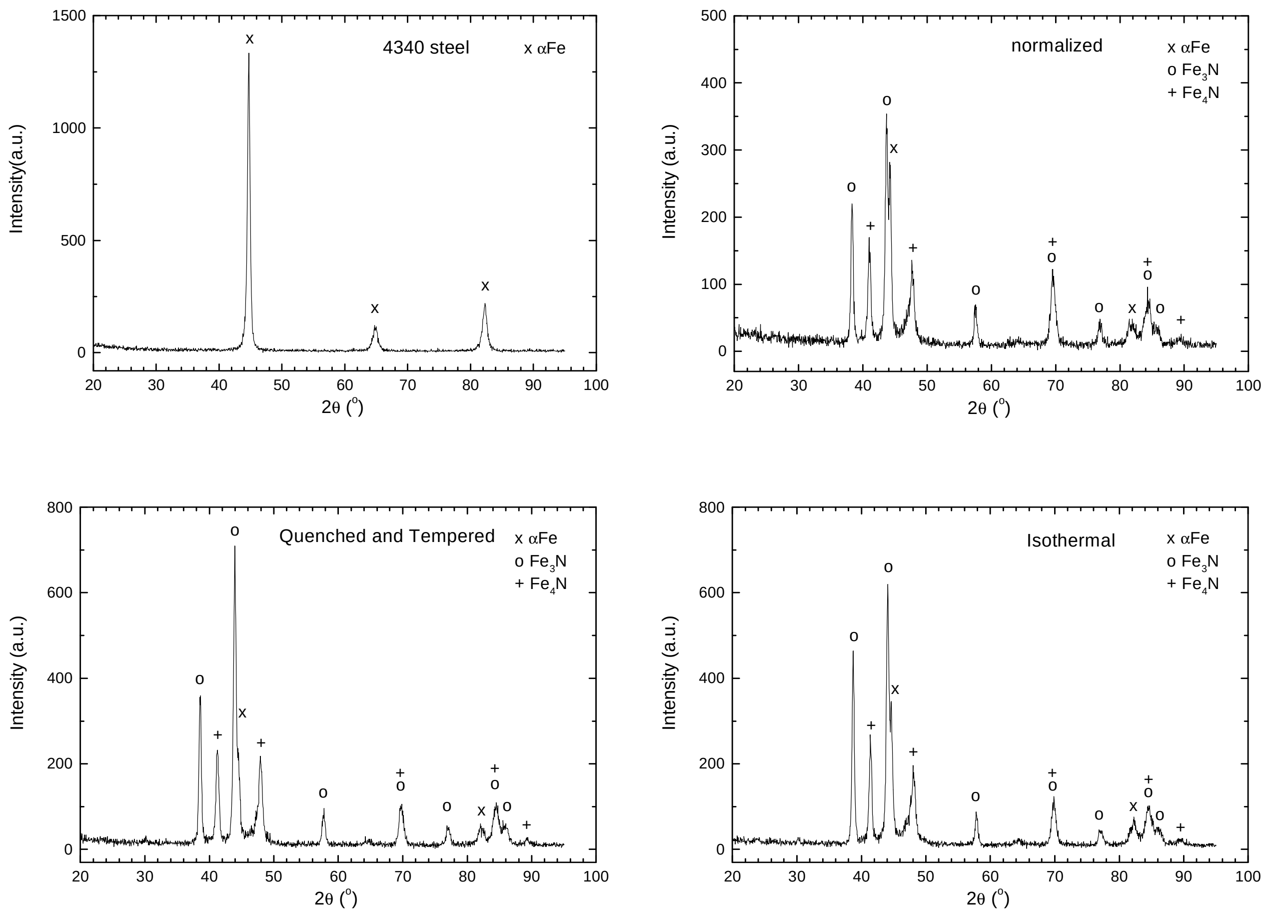


**Figure 6:** AISI 4340 XR Diffratometer: Nitrocarburizing on three heat treatment conditions.

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## 4. CONCLUSIONS

- The Plasma Nitriding was accomplished at 500ºC (10.8 ks) performing changes on the AISI 4340 steel microstructure thermally treated, whereas it acts as another tempering cycle.
- As a result of the microstructural changes introduced by plasma nitriding, it has a substrate hardness reduction. This reduction varies as a function of the steel structure. The less variation, approximately 20 HV, occurred for the ferritic-perlitic structure formed by normalization, whereas the main reduction, around 100 HV, occurred for the bainitic structure formed through isothermal treatment.
- The influence of different structures on the phases, thickness and layer hardness is short. However, the thickest layer (10 μm) is formed on the normalized surface steel, showing that the ferritic-perlitic structure is able to absorb more nitrogen.
- A variation of hardness was observed between 25 μm and 50 μm depth, in the isothermal treatment and it shows the occurrence of a carbon migration into this region induced by plasma nitriding.
- The influence of the different structures on the diffusion layer thickness is negligible showing that the nitrogen diffusion into the substrate is alike in the three researches conditions.

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