

Changing in Fatigue Life of 300 M Bainitic Steel After Laser Carburizing and Plasma Nitriding

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Abstract. In this work 300M steel samples is used. This high-strength steel is used in aeronautic and aerospace industry and other structural applications. Initially the 300 M steel sample was submitted to a heat treatment to obtain a bainitic structure. It was heated at 850 °C for 30 minutes and after that, cooled at 300 °C for 60 minutes. Afterwards two types of surface treatments have been employed: (a) using low-power laser CO₂ (125 W) for introducing carbon into the surface and (b) plasma nitriding at a temperature of 500 °C for 3 hours. After surface treatment, the metallographic preparation was carried out and the observations with optical and electronic microscopy have been made. The analysis of the coating showed an increase in the hardness of layer formed on the surface, mainly, among the nitriding layers. The mechanical properties were analyzed using tensile and fatigue tests. The results showed that the mechanical properties in tensile tests were strongly affected by the bainitic microstructure. The steel that received the nitriding surface by plasma treatment showed better fatigue behavior. The results are very promising because the layer formed on steel surface, in addition to improving the fatigue life, still improves protection against corrosion and wear.

1 Introduction

The 300 M is an important steel grade developed by 4340 steel to improve the tensile strength and toughness of the steel. This proposal was reached with the increased of percentage of silicon and introduction of vanadium[1]. This steel is used in aircraft landing gear, rocket motor envelopes, junctions of some parts of satellite launch vehicles and other structural purposes in the automotive and equipment industries [2].

Among the possible structures of 300 M steel, the formation of bainite is one of the most indicated ways to improve the toughness of steel. In this work, a heat treatment was carried out at 300 °C leading to formation of a bainitic structure. This microstructure improved the yield and tensile strength [3,4].

The good properties of the bainitic structure result from the fine or ultra fine grains of the bainitic ferrite with fine uniformly distributed secondary phase structures. This fact has led to a growing industrial demand for the use of bainitic steels in recent decades [5,6].

When working with steels it is always important to prevent corrosion and wear. In this way, surface thermochemical treatments contribute to the formation of a hard layer and a corrosion resistant sample. In this research two types of treatments were used: plasma nitriding and laser carburizing. These processes can still

improve fatigue life because it hinders the crack initiation and delays its propagation, due to residual compression stresses introduced into the surface [7-10].

In the last two decades there has been a lot of progress in the technology used in surface treatment processes. Among these developments are the plasma nitriding method in vacuum oven and ion implantation. Another important process is associated to the use of laser, such as laser carburizing [11-13].

In this work, the changes in the mechanical properties of tensile, fatigue and hardness of a 300 M steel were evaluated. These properties were evaluated before and after the steel passed through heat treatment to form a bainitic structure and after receiving the surface treatments of plasma nitriding and laser carburizing.

2 Test methods

The 300 M steel for test is taken from the shapes with 3mm; the chemical composition is shown in Tab.1.

Table 1. Chemical composition of 300 M steel in wt%

Elements	C	Si	Mn	P	S	Ni
% Weight	0,39	1,83	0,82	0,008	0,006	1,68
Elements	Cr	Mo	Cu	Al	Ti	V
% Weight	0,77	0,39	0,14	0,036	0,0037	0,08

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2.1 Specimens and heat treatment

Tensile specimens were made according to ASTM E8M and fatigue specimens in according to ASTM E466. These were initially subjected to the heat treatment to obtain the bainitic structure by an isothermal treatment. This was done from the austenitization of the specimens at 850 °C for 30 minutes and isothermal cooling in a salt bath at 300 °C for 60 minutes.

The specimens of the 300M steel were divided into four sets: (a) annealed steel - A, (b) steel with bainitic structure - B, (c) bainitic structure with plasma nitriding surface treatment - BN and (d) bainitic structure with surface treatment of laser carburizing - BC.

The samples in each condition were metallographically prepared and etched with Nital 2% to characterization by optical or scanning electron microscopy.

2.2 Surface treatments

2.2.1 Laser Carburizing

After the isothermal treatment the samples were ground and sanded to 1000 mesh, washed and dried. The coating with carbon black was applied by spraying and irradiated via low power CO₂ laser. A carbon black solution used was prepared with 10 g of carbon black and 0.1 g of carboxymethylcellulose (CMC) in 100 ml of ethanol.

The laser used was a CO₂ laser with wavelength of 10.6 μm, output power equal to 125W, diameter of the laser beam 300μm (brand: Synrad and model: J48-5W), and nitrogen flow to act as a protector in the irradiated layer, preventing oxidation [14-15].

The application of the CO₂ laser has as parameters: resolution 500 dpi (dots per inch), 125W power and velocity of 600 mm/s. The image of the steel after coating with carbon black and application of CO₂ laser is showing in Fig.1. This treatment was applied in the useful area of the specimens for fatigue tests.

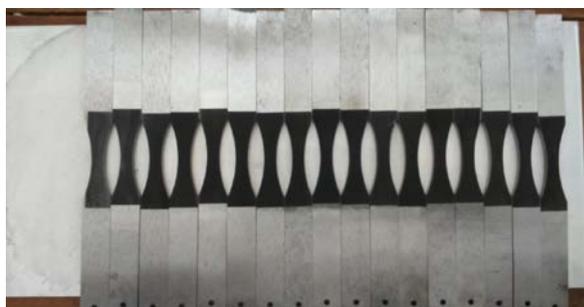


Fig. 1. Fatigue specimens with laser carburizing (black).

2.2.2 Plasma nitriding

The plasma nitriding treatment took place at a temperature of 500 ° C for 3 hours. The reactor employed for sample used a dc- pulsed source of approximately 370 V. The samples were treated in a gas

environment containing 75% N₂ and 25% H₂, the pressure was kept constant at 3 mbar.

2.3 X-ray diffraction

X-ray diffraction analysis was performed on the surfaces of the samples studied. These analyzes were performed on an equipment using copper tube with graphite monochromator. A voltage of 40kV and a current of 30mA were used. The measurements were made by sweeping the samples from 2θ = 20° to 95°, with angular pitch of 0.05° and counting time of 1s per step.

2.4 Mechanical Properties

For tensile and fatigue tests an MTS 810.23M machine was used, with a load cell with a capacity of 250 kN. Three specimens per condition were used in the tensile tests. The tensile tests have been performed at a speed of 1mm/min. For the fatigue tests, the load ratio was 0.1 and the frequency of 25 Hz was used.

Vickers hardness measurements were performed by microindentation with the objective of obtaining the microhardness profile of the samples from the surface towards the substrate, the load used was 50 gf for a time of 10 s.

3 Results and Discussion

The micrographs of steel 300M without the application of heat treatment (Condition A) are shown in Fig.2. The microstructure shows the typical formation of perlite with ferrite (gray) and cementite in lamellar form (white).

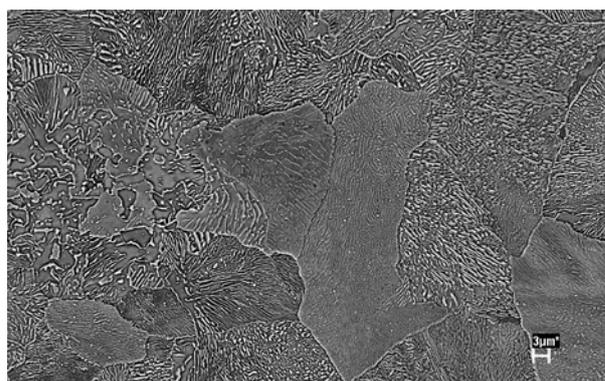


Fig. 2. SEM show 300M steel without heat treatment (annealed steel). Etching: nital 2%. Condition A.

The micrographs of steel after the heat treatment to form a bainitic structure (Condition B) are shown in Fig.3. Besides the bainitic structure, small portions of other phases such as martensite (M) or ferrite (F) are present.

The applied heat treatment produced the formation of a predominant bainitic structure, leading to an increase in the ultimate strength (σ_u) and yield strength (σ_y) and a reduction in ductility as measured by elongation.

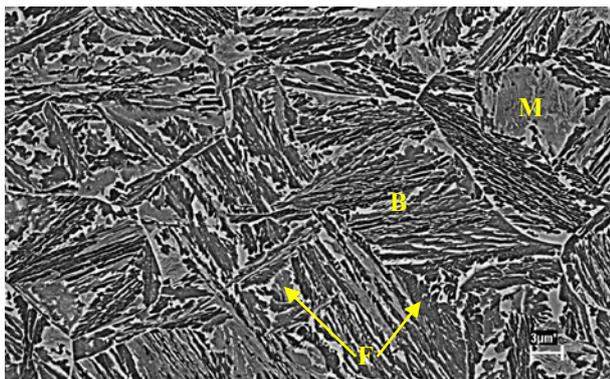


Fig. 3. SEM - shows 300M steel with heat treatment (bainitic structure). Etching: nital 2%. Condition B. B=bainite; M=martensite, F= Ferrite.

In table 2 the results of the tensile tests for both conditions: bainitic (B) – formed by isothermal treatment and annealed condition (A) – as received material are shown.

Table 2. Mechanical properties of 300M steel.

Heat Treatment	Structure	σ_y (MPa)	σ_u (MPa)	Elongation (%)
Annealed Cond. A	Perlite	951±12	1496±25	16,2±1,8
Isothermal Cond. B	bainite	1067±11	1615±25	10,0±0,9

After de laser carburizing a thin white layer about 20 μm thick was formed. Just below it another layer was formed where heating occurred due to the application of the laser (heat affected zone - HAZ) of about 60 μm. In Fig.4 a white layer with a high concentration of carbon and iron on the surface is shown. Below is the heat affected zone (HAZ), on which the occurrence of localized partial quenching is observed. Below these regions lies the base metal composed mainly of bainite.

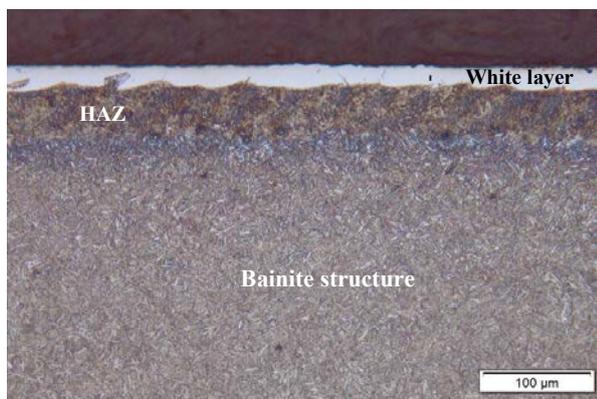


Fig. 4 - Optical Microscopy: Typical zones formed in surface after laser treatment.

It was observed that the hardness of the white layer increased to about 596 ± 21 HV and the HAZ remained around 464 ± 39 HV, similar to that found in the bainitic structure 476 ± 28 HV.

In Fig.5, a micrograph of 300 M steel with bainitic structure that received surface treatment of plasma nitriding is shown. The formation of a thin layer of nitrides on the steel surface, about 3 mm thick, can be noticed. This layer has a high hardness, with 815 ± 25 HV. This increase in hardness occurred due to the formation of hard nitrides of two types, $Fe_{2.3}N$ and Fe_4N .

The hardness measurements found for the various regions of the steel subjected to surface treatments can be seen in Table 2. Below the white layer, the diffusion of the nitrogen atoms occurs, narrow visible in the image, not withstanding detected by the increase of hardness. This diffuse bed of nitrogen reaches about 80 μm. In this region a gradient decreases in hardness from the nitride layer (white) until the substrate hardness is found at about 80 μm from the surface. In this region the average hardness value was 728 ± 74 HV.

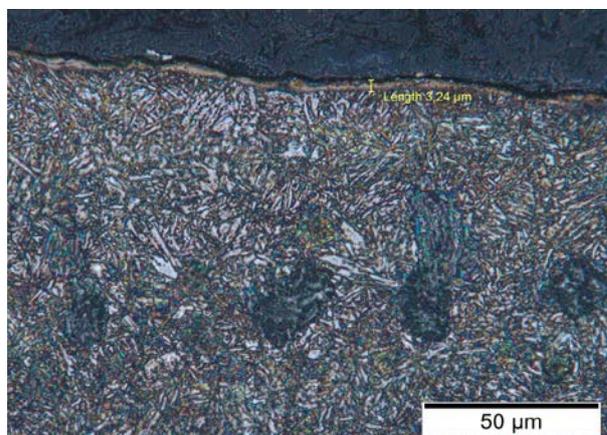


Fig. 5 - Optical Microscopy: Typical zones formed on surface after plasma nitriding treatment.

In Table 2, it is shown that the base material had little influence on hardness due to surface treatment, the significant change occurred only due to the heat treatment for the formation of the bainite structure. The major change in surface hardness occurred due to the treatment with plasma nitriding.

Table 2. Hardness values after surface treatments.

	With surface treatment		Without surface treatment	
	Laser Carburizing (HV)	Plasma nitriding (HV)	As received (HV)	Bainitic Structure (HV)
White layer	596±21	815 ±25	287±9	473 ±12
HAZ	464±39	728±74		
Base material	476±28	489 ±18		

In Fig.6 the X-ray diffraction for the three conditions studied in this work is shown: 300 M steel with bainitic structure without surface treatment and steel with surface treatment with plasma nitriding and laser carburizing.

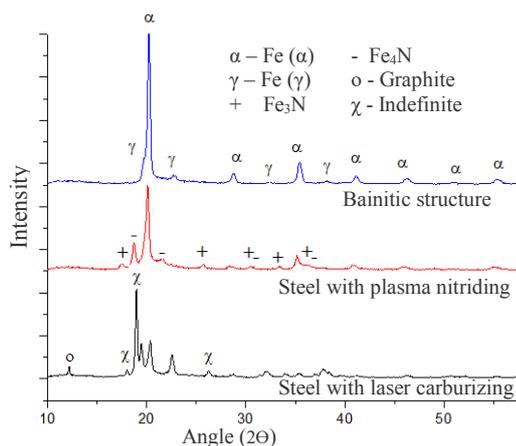


Fig.6 - X-ray diffraction on the surface of the steel for the conditions: bainitic structure, plasma nitriding and laser carburizing.

The mechanical properties observed in tensile are shown in table 3. After the heat treatment in the material as received, the microstructure was transformed from ferritic-pearlite to bainitic, increasing the value of the yield and ultimate strength. Surface treatments did not produce significant changes on the mechanical properties of the steel, however, a reduction in the ductility due to the treatment with plasma nitriding was observed.

Table 3 - Mechanical Properties

Heat treatment	Yield Strength (MPa)	Ultimate Strength (MPa)	Elongation (%)
As received	843 ±15	973 ±0,2	16 ±1,8
Bainitic	913 ±13	1364 ±16	13 ±0,6
Laser carburizing	907 ±23	1353 ±42	12 ±0,9
Plasma nitriding	951 ±23	1356 ±31	09 ±0,3

In Fig. 7, the three S-N fatigue curves for the studied steels are shown: steel with bainitic structure, with laser carburizing and plasma nitriding treatment. It is noticed that there is a fatigue life increase for the steel that was nitrided and a reduction in the fatigue performance for the carburizing steel. One of the factors that influenced the reduction of the life of the carbonated steel was the increase on the surface roughness. In the bainitic and carburizing conditions the roughness values were similar: Ra (0.16 ± 0.04) and Rz (1.09 ± 0.28). For the carburizing condition, due to laser irradiation, these values increased to Ra (0.36 ± 0.06) and Rz (3.41 ± 0.69). The surface of the carburizing steel was also more irregular, with the formation of several phases due to heating and a region of low surface hardness due to the formation of graphite. On the other hand, on the surface of the plasma nitrided steel there was a hard layer and a deeper atomic diffusion zone that contributed to delay the start of fatigue cracking. The tests were interrupted by reaching the mark of 1,000,000 cycles (run out).

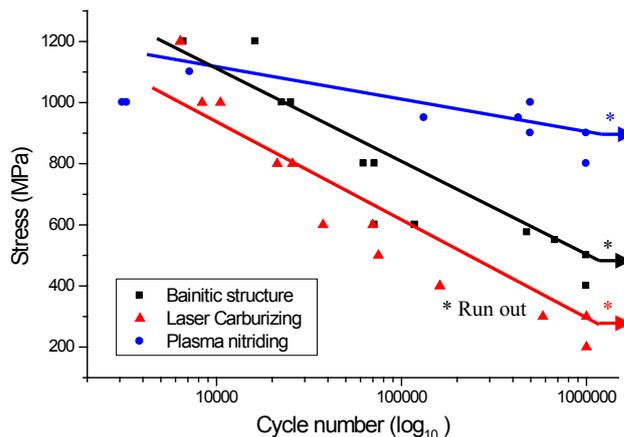


Fig.7 - Fatigue curves (S-N) for the conditions: 300 M Steel with bainitic structure and steel with surface treatment of plasma nitriding and laser carburizing.

4 Conclusions

The plasma nitriding of the 300 M steel with bainitic structure provided the formation of a homogeneous layer with high hardness, contributing to improve fatigue performance for a high number of cycles.

The results of the tensile tests were similar for all the studied conditions; there was no significant change in the yield or ultimate strength. There was a reduction in ductility as measured by elongation to the nitrided plasma condition.

The fatigue performance for the steel subjected to the laser carburizing treatment was inferior to the steel with bainitic structure. The hardness of the carburizing region was similar to that observed in the bainitic structure and did not contribute to delay the appearance of surface microcracks. There was also an increase in surface roughness, a factor that contributed to worsen performance in fatigue.

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