

## WEAR RESISTANCE OF AISI D2 MICROABRASIVE PLASMA NITRIDED

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**Abstract.** *The nitriding by plasma has been widely used in many industrial applications to provide improvement in mechanical properties and tribology. This work studies the influence of time of treatment in the formation of nitride layer of AISI D2 tool steel and the resistance to micro-abrasive wear from the technique of nitriding in plasma. The samples were nitrided at 400 ° C with a pressure of 4.5 mbar (450 Pa) and using a gas mixture of 80% vol.H<sub>2</sub> and 20% vol.N<sub>2</sub>. The times of treatment were: 30, 60, 120, 180 and 360 minutes. The properties of the layers in the samples obtained nitrides were assessed by surface microhardness, X-ray diffraction and test for resistance to micro-abrasive wear. The results show that for all times of treatment there was a tightening surface and a significant increase in the resistance to micro-abrasive wear on the non-nitride material. However, the best results for nitriding to 400° C, was obtained with the time of treatment of 360 minutes. In this case the increase in surface hardness was 94.6% and resistance to microabrasive wear of 15%. This increase in hardness may be associated with high concentration of nitrogen in the crystalline network of iron- $\alpha$  gives greater distortion of the reticulated hindering the movements of dislocations and additional training of nitrides. The use of low temperature of nitriding reduces between grain fragility of treated steel to reduce the likelihood of precipitation of nitrides in a continuous manner in the austenite grain boundaries and the absence of previous  $\epsilon' + \gamma$  phases.*

**Key-words:** nitriding, microabrasive wear, steels AISI D2

### 1. INTRODUCTION

The study of tribological properties of surfaces has become essential in many industrial applications. Several techniques have been developed for the surface treatment to improve the surface properties of materials, including mechanical, thermal, physical and chemical.

The plasma nitriding is also known as ion nitriding or glow discharge nitriding (Gomes et al, 2003; Jeong et al 2001). It is a physical-chemical treatment thermally activated of hardening by the introduction of atomic nitrogen. The process involves a gas mixture composed of nitrogen and hydrogen in varying amounts, in a chamber with a pressure between 1333 Pa and 133.3 Pa, which is applied a potential difference between the part to be nitrided and reactor. The gas mixture is ionized and ions N<sup>+</sup> are accelerated against the part (Wolfart 1996). Through of process of adsorption and diffusion of nitrogen lattice distortion occurs through of the formation of iron nitrides and nitrides of alloying elements besides nitrogen in solution solid. Thus, it is achieved, mechanical and tribological properties superior to those of the base material (Wolfart 1996). This process has advantages over conventional nitriding treatment (Osbyal et al 1986; Leyland et al 1993) especially to the control of the characteristics of obtained layers: depth of nitriding, microstructure and thickness of the deposited compound. Additionally, ion nitriding has a low energy consumption and less distortion of parts.

The aim of this study was to perform the surface treatment of ion nitriding on AISI D2 tool steel at low temperature (400°C) and identify the changes in the nitrided layers with variation of treatment time. These microstructural changes are responsible for hardening having direct influence on the wear resistance of the material. The AISI D2 is used in several situations, including dies for stamping and forging. Applications where beyond of good mechanical strength, it is important a high resistance to wear (Chiaverini 1977).

### 2. MATERIALS AND METHODS

The material used this work was the tool steel AISI D2. The chemical composition determined by mass spectrometry, is presented in Table 1.

Table 1: Chemical composition (wt%) of AISI D2

Element	C	Mn	Si	Cr	V	Mo	Ni	S	P
%	1,49	0,35	0,42	12,04	0,68	0,64	0,18	0,003	0,023

The tempering was performed in vacuum furnace heated to 1050°C for 30 minutes followed by cooling in N<sub>2</sub> gas at a pressure of 5 bar up to 25°C. Then the specimens were subjected to double tempering at 540°C for 2 hours. The final hardness of 57 HRC was achieved.

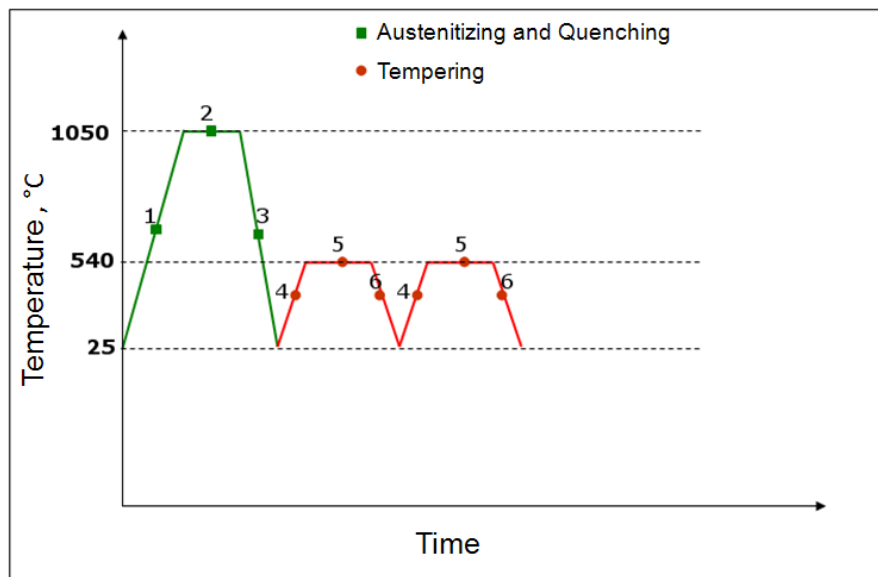


Figure1- Representation of the heat treatment cycle used for the AISI D2.

The ion nitriding of tool steel was conducted at the Department of Physics, UFSCar, as illustrated in Figure 2. The nitriding parameters of the specimens were: temperature 400°C, pressure of 4.5 mbar and gas mixture of 80% and 20% vol.H<sub>2</sub> vol.N<sub>2</sub>. The treatment time was 30 minutes, 60 minutes, 120 minutes, 180 minutes and 360 minutes.



Figure 2: Equipment for ion nitriding.

To characterize the samples, several tests were performed. In the microstructural characterization was used metallographic optical microscopy Olympus BX60M and Scanning Electron microscopy. To determine the microhardness, we used a microhardness HMV model 2 version 1.23 (Shimadzu, Japan) with Vickers indenter and load 500 gf. The identification of the nitrides of the nitrified layers was performed by X-ray diffraction. We used a copper K $\alpha$  radiation with scanning between 30° <math>\angle 2\theta < 90^\circ</math> and angular speed of 0.5 °/ minute.

The test wear microabrasive was conducted in a microabrasion equipment free ball type (brand CSEM) belonging to the Laboratory of Tribology and Materials, Federal University of Uberlandia (Figure 3).

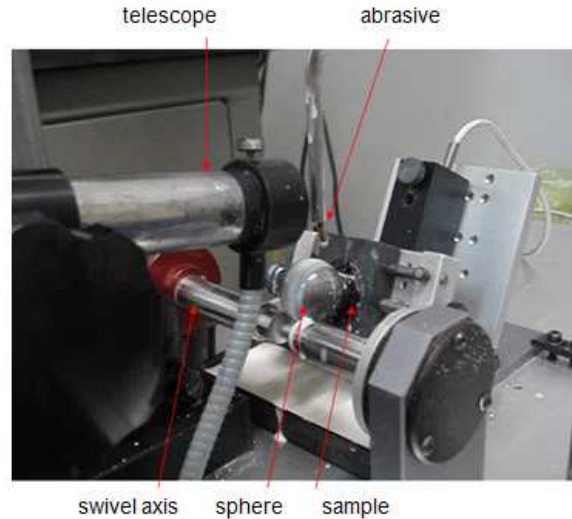


Figure 3: Overview of the microabrasion equipment.

To conduct the wear test, we used an AISI 52100 steel ball with a diameter of 25.4 mm. The abrasive medium was a suspension of abrasive particles composed of silicon dioxide (SiO<sub>2</sub>) in distilled water at a concentration of 0.75 g of abrasive per cm<sup>3</sup> of water. The size of the abrasive particles was between 0.5-10 $\mu$ m, with approximately 80% between 1-5 micrometers. The mixture was pumped up the ball-sample interface, using a peristaltic pump. The flow of the abrasive is fixed at about one drop every 3 seconds. The rotation of the motor shaft was maintained at 150 rpm, generating a velocity between the ball surface and the sample of about 0.1 ms<sup>-1</sup>.

The formulation for calculating the volume of worn and wear coefficient depends on the similarity between the crater wear and the shape of the ball used in the test. The wear volume (V) and wear coefficient (K) after each interval of sliding distance, were determined using the equation (Cozza 2006).

$$V \cong \frac{\pi \cdot b^4}{32 \cdot \phi} \quad \text{e} \quad k = \frac{\pi \cdot b^4}{32 \cdot \phi \cdot L \cdot F_N} \quad \text{to } b \ll \phi$$

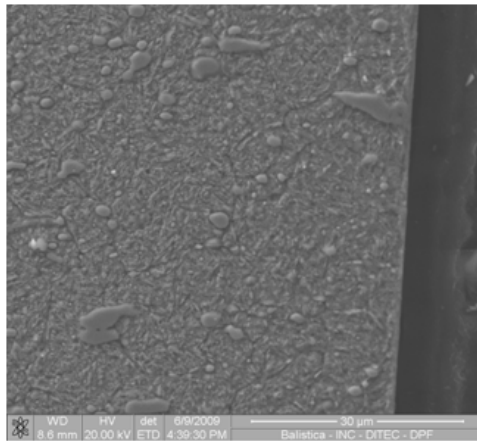
Where: b, the diameter of the shell of wear;  $\phi$ , sphere diameter test, F<sub>N</sub>, normal force to the sample, L, sliding distance.

### 3. RESULTS AND DISCUSSION

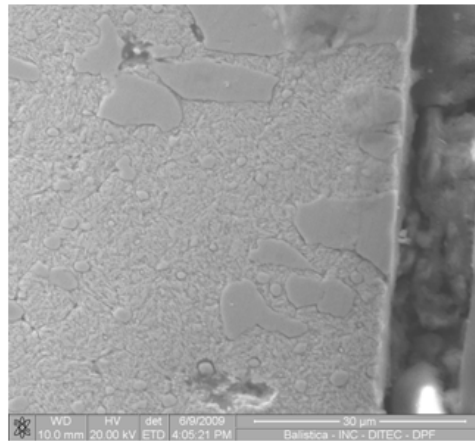
#### 3.1. Characterization of the nitrified material

##### 3.1.1. Microstructure

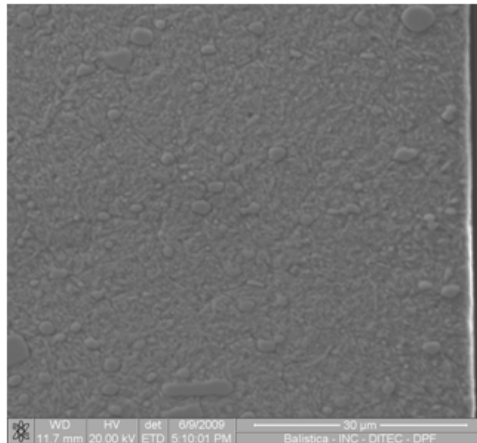
The sequence of Figures 4 (a) to (f), obtained by scanning electron microscopy, show the material nitrified according to the time of treatment.



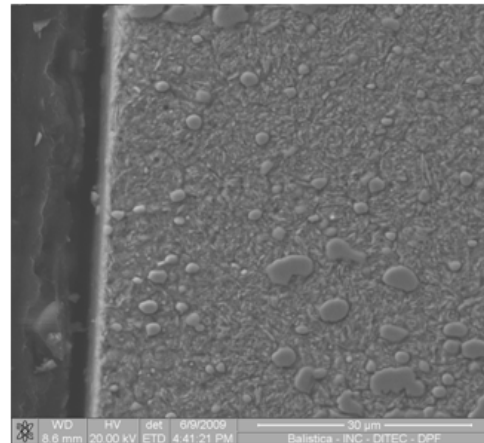
(a) untreated material



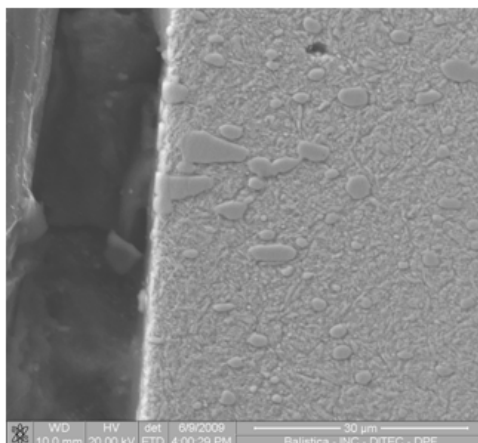
(b) 30 minutes



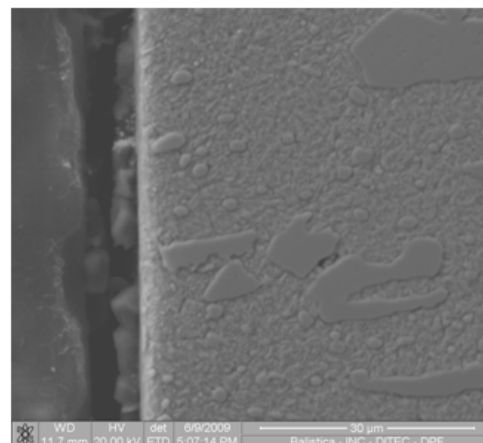
(c) 60 minutes



(d) 120 minutes



(e) 180 minutes



(f) 360 minutes

Figure 4: Microstructures obtained in Scanning Electron Microscopy, of the nitrided samples, depending on the time of nitriding. Attack: Nital 2%. Magnification: 4000X.

The micrographs of the material nitrided compound layer is not present in any of the conditions studied. There is a martensitic microstructure with carbides primary, secondary and a small diffusion layer. There was no

formation of new phases continuous in grain boundary, because the interstitial nitrogen concentration does not exceed the solubility limit in crystalline structure of  $\alpha$ -iron.

The diffusion layer is responsible for changes in the mechanical properties of the material surface. This layer promotes increased of hardness and compressive residual stresses that are responsible for the increase in mechanical properties (Tier 1998; Farrahi et al 2006).

### 3.1.2. Surface Microhardness

Figure 5 shows the variation in the surface hardness of nitrided layers as a function of nitriding time. Also in this figure are shown the values of microhardness of the untreated material. The values represent an average of 5 indentations.

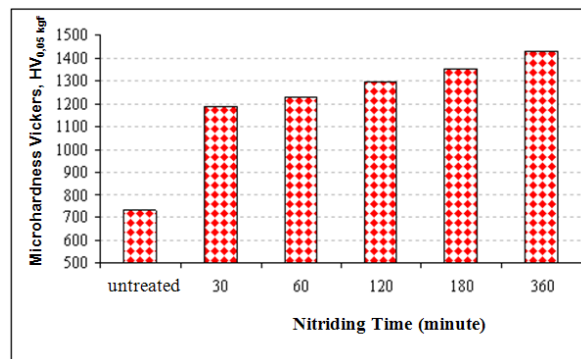


Figure 5: Microhardness of the samples surface.

Microhardness on the surface of the nitrided layer increases with the increase of treatment time reaching 1426.4 HV to 360 minutes. With increasing time, there is a higher concentration of nitrogen in solid solution that lead to greater distortions in the matrix, promotes the increase of hardness. In times smaller, the hardness increase is smaller, but significant in relation untreated material.

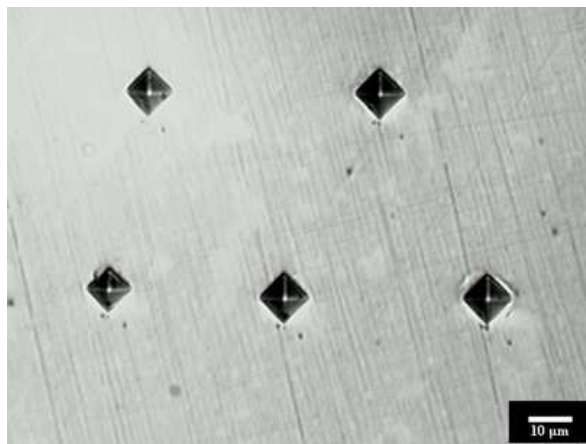


Figure 6: Vickers indentations in the top of untreated sample. Used load: 0.05 kgf Magnification: 400X

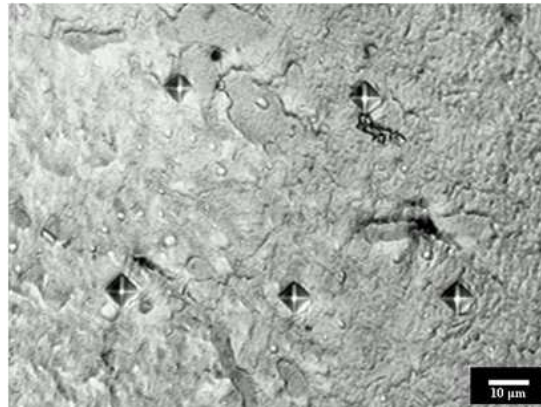


Figure 7: Vickers indentations on the surface of the nitrided layer for treatment time of 360 minutes. Used load: 0.05 kgf. Magnification: 400X

### 3.1.3. Determination of phases by X-ray diffraction

The Figure 8 (a) to (f) shows the patterns obtained by analysis of X-ray diffraction to identify possible nitrides formed in the nitrided layers.

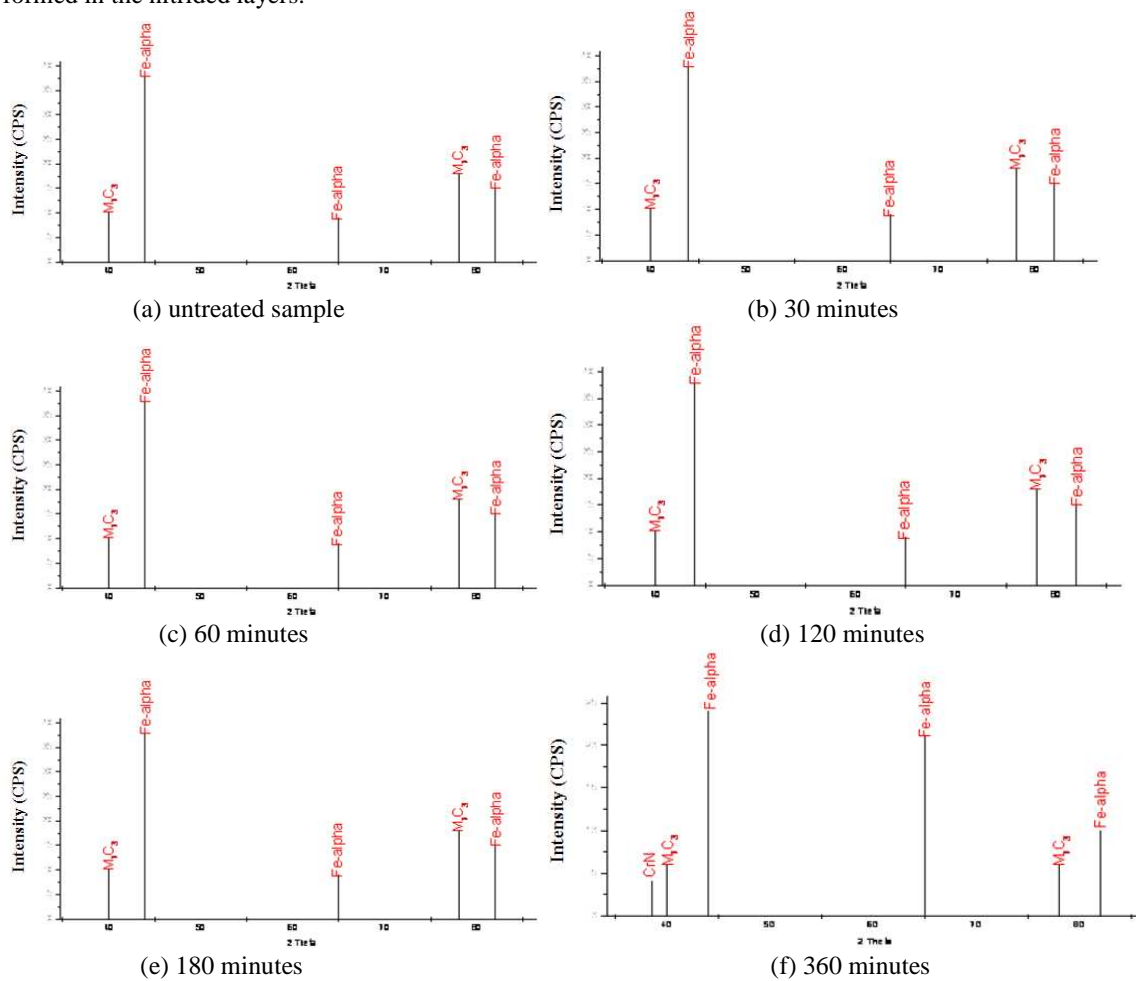


Figure 8: Patterns of X-ray diffraction of the nitrided samples in the five different times and for the sample non nitrided.

As can be seen in the diffractograms of Figure 8, for the longest time, it appears the additional step of chromium nitride. In other times of nitration were not observed at the level of resolution of the equipment used, stages related to the formation of nitrides.

### 3.1.4. Wear tests by microabrasion

The results of this test, described as worn volume of the material and the wear coefficient, are illustrated in Figures 9 and 10.

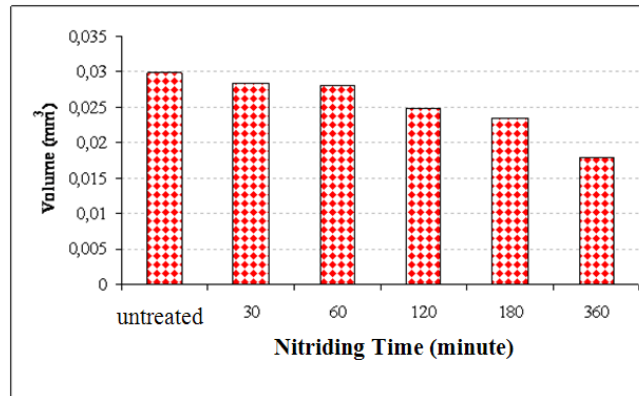


Figure 9: Volume of material removed in one hour test for each time of nitriding.

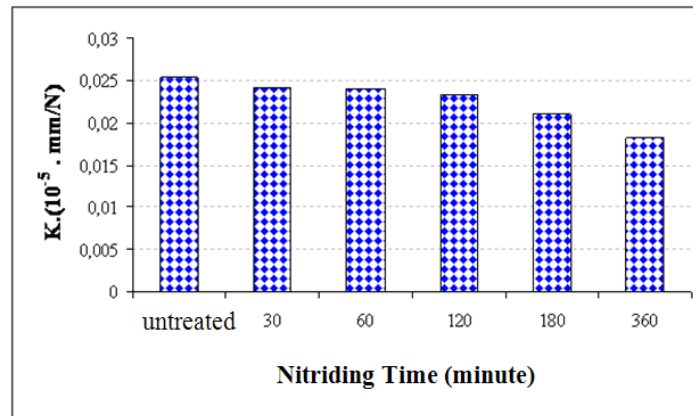


Figure 10: Coefficient of wear of material for each nitriding time

As shown in Figure 11, the wear rate tends to stabilize only after a sliding distance traveled approximately 260 meters (40 minutes) test. This sliding distance minimum to stabilize the coefficient of wear was observed for all treatment conditions.

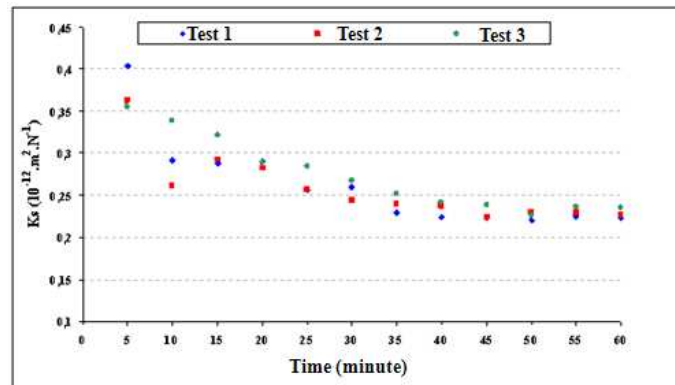


Figure 11: Coefficient of wear versus time test. Sample nitrided in time of 180 minutes.

The behavior exhibited by each of the curves of microabrasive wear is associated with the microstructural changes that occur in the substrate with increasing time of nitriding treatment (Pessin 1999). In general we observe that the nitrided samples showed a better behavior in the volume of worn material than untreated. The decrease was more significant for longer used.

The significant increase in wear resistance for low temperature nitriding treatment can be correlated to no formation of continuous phases at grain boundaries, increased hardness, the formation of precipitates and the depth of hardening. The presence of continuous phases with high hardness at grain boundaries concentrate stress which, together with the external loading (tension trative), facilitates nucleation and propagation of cracks. Thus, the presence of a film with high hardness at grain boundaries weakens the layer favoring the wear mechanisms. Probably, this good performance due to wear can also be related with the formation of compressive residual stresses. These values of compressive residual stresses formed on the surface are higher when the nitration is effected at low temperatures (Tier 1998; Farrahi et al 2006). Residual stresses occur when there are physical restrictions to volumetric change in specific parts of the material. In nitriding mainly occur due to the gradient of composition of the surface layer making the substrate (compositional tension) and the different linear thermal expansion coefficients between layer and substrate (thermal stress). The thermal stress in the layer is induced during cooling from the nitriding temperature to room temperature. The composition gradient occur due the presence of a concentration profile of nitrogen, an increase of its volume fraction in the displacement direction surface. Once imposed a cohesive force between the layer and substrate by an elastic accommodation, a compressive stress parallel to the surface occurs with a magnitude that increases with the distance of interface in direction the surface. This is offset by not nitrided core resulting in tensile stresses.

#### 4. CONCLUSIONS

The nitriding treatment at low temperature caused significant increases in wear resistance of microabrasion tool steel AISI D2, being more relevant to the time of 360 minutes, which increased by approximately 15% compared with the untreated steel. With the use of low temperature treatment was possible obtain high levels of abrasion resistance and reduce the probability of precipitation of carbides and nitrides at grain boundaries. In this case, the absence of solid phases with high hardness at the interfaces of grains reduced the stress concentration and impaired the performance of wear mechanisms. In none of the cases studied there was formation of the compound layer ( $\gamma'$  phase ( $\text{Fe}_4\text{N}$ ) and  $\epsilon$  ( $\text{Fe}_{2-3}\text{N}$ )). The process conditions used were appropriate considering that the possible presence of these two phases would leave the surface layer more brittle and susceptible to cracking due to weak adhesion between the two phases and the difference between the coefficients of thermal expansion

The increase of surface hardness, for all times of treatment was provided by the presence of nitrogen in solid solution and nitrides. Nitrogen atoms in the interstices of the crystal lattice of  $\alpha$ -iron and the formation of nitrides distorting the crystal lattice, reducing the movement of dislocations and consequently difficult plastic deformation.

The surface hardening was greater for the nitriding time of 360 minutes. This increase of surface hardness was of 94.6% compared with untreated. This increase is due to higher concentration of nitrogen diffused in martensitic matrix that provides a greater distortion of crystal lattice and also due to additional formation of nitrides.



## 5. ACKNOWLEDGEMENTS

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