

Influence in Fatigue Properties Due a Plasma Nitriding and Laser Carburizing in a Bainitic Steel

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The following work analyzed the changes in the properties of Fatigue in a 300M aeronautical steel after the application of thermochemical treatments of plasma nitriding and laser carburizing. The microstructural characterization of the formed layers and the hardness obtained after the surface treatments were conducted. Thus, a comparison was made between the two treatments to verify which one has better efficacy. It has been observed that the treatment of plasma nitriding improves significantly the fatigue properties of 300 M steel. It was also noted that laser carburizing was not efficient to improve fatigue life.

Keywords: *Surface treatment, 300M steel, Heat treatment, Fatigue Properties.*

1. Introduction

The 300M steel is a commercial low-alloy and ultra-high-strength steel, this steel has been used to replace the AISI/SAE 4340 steel alloy, due to a few adjustments in the chemical composition, which presents good results in terms of heat treatment and welding¹. Differentiating itself by the higher silicon content and higher resistant carbon content, in addition to vanadium and molybdenum (0.37%), thus increasing the yield strength and tensile strength values of the 300M. The improvement in the amount of silicon aims to prevent embrittlement during its heat treatment².

Some properties of 300M steel are similar to those of 4340 steel, except for the deeper hardenability, increased solid solution hardening, and greater resistance to softening at high temperatures. This steel has excellent temperature and ductility, in addition to a tensile strength limit, with heat treatment, it is expected that 300M reached a yield strength greater than 1,400 MPa, good toughness, high resistance to fatigue, good weldability, excellent temperature, and ductility, with tensile strength limit varying between 1860 and 2070³.

Carbon steels have a deficiency in terms of corrosion, so for many applications surface treatments such as nitriding or carburizing are carried out. These treatments can also contribute to improving properties regarding wear resistance and improvement in fatigue life^{3,4}.

The austempering heat treatment, different from conventional tempering, allows the formation of the bainitic structure to have hardness properties and intermediate resistance to the martensitic and pearlitic structures.

In the conventional nitration process, water is introduced onto the steel surface, through the use of a substance rich in water (liquid or gaseous), and by heating at certain temperatures, a hard layer of nitrides is formed. Because it uses lower temperatures than that cementation, nitriding produces less traction and is less likely to cause cracks in

the material. After nitriding, quenching is not necessary to produce hardening in the nitrided layer.

Plasma nitriding differs from traditional nitriding in that it uses a nitrogen gas ionization process during nitriding. Inside a chamber, the air was removed and it was vacuum sealed, nitrogen gas (usually together with hydrogen) is introduced, and a plasma is created by applying a field of electricity and the nitrogen ions are accelerated towards the part (cathode). This bombardment of ions heats the part and cleans the surface, in addition to providing active nitrogen to be absorbed by the steel. In relation to gaseous nitriding, ionic nitriding presents better control of the uniformity and chemical composition of the layer, in addition to causing less distortion in the parts¹.

Laser carburizing occurs by heating the surface below the melting temperature to produce a phase transformation in the solid state without altering the substrate. The body absorbs heat in a short time and when that source is withdrawn, a harder phase is formed in this heated zone. The interaction time of the laser beam with the surface must be sufficient to reach temperatures above 727 °C, without allowing the entry of the liquid phase, and for a sufficient period to cause carbon diffusion. After this time interval, there is a rapid cooling by heat transfer to the volume not affected by heat⁵, thus forming white areas representing the ferrite and austenite phases and in the dark areas martensite, bainite and pearlite¹.

The present work is part of a broader project, which aims to characterize high-strength steels for aerospace applications. The steel used in this work has important aerospace applications, in addition to the engine envelope, it is used in aircraft landing gear and in applications that in general require high mechanical properties⁵. Steels have a wide range of mechanical properties, but, in general, are susceptible to adhesion, one of the purposes of conventional treatments is protection against adhesion. Plasma nitriding or laser carburizing treatments are also intended to improve the tribological properties of steel. This surface treatment would combine the good mechanical properties of steel with the surface properties added to the proposed treatments.

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This study applied important surface treatments in many applications, treatments that can increase surface hardness and modify, including fatigue properties. Fatigue is a very relevant aspect, almost materials exposed to dynamic efforts tend to fail due to fatigue and at resistance levels below the yield strength^{6,7}.

This study gains even greater importance when evaluating structural elements of great responsibility such as aircraft, missile and rocket parts.

2. Materials and Methods

This work is a continuation of a work by Gorges et al.⁸, that applied plasma nitriding and laser carburizing surface treatments on 300M steel. This study repeated the same treatment route to analyze the fatigue behavior, due to it being a very important characteristic for applications.

The material used in this study was 300M steel, the chemical composition is shown in Table 1, the chemical analysis was made by the Chemical Analysis Laboratory of the Materials Division of the IAE / DCTA, according to the ASTM-E-39-84 standards and ASTM-E-350-87. Before receiving the surface treatments, the steel underwent a heating treatment at 900 °C for 30 minutes, then it was cooled to 300 °C, where it remained for 2 hours to form the bainitic structure, then it was cooled in air.

The fatigue specimens were manufactured according to ASTM E 466, and the fatigue tests were conducted in a load of constant amplitude with a load ratio of 0.1 and a frequency of 20 Hz. The tests were carried out in a fatigue machine type MTS 810.23 M, with a load cell of 250 kN.

For microstructural characterization were used techniques of Optical following the process of metallographic preparation (sanding and polishing) and chemical etching in 2% Nital. The hardness of the layer and the hardened region was analyzed with microindentation hardness tests. The microindentations

were performed in a Future-Tech FM 700 Microdurometer, on the IEAv /DCTA. For the measurements of hardness, were used a load of 50gf and an indentation time of 10s.

The plasma nitriding was carried out at 500 °C for 3 hours. This treatment was done in a reactor with a gas mixture of 75% N₂ and 25% H₂. For laser carburizing an initial spray was made on the specimen with graphite and, later, the specimens were irradiated by laser to provide the formation of the hardened layer on the surface. For carburizing, a pulsed CO₂ laser was used, with a wavelength of 10.6 μm, and an output power of 125 W. The application was made keeping the laser at maximum power, with a resolution of 500 dpi (dots per inch) and a scanning speed of 600 mm/s. These parameters were based on previous work⁹⁻¹¹.

3. Results and Discussion

Figure 1a shows the microstructure of the 300 M steel as received, etched with 2% Nital. There are darker regions formed by perlite and lighter regions represent ferrite. To improve the mechanical properties of the steel, a quenching heat treatment with controlled cooling at 300 °C was initially carried out, for the formation of the bainitic structure Figure 1b. The hardness tests showed the hardness increased 300 HV of the ferritic/perlitic structure to about 500 HV for the bainitic structure, showing the effectiveness of the applied heat treatment. The increase is related to the type of microconstituent, as bainite is harder than ferrite or perlite.

After plasma nitriding, there was a significant increase in the surface hardness of the specimens, increasing the hardness to about 800 HV. Figure 2a shows the image obtained by MO that allows the visualization of the nitriding layer. Figure 2b shows the carburizing layer by the laser process, it does not have a hardness as high as the nitriding, but it has a greater thickness, it is hardness reached a value around 600 HV.

Table 1. Chemical composition of 300M steel (wt %).

Alloy elements	C	S	P	Si	Mn	Cr	Ni	Mo	Al	V	Cu
Wt %	0.39	0.0005	0.009	1.78	0.76	0.76	1.69	0.4	0.003	0.08	0.14

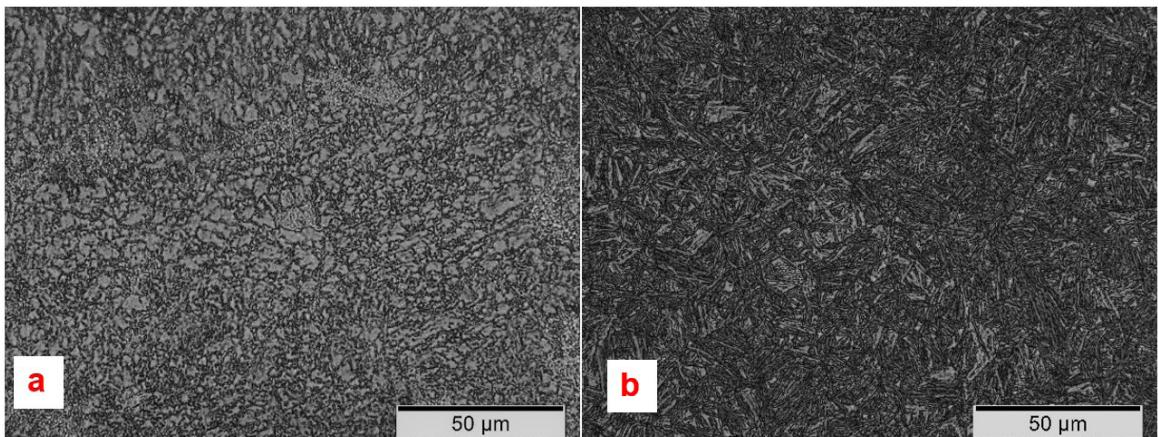


Figure 1. Microstructure of the 300 M steel: (a) before the heat treatment (ferritic-pearlitic structure), and (b) after the isothermal treatment (bainitic).

The white layer of nitriding steel has a white layer with approximately $3\ \mu\text{m}$ (CZ - Coated Zone), below, the diffuse layer (CD) is observed, this zone is deeper, reaching about $60\ \mu\text{m}$. The white layer has a high nitrogen content and significantly increases the hardness observed in the region. In the diffuse layer, as it penetrates toward the interior of the material, the nitrogen content decreases, giving a decreasing gradient in the hardness value. These changes in hardness are related to the formation of iron nitrides, which have a great influence on the mechanical properties of steel and are hard and fragile¹.

Due to the laser surface treatment applied, identified in Figure 2b, it is noted that there was the formation of a white layer with a high concentration of carbon and iron, coated zone (CZ). Below this layer, there is the region affected by the heat produced by the laser (HAZ, with about $60\ \mu\text{m}$), in which the occurrence of localized partial quenching is observed¹¹.

To evaluate the hardness of the treated surface and the region close to the surface, a hardness profile was performed on the surface, to show how the hardness varies until reaching

the substrate hardness value. Figures 3a and 3b shows the hardness profile for the two treatment conditions studied.

It is noted that the plasma nitriding has a less thick layer, but with a high hardness, superior to the hardness presented by the laser carburized treatment. The depth of the diffuse layer, formed due to plasma treatment, was also shown to be greater (about $60\ \mu\text{m}$), while the HAZ layer, formed by the laser process was reduced, to around $40\ \mu\text{m}$. This phenomenon occurs due to the high scanning speed of the laser used ($600\ \text{mm/s}$), while in the plasma process, the treatment is slower, as it depends on the nitrogen diffusion speed in the steel, the treatment time was 3 hours, at a temperature of $500\ ^\circ\text{C}$.

In a complementary study, Gorges et al.⁸ studied the difference between the two scanning speeds of laser carbide, observing only an increase in hardness for regions close to the surface in the process that used a laser speed of $800\ \text{mm/s}$. In this work, there is a more noticeable decreasing gradient of surface hardness towards the center of the sample, this effect is more accentuated due to the greater thickness of the sample, which facilitates the removal of heat and the rapid cooling of the same, favoring the formation of harder on steel¹⁰.

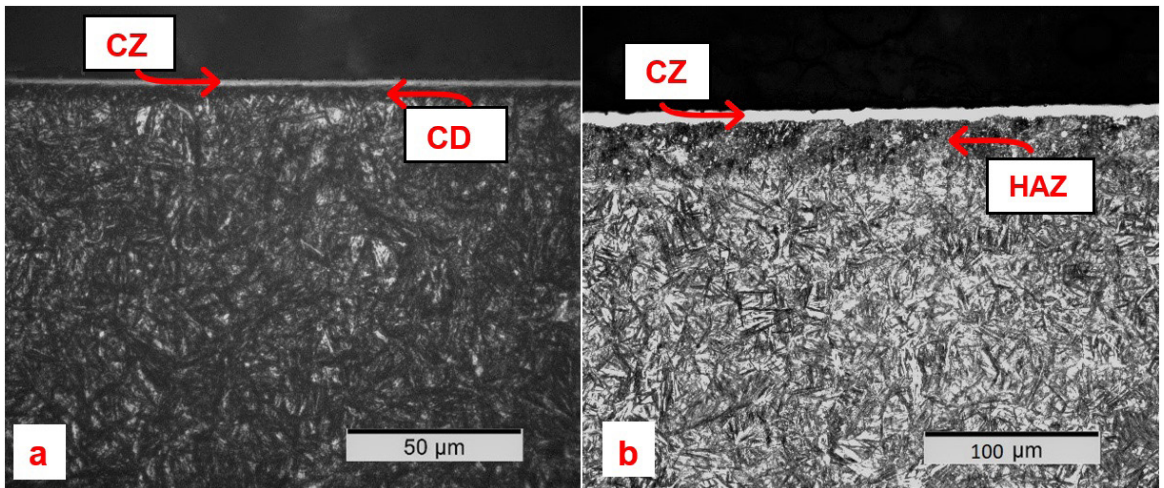


Figure 2. Microstructure of 300 M steel surface protection layers: (a) after plasma nitriding, (b) after laser carburizing. CZ - Coated zone; DI - Atomic diffusion layer; HAZ - Heat affected zone.

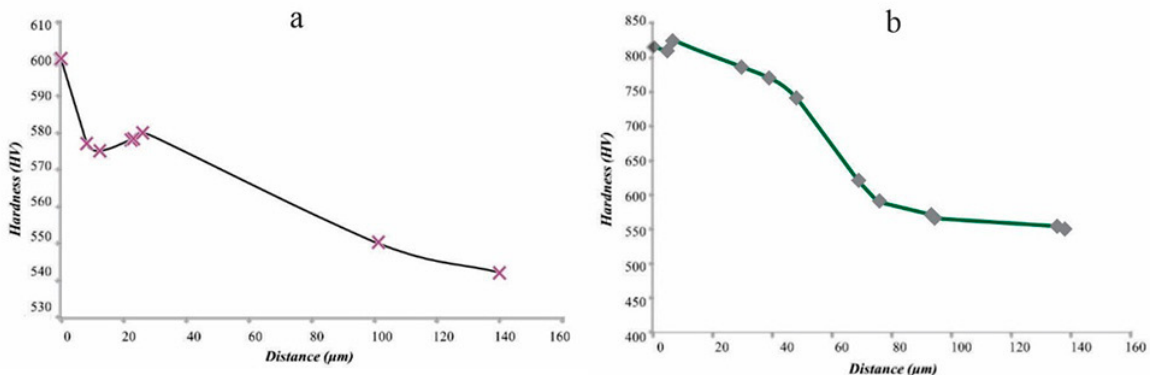


Figure 3. 300 M steel hardness profile: (a) after laser carburizing, (b) after plasma nitriding.

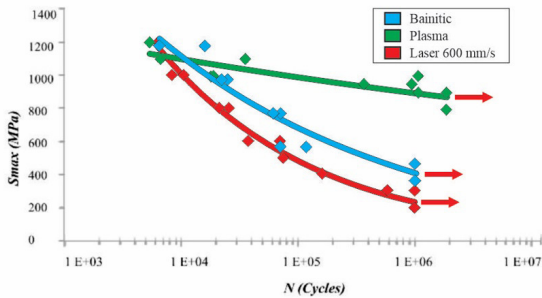


Figure 4. Fatigue S-N curve, for 300 M steel without surface treatment, with laser carburizing treatment, and with plasma nitriding treatment.

In this work, we analyzed how these treatments affect fatigue properties. The curves shown in Figure 4 show how this property has been changed due to surface treatments. Comparing the curves of bainitic steel without surface treatment with laser carbide, it is noted that there is a small loss in fatigue life for higher stress levels (between 1,000 and 1,200 MPa), however, when analyzing the limit of fatigue, for 10^6 cycles, and life for lower stress levels, it is noted that the response of only bainitic steel was better⁸. This reduction in fatigue life is mainly associated with the lower hardness observed on the surface, which, for this type of test, is quite relevant.

When carrying out the same type of comparison, with the 300 M steel that was plasma nitrided, it is noted that the treatment provided a large increase in fatigue life, mainly for the lowest stress levels. The fatigue limit, considered in this work for 10^6 cycles, was increased by about 200 MPa for the laser carburizing steel, about 400 MPa for bainitic steel only, and about 800 MPa for plasma-nitrided steel. This elevation is very significant and allows a significant increase in fatigue life. This fact is associated with the high hardness of the nitriding layer produced, and it is known that this treatment introduces compressive stresses on the surface and this effect is beneficial for fatigue life. This phenomenon delays the nucleation of the fatigue crack, increasing the useful life of the component.

4. Conclusions

The results obtained in the present study showed that the heat treatment applied was effective in changing the microstructure and the mechanical properties of the 300M steel and that is possible to choose the better heat treatment applied to conform the use desired to this steel.

In the plasma nitriding treatment, the white layer formed was of high hardness (about 800 HV) with a thickness of about 60 μm . This hardness had a decreasing gradient until it reached the substratum hardness. In the Laser carburizing

treatment, the hardness was less (600 HV) and had rapid decreases that are not good for fatigue properties.

In the fatigue tests, some changes were observed, considering the fatigue life in a level of 10^6 cycles. Among them, it was observed that the laser treatment produced a reduction in the fatigue performance of the 300M steel to levels below 400 MPa, this reduction becomes even more pronounced when compared with the plasma nitriding treatment, which produced a beneficial effect, significantly raising the fatigue performance of the steel to levels above 800 MPa. By the way, we can conclude that the laser treatment obtained the worst fatigue performance of the three conditions studied, because even the steel only in the condition with the bainitic structure, without heat treatment of the surface, obtained results higher than the laser treatment.

5. References

1. Abdalla AJ, Baggio-Scheid VH. Tratamentos termoquímicos a plasma em aços carbono. *Revista Corrosão e Proteção de Materiais*. 2006;25:92-6.
2. Poole SW, Franklin JE. High-strength structural and high-strength low-alloy steels. In: ASM International Handbook Committee, editor. ASM handbook: Properties and selection: irons, steels, and high-performance alloys. Materials Park, OH: ASM International; 1990. v. 1, p. 389-423.
3. Abdalla AJ, Baggio-Scheid VH, Baptist ACR, Barbosa MJR. Improvement in the properties of a low carbon steel treated thermochemically to plasma. In: VIII CIBIM. Cusco: 8^o Congresso Iberoamericano de Ingeniería Mecánica - Memoria Técnica. Proceedings. 2007. Rio de Janeiro: ABCM.
4. Anazawa RM, Abdalla AJ, Hashimoto TM, Pereira MS. Estudo das Propriedades Mecânicas do aço 300M devido à utilização de retífica após tratamentos térmicos isotérmicos e intercríticos. In: 6th Brazilian Conference On Manufacturing Engineering. Proceedings. Caxias do Sul: COBEF; 2011.
5. Abdalla AJ, Hashimoto TM, Pereira MS, Anazawa RM. Formação da fase bainítica em aços de baixo carbono. *Revista Brasileira de Vácuo*. 2006;25:175-81.
6. Souza RC. Study of ABNT 4340 steel fatigue behavior coated with tungsten carbide by the HVOF/HP system [dissertation]. Guaratinguetá: FEG/UNESP; 1998.
7. Yánes TR, Houbaert Y, Mertens A. Characterization of TRIP-assisted multiphase steel surface topography by atomic force microscopy. *Mater Charact*. 2001;47:93-104.
8. Gorges AGS, Vasconcelos G, Scheid VHB, Abdalla AJ. Microstructural characterization of 300 M steel thermochemically treated by plasma and laser. São José dos Campos: IEAV - SCTI; 2016.
9. Cardoso ASM, Abdalla AJ, Baptista CARP, Lima MSF. Comparison of high cycle fatigue in 4340 and 300M steel welded with fiber laser. *Adv Mat Res*. 2014;891-892:1507-12.
10. Cardoso ASM. Mechanical properties of aeronautical steels heat treated and thermochemically plasma after laser welding [thesis]. São José dos Campos: Instituto Tecnológico de Aeronáutica; 2015.
11. Dian GH. A Influência da deformação plástica sobre a estabilidade mecânica da austenita retida em aços 300M. São José dos Campos: PIBIC; 2006. 33 p.