ESTUDY OF THE GAS ATMOSPHERE IN PLASMA NITRIDING OF STEEL AISI H13

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Abstract. *In the last decades a great advance has been observed in processes for the metals surfaces modifications and the plasma nitriding has become one of these techniques. In steel alloys for hot working such as the AISI H13, the plasma nitriding is solution to increasing tool's lifetime, thus it increases the superficial hardness and the wear resistance. The plasma nitriding of AISI H13 steel was performed and the influence of the composition of different gaseous mixtures (N_2-H_2) on the microstructure of the nitrided surface was evaluated. As process parameter, temperatures of 520ºC were used for 3 hours and atmospheric composition has been varied (25%N_2+75%H_2, 50%N_2+50%H_2 and 75%N_2+25%H_2). The use of low nitrogen in plasma nitriding prevented formation of a white layer, allowing to get only from the diffusion zone, region of 860 HV hardness, whereas a high nitrogen potential favored the formation of white layer of approximately 1000 HV. The results had shown that the plasma nitriding produces surfaces with good characteristics of hardness and microstructure. The control of the nitriding parameter (atmosphere) can lead to nitriding surfaces with different microstructures for the same steel, allowing to increase the wear resistance of the AISI H13 machined molds.*

Keywords: *plasma nitriding, AISI H13, microhardness.*

1. Introduction

The development of new types of steel and optimization of existing qualities has been the traditional method of improving tools performance. However, development of new steel alloys has a high cost of research and development, despite process risk taking. Improvement of the quality by making use of special remelting and processing procedures raises material costs and, in many cases, ruins cost-benefit ratio.

The constant need for producing components with high mechanical properties, using material and processes of low cost, have been an endless technological search. Due to these reasons, surface engineering has grown, with the aim to improve the tribological properties of conventional materials, searching the increase in tools performance.

The nitriding is a thermo chemical process that confers special properties to steel surface. This treatment involves the nitrogen diffusion into the steel, in temperatures between 400 and 600ºC. The nitriding can be achieved by different processes: gaseous, liquid or plasma.

During years, different types of steel tools have been used in the manufacture of plastic injection molds. According to Pinedo (2004), AISI H13 steel is used for plastic injection, therefore greater wear resistance is demanded, since the polymers have abrasive properties. Achieving increase of superficial hardness after thermal treatment has been done for 45 to 52 Rockwell C hardness steel, by means of plasma nitriding, conferring to the tool a higher wear resistance observed in regular molds.

Surface engineering increases around the world as an alternative method to getting higher wear resistance of conventional materials, searching an improvement in tools performance. The nitriding is a thermo chemical treatment that increases the surface hardness and steel resistance to wear, corrosion and fatigue. The process of steel tools nitriding has been frequently used aiming to improve performance of tools in service, reduction of wear rate due to the increase of superficial hardness in parts and the reduction of attrition coefficient in nitrided steel.

The traditionally used processes of nitriding in industries are the gaseous nitriding (with the ammonia use) and the salt - bath nitriding. However, these processes are limited in relation to the control on the microstructure of nitriding surface, the maintenance of superficial integrity, and have restrictions to environmental points of view and the health of human being.

Plasma nitriding or also called ionic nitriding is a relatively recent thermal treatment, that consists basically of ionizing a gas or gaseous mixture, having the main element the nitrogen, through a luminescent discharge generated by a potential difference (400 until 1000Volts), between the cathode (shows to be nitrided) and the anode (generally the carcass of the reactor), in an atmosphere in a low pressure (1 to 10 Torr), main condition for maintenance of electric discharge (Alves Junior, 2001).

Ions produced in the plasma are sped up in direction the sample, shocking itself with it and supplying energy enough to heat it until the temperature of nitriding and to provoke the diffusion of nitrogen in material forming two distinct zones or subs-layer, the white layer and the zone of diffusion. These layers depend on the parameters of process, such as: gaseous atmosphere, time and treatment temperature.

The plasma nitriding process has several advantages worth of mentioning: low treatment temperature (disabling dimensional distortions of part), metallurgical control of the nitrided layer, reduced treatment time, evenly constant thickness of the layer, nitriding of specified parts and more economy.

The steel tools are widely used in metal mechanical industry, to manufacture tools that enclose operations of cut/conformation in cold and hot operation, casting of not ferrous alloys and polymer injection. The manufacture of these tools uses the most different types of steel tools, specific for each type of application.

According to Oliveira *et al* (2003), wear resistance is one of the most important factors which ends tools life when using the steel tools. Therefore, the life of a tool and its integrity depend mainly on the mechanical properties of its surface, and the nitriding is one of the used processes to optimize the life of tools.

Another dimension to studying the current session, Pinedo (1996), observed that it is possible to program nitriding parameters that control the microstructure in the surface, in order to form or not the white layer. White layer forms itself when the nitriding is carried through with a rich gaseous mixture in nitrogen to an increased temperature and the same happens with opposite.

Oliveira *et al* (2003) observed that reduction of nitrogen potential and the reduction of temperature suppresses the formation of white layer when is used treatment conditions as described in the Tab 1. The use of higher temperature during the treatment results in a deeper nitriding, even when time experimented is three times lesser.

Table 1. Parameters of plasma nitriding and obtained properties.

Steel	Temperature (˚C)	Time (hours)	$Hv_{máx.}$	White layer thickness
H13	540	4,0	1.097	8,0
	520	12,0	1.018	------

Treatment conditions for 10 h and gaseous composition (10%N_2 and 90%H_2), did not allow the growth of the white layer, but the formation of diffusion zone. The non-white layer formed in these conditions is due to lack of nitrogen in the gaseous composition and formation of ferrous nitrate, are restricted in the regions of grain contour.

According to Hochman, this process keeps a high hardness surface with good properties of wear resistance, tenacity and resistance to annealing even when white layer is not formed.

This work presents the preliminary results of AISI H13 steel plasma nitriding, its evaluation in conditions of used gaseous mixture according to the properties gotten in process.

2. Material and Methods

This study used AISI H13 tool steel (see Tab. 2). Steel AISI H13 is very used in metal mechanics industry as tool to hot operation, with excellent mechanical properties in raised temperatures, high machinable and great dimensional stability on thermal treatment.

Table 2. Chemical composition of AISI H13 steel.

Chemical composition (%)					
Steel	**C**	**Si**	**Cr**	**Mo**	**V**
AISI H13	0,40	1,00	5,30	1,40	1,00

This steel is used for making molds of polymer injection where wear and corrosion resistance is primordial factors. However, the H13 is a little sensible to thermal shocks that occur in water-cooled tools. Due to this reason plasma nitriding was applied in this steel, thus it will influence in mechanical and thermal resistance improvement.

AISI H13 steel in supplied state (5HRC, 150Kgf), was thermally treated under conditions state of tempering between 1030 - 1050°C and annealing between 560 - 600°C.

On the plasma nitriding, it was used a reactor with internal dimensions of 600 mm of diameter and 1200 mm of height, being able to deal with parts up to deal up to 200kg. The process was lead in vacuum, under plasma in beaten direct current and used gases, such as: nitrogen (99,99%) and hydrogen (99,99%). The parameters of plasma nitriding used at this study are presented in Tab. 3.

Table 3. Parameters of plasma nitriding.

AISI H13 Steel	**Temperature (˚C)**	**Time (hours)**	**Hydrogen (%)**	**Nitrogen (%)**
Condição (a)	520	3	75	25
Condição (b)	520	3	50	50
Condição (c)	520	3	25	75

After nitriding superficial treatments, samples had been parted and metalographically prepared in transversal section. The microstructure was evaluated by optic microscopy. The hardness of substratum after thermal treatment of tempering and annealed was evaluated through hardness Rockwell C. The profile of microhardness of nitriding surface was carried through HV 0,025 Vickers measures of microhardness. The evaluation of gotten results he used parameters of processing was carried through.

3. Results

All materials had been thermally treated before nitriding to obtain hardness. The treatment was selected according to nitriding cycle characteristics of steel AISI H13. The hardness obtained after thermal treatment was 50-52 HRC.

In relation to gaseous composition, a high nitrogen potential favored the formation of compounds layer and a low potential, of diffusion zone. Although, plasma nitriding technique has been discovered more than 70 years, nitrided control is still empirically made through a monitoring process variation, such as pressure, temperature and gaseous mixture composition.

Figures 1 and 2 show the results for this research.

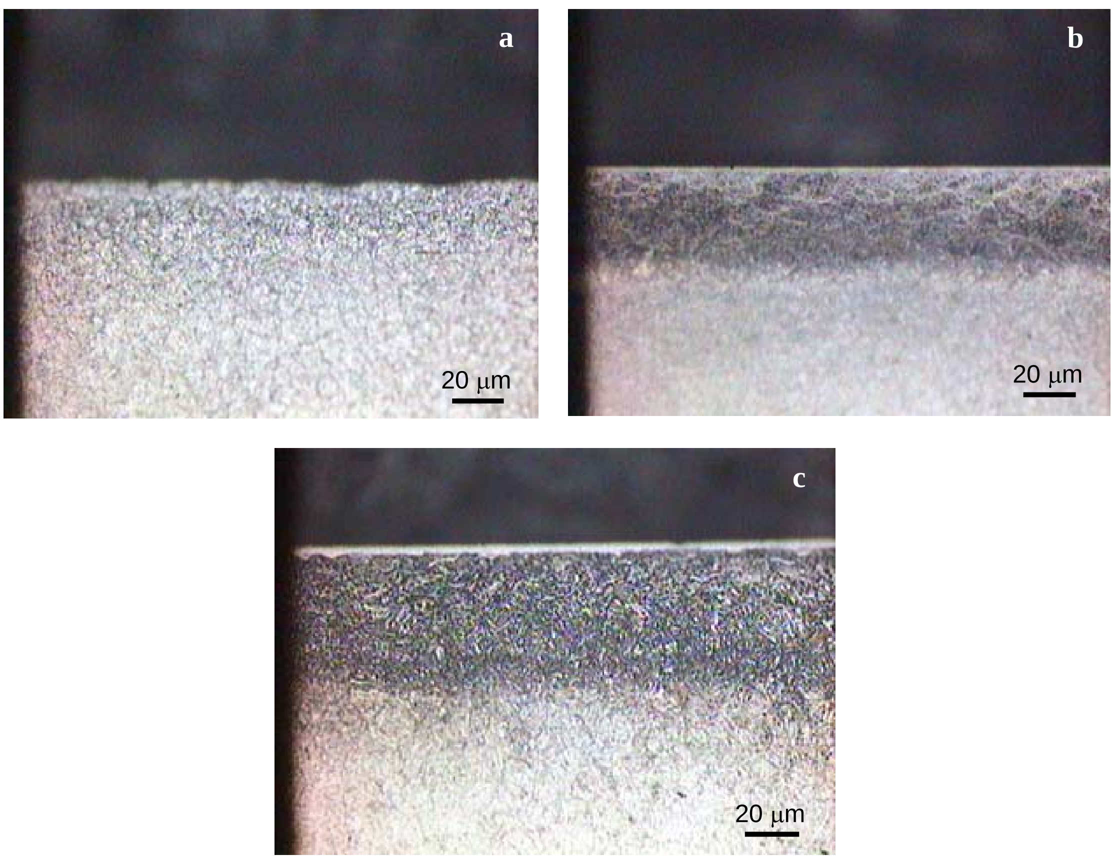

Figure 1. Micrographs of transversal section presenting the nitrided surface of steel samples AISI H13: (a) 25%N_2+75%H_2, (b) 50%N_2+50%H_2 and (c) 75%N_2+25%H_2.

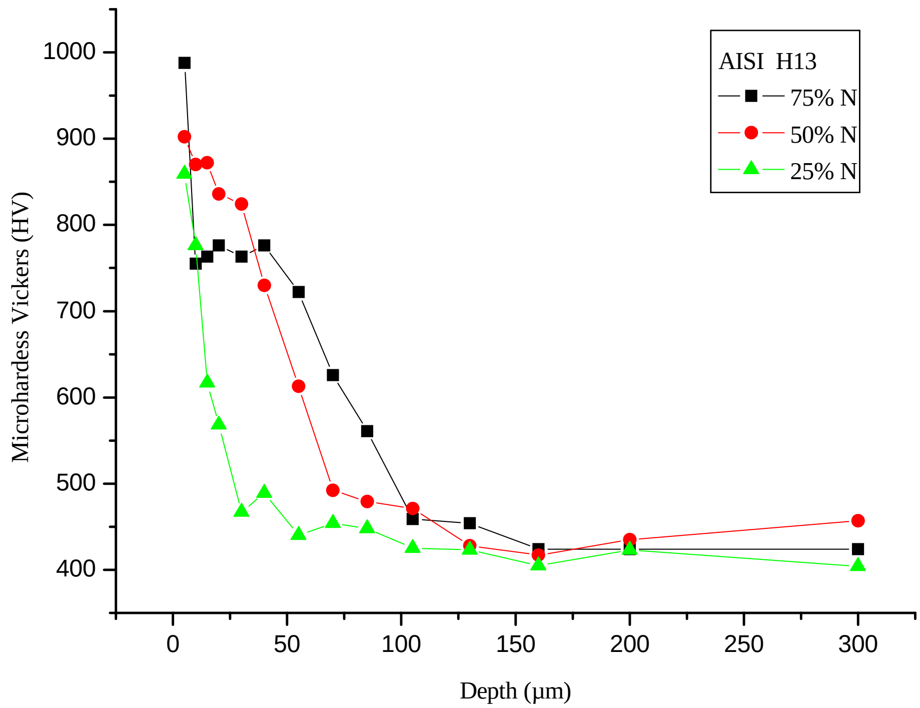

Figure 2. Profile of microhardness of steel samples AISI H13.

4. Discussion

The majority of main reactions in layer nitrided happens in cathode (nitrided material). Due to it, the cathode is the most important region in nitriding research. The most important facts that occur in reactions are the secondary electrons emission, surface sputtering, heat dissipation due to particles bombarding, defects that appear in crystalline structure of parts, deposition of nitrates, adsorption and diffusion of nitrogen.

The collision processes between gas particles dissociate molecules, exciting and ionizing atoms and molecules. To achieve this initial period of ionization, low gas pressure and a 300V voltage between the two electrodes (cathode and anode) contained in chamber that may be hermetically closed, and new loads are continuously produced through reactions of ionization and excitement, completing plasma nitriding cycle.

The obtained results for plasma nitriding treatment are summarized in Tab. 4.

Table 4. Properties after plasma nitriding condition.

Condição	ECC	EZD	HV 0,025
(a)	---	30	850
(b)	2	55	900
(c)	5	75	990

ECC: Thickness of layer compounds (mm).
EZD: Thickness of diffusion zone (mm).
HV: Microhardness Vickers (5 μm depth).

It was observed that there is the possibility of programming nitriding parameters for AISI H13 steel that control surface microstructure, in order to form or not white layer. This white layer is formed when the nitriding is done with high nitrogen gaseous mixture at a high temperature. On the contrary, there is also a significant reduction of white layer formation, when nitrogen concentration is lesser. Hardness of 850 HV was verified that in AISI H13 nitrided steel as condition (a), did not form white layer, leading only to nitrogen diffusion for the interior of material. This test indicated a significant reduction in interior of substratum to a depth of 35 μm.

As a result, this process allows to increase the superficial hardness keeping good properties of wear resistance, tenacity and resistance to annealing when applications with the presence of layers of hard nitrites of tool surface is not favorable (forging punctions, gears, machines components, knives of shears the hot one).

A white layer formation at approximately 900 HV followed by the diffusion zone was observed during the proposed condition (b).

For proposed condition (c) a rich gaseous mixture in nitrogen was used. The result was a layer compounds formed at approximately 990 HV followed by diffusion zone, this kind of layer is useful in tools surface for plastics injection molds, molds in general and machines components of under pressure casting.

The use of nitriding parts with presence of layer compounds guarantees ceramic characteristics, favoring polymers corrosion resistance.

The conditions (b) and (c) indicated a reduction of superficial hardness profile to the interior of samples to a depth of 100 μm.

5. Conclusion

The plasma nitriding revealed itself efficient for superficial hardening of AISI H13 steel. Some authors have mentioned that plasma nitriding is a solution for increasing lifetime of parts and components and can be applied in steel alloys for hot and cold working operations, steel fast, tool steel, carbon steel or non carboneted steel and ferrous casting. Other advantages are related to formation of a non-porous surface, observing different layers were formed, allowing to optimize the properties for each material application and absence of environmental pollutants.

The hardening obtained in material surface makes possible an improvement of final performance of mold due to the increasing of wear resistance. The ceramic characteristics are formed when the white layer is present and it also improves corrosion properties.

The hardness of superficial layer is influenced by the temperature and nitriding time. As mentioned earlier, the temperature was 520ºC and 3 hours treatment. The treatment performed with high nitrogen ($75\%N_2+25\%H_2$), favored the formation of white layer and, the use of low nitrogen, favored only a diffusion zone formation. The plasma nitriding of AISI H13 steel has demonstrated that the metallurgy of nitrated surface can be controlled to produce great specific applications for steel alloys.

The application of plasma nitriding is enable in some segments of the industries whereas greater performance in corrosion resistance and better mechanical and chemical properties are necessary. Development of such process has also demonstrated significantly improve results due to the easiness and security to handle gas nitrogen and for the properties gotten too.

6. Acknowledgments

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