

# Investigation of the Nitrided Layers in Ductile Iron

Casteletti, L. C.<sup>1</sup>, Arnoni, E. A. B.<sup>2</sup>, Nucci, R.<sup>2</sup>, Soufen, C. A.<sup>3</sup>

<sup>1</sup> Escola de Engenharia de São Carlos - Departamento de Engenharia de Materiais, Aeronáutica e Automobilística - EESC-USP - Av. Trabalhador São-carlense, 400 - Centro - São Carlos - SP - Brasil - CEP: 13566-590, [castelet@sc.usp.br](mailto:castelet@sc.usp.br); <sup>2</sup> OGRAMAC Metalização; <sup>3</sup> UNESP-Bauru

## Abstract

The ductile cast iron has been presenting an accentuated growth of production due to its interesting mechanical properties and economic advantages of production, in comparison to the steel or other ferrous alloys. In this work, samples of three compositions of this material in the as-melted state and samples austenitized at 900 °C and austempered for 320 °C in melted salts, were plasma nitrided for 5 hours at 500 °C using continuous current and atmosphere constituted of the 80% H<sub>2</sub> - 20% N<sub>2</sub> gaseous moisture. The austempered samples present the highest hardness values of nitrided layers and the best resistance to wear. In the case of pearlitic matrix, the nitrogen diffusion and the consequent nitrides formation will happen in the ferrite lamellas. Such diffusion will be associated to the position of those lamellas in relation to the surface, where the nitrogen atoms come from.

**Keywords:** cast iron, ductile, nitriding, layer, wear

consists of graphite nodules embedded in a ferrite, pearlite or ferrite-pearlite matrix<sup>[3]</sup>. The microstructure presented by a material is of practical importance because it determines its mechanical properties. After austempering treatment the microstructure consists of acicular ferrite and austenite, which gives high strength and ductility, and offers a good combination of mechanical resistance and resistance to wear, with low cost and good toughness<sup>[4]</sup>. Their densities are about 10% inferior to those presented by steels and its ultimate tensile strengths vary from 850 to 1600 MN/m<sup>2</sup>.<sup>[5]</sup> Therefore the strength/weight ratio of these materials is superior to that of most steels commercially used. Treatments of surface hardening can increase even more the advantages of these materials as to wear and to fatigue. Plasma nitriding is a suitable treatment due to the process control. It produces a "compound layer" on the surface which has good tribological properties. In addition, the fatigue characteristics of the material can be considerably improved, particularly when nitrogen is retained in solid solution in the "diffusion zone" beneath the "compound layer"<sup>[6]</sup>.

Plasma nitriding is a thermochemical diffusion process that modifies the surface chemical composition, adding chemical elements, such as nitrogen or carbon in the crystal lattice of the substratum. Those elements combine chemically with iron and other alloy elements in the materials surface, forming an external layer that promotes the improvement of fatigue properties<sup>[1, 7]</sup>. Several models were proposed in the past to explain the mass transfer and the layer formation in the surface of the piece. The best accepted model is that suggested by KÖLBEL, based on the sputtering by ion bombardment<sup>[8]</sup>. The iron nitride FeN that is condensed in the work piece surface is unstable, and on constant bombardment due to the coming ions of the plasma, it is decomposed progressively into the phases  $\epsilon$  (Fe<sub>2</sub>N e Fe<sub>3</sub>N), and  $\gamma$  (Fe<sub>4</sub>N), forming a compound layer and a diffusion layer.

## Introduction

The ductile cast iron has been presenting an accentuated growth of production due to its interesting mechanical properties and economic advantages of production<sup>[1]</sup>, such as: low melting point, high fluidity low shrinkage and good machinability, in comparison to steel or other ferrous alloys<sup>[2]</sup>. The microstructure of typical commercial ductile cast irons in the as-cast condition

In each stage of the condensation process, atomic nitrogen is liberated, both from the plasma and from the surface of the work piece<sup>[7]</sup>. The two processes, sputtering and condensation, are influenced by the type of gas used, pressure variation and applied voltage.

The present investigation was carried out to study the nitrided layers produced on ductile iron with different chemical compositions, in the as cast and austempered conditions, by plasma nitriding.

## Materials and Methods

Ductile cast iron of three different chemical compositions (1 to 3) was used in this work. The first alloy used in this investigation is a conventional ductile cast iron. The second alloy contained additional 0,24% copper. The third alloy presents the highest carbon content of the group.

The alloys used in this work were melted in an induction furnace and poured in sand moulds produced according to the ASTM standard A897-90. Cylindrical samples were obtained from those alloys, with diameter and height of 10mm.

Samples in the as-melted state and samples austenitized at 900 °C and austempered for 320 °C in melted salts, were nitrided for 5 hours at 500 °C using continuous current and atmosphere constituted of the 80% H<sub>2</sub> – 20% N<sub>2</sub> gaseous moisture.

Microhardness profiles of the layers were determined and examination by optical microscopy was performed after attack of the samples with the reagent 2% HNO<sub>3</sub>- 98% C<sub>2</sub>H<sub>5</sub>OH. X-ray analyses of the layers in a Rigaku Rotaflex equipment (Cu K $\alpha$  radiation – 5° to 120°) was also performed.

The wear test realized was of the type “pin-on-disc”, being used as abrasive sandpapers of silicon carbide with 600 mesh. The rotation of the disc was of 45 rotations per minute and the load of 320g. To each 200 turns of the disk the sample was weighed for determination of mass loss. With those results, it was plotted the graph of specific mass loss x number of turns, in agreement with the equation:

$$Pe = (Mo - Mn)/Mo, \text{ were:}$$

Pe = specific mass loss

Mo = initial mass sample

Mn = mass to each 200 turns.

## Results and Discussion

The chemical compositions of the alloys are reported in the table 1.

Table 1: Chemical compositions of the alloys (wt%)

Alloy	C	Si	Mn	Cu	Fe
1	2.720	2.830	0.490	-	bal.
2	3.290	2.910	0.310	0.240	bal.
3	3.590	2.400	0.490	-	bal.

The microhardness profiles after the nitriding are presented in the figure 1. It is verified that the tickness layers, including the compound layer and the diffusion zone, is about 130µm. The maximum value of layer hardness was obtained with the previously austempered alloy. The substrate hardness of austempered samples is bigger than those of the as-cast samples. The austempered and nitrided samples presented hardness levels superior to those of the as-cast nitrided alloys. Figure 2 graphically illustrates the X-ray diffraction results of the layer produced in the alloy number 3 austempered at 320°C and nitrided. It is verified that the layer is biphasic is constituted by  $\gamma$  (Fe<sub>4</sub>N) and  $\epsilon'$  (Fe<sub>24</sub>N) nitrides. Figures 3 to 9 show the optical photomicrographies of the alloys. Figure 3 shows alloy 1, whose matrix is constituted of ferrite and graphite nodules due to the smaller carbon content. The presence of the nitride layer and the preferential formation of nitrides in the ferritic grain boundary are verified, due to the easiness of the nitrogen diffusion for the same ones. In figure 4 and 5 (alloy 2) the matrix is pearlitic with ferrite in smaller proportion. It is possible to verify the presence of the surface nitride layer on the pearlite and the preferential nitrides formation in the ferrite lamellas and grain boundaries. Figure 6 shows the pearlitic microstructure of alloy 3. It shows the formation of the nitride layer on a pearlite surface grain. The ferrite lamellas normal to the surface of the sample were the preferential areas for the diffusion and nitrides formation.

Figures 7 to 9 present the photomicrographs of the austempered and nitrided alloys. It is verified the bainitic microstructures and thickness of the nitrided layer which is wider than that presented in the samples in the as-cast and nitrided state.

Figure 10 shows the results of the wear tests of the austempered and nitrided alloy. The best performance is verified for the alloy with the smallest carbon content and that also presented the highest nitrided layer hardness. The alloy 3 austempered presented the worst wear resistance probably due to the brittleness of the biphasic layer compound.

Figure 11 shows the results of the wear tests of the as-cast and nitrided alloy. In this case, the alloy 1, with ferritic substratum, presented the lowest hardness of the nitrided layer and the worst wear resistance probably due to the lower substratum resistance to support de nitrided layer.

The austempered samples present better performance as to hardness levels reached in the nitriding, in comparison to those of as-cast samples. In the case of pearlitic matrix, the nitrogen diffusion and the consequent nitrides formation will

happen in the ferrite lamellas. Such diffusion will be associated to the position of those lamellas in relation to the surface, where the nitrogen atoms come from.

## Conclusions

The nitrided layer produced in the austempered ductile iron is biphase and is constituted by  $\gamma$  ( $\text{Fe}_4\text{N}$ ) and  $\epsilon'$  ( $\text{Fe}_{2.4}\text{N}$ ) nitrides.

Austempered ductile irons presented thicker nitrided layers and with higher hardness levels than those obtained in the as-cast and nitrided alloys.

In the as-cast and nitrided conditions, the nitrogen diffusion and consequent nitrides formation will happen preferentially in the grain boundaries or in the ferrite lamellas with positions normal to the surface.

## Acknowledgments

The authors would like to acknowledge the help of FAPESP, especially in providing the nitriding equipment (Proc.1995/0677-0).

## References

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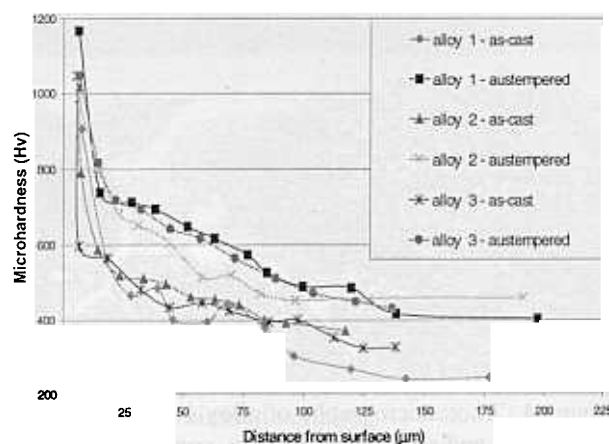


Figure 1 : Microhardness profiles

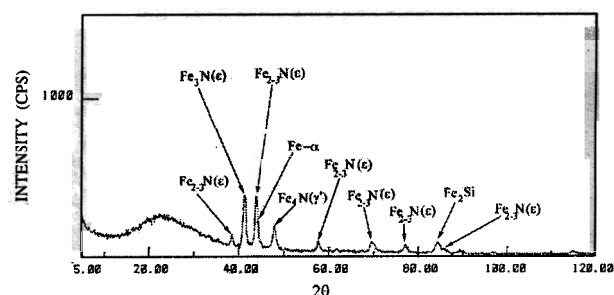


Figure 2 : X-ray diffraction result of the nitrided layer on the austempered and nitrided number3 alloy

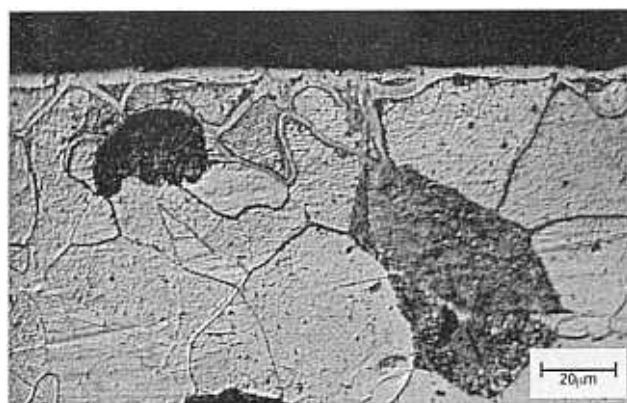


Figure 3 : Photomicrography of alloy 1- Nitrided layer on the as-cast and nitrided matrix constituted by pearlite and ferrite



Figure 4 : Photomicrography of alloy 1- Nitrided layer on the as-cast and nitrided substratum constituted by ferrite, and graphite nodules

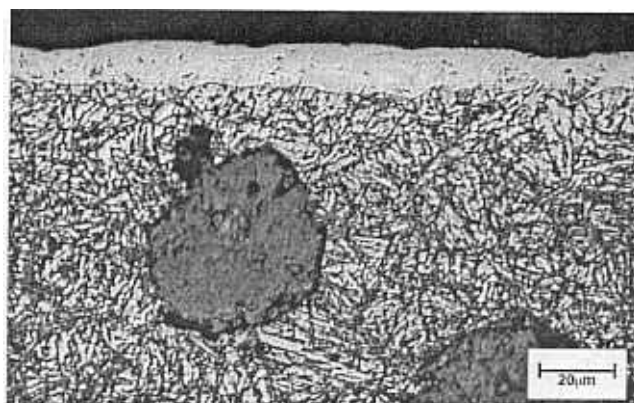


Figure 7 Photomicrography of alloy 1- Nitrided layer on the austempered and nitrided substratum constituted by bainite and graphite nodules

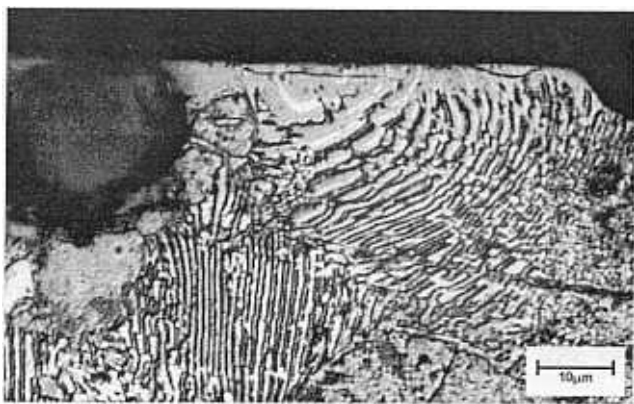


Figure 5 : Photomicrography of alloy 2- Nitrided layer on the as-cast and nitrided substratum constituted by pearlite and graphite nodules

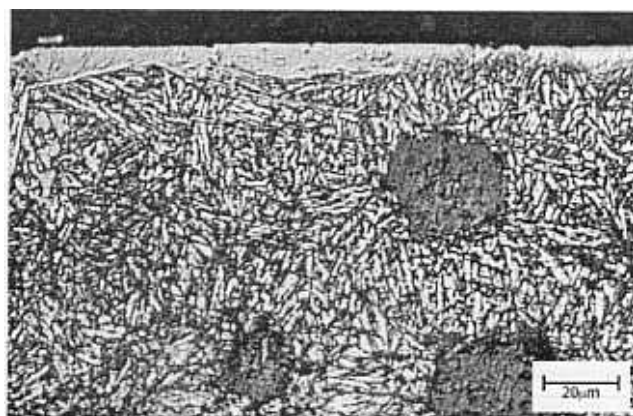


Figure 8 : Photomicrography of alloy 2- Nitrided layer on the austempered and nitrided substratum constituted by bainite and graphite nodules

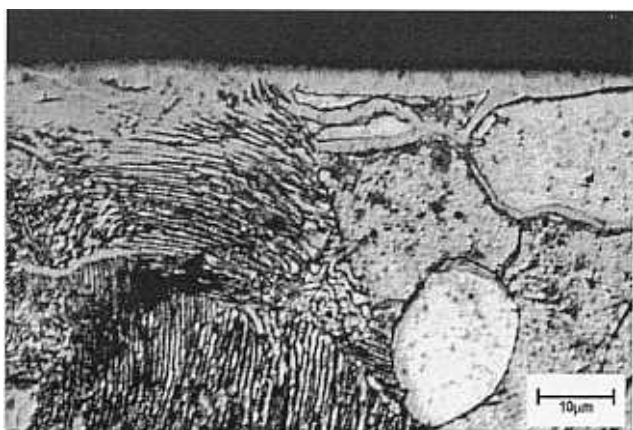


Figure 6 : : Photomicrography of alloy 3- Nitrided layer on the as-cast and nitrided substratum constituted by pearlite, ferrite and graphite nodules

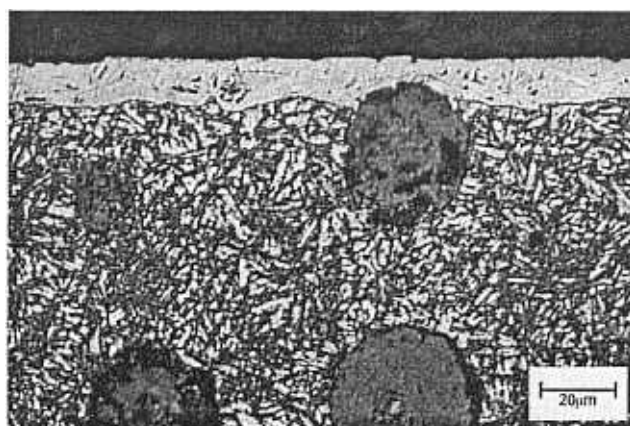


Figure 9 : Photomicrography of alloy 3- Nitrided layer on the austempered and nitrided substratum constituted by bainite and graphite nodules

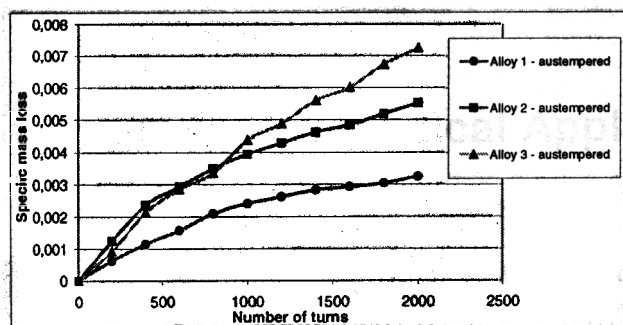


Figure 10 – Specific mass loss plotted against number of turns – austempered and nitrided samples.

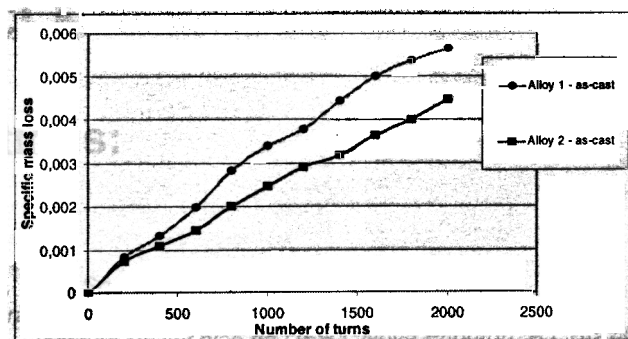


Figure 11 – specific mass loss plotted against number of turns - as-cast and nitrided samples.