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Surface Characterization of Through Cage Plasma Nitriding on the Surface Properties of Low Alloy Steel

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Abstract. *DIN 1.6580 low alloy steel has been plasma nitrided using the novel Through Cage (TC) plasma nitriding technique. Experimental results show that the thickness of the compound layer increases with temperature, as well as nitrogen percent, in the gas mixture, that an increase in screen hole size has no effect on the compound layer thickness under screen top lid conditions and, also, that an increase in screen hole size at the same temperature results in an increase in the thickness of the compound layer under iron plate top lid conditions. The results of micro-hardness measurements indicate that surface hardness increases with temperature, as well as nitrogen volume, in the treatment gas. It was also found that the hardness values obtained using a screen top lid are relatively greater than those obtained using an iron plate top lid.*

Keywords: *Plasma nitriding; Through Cage Plasma Nitriding (TCPN); Active screen; Compound layer; Low alloy steels.*

INTRODUCTION

Before the development of plasma nitriding, this process was carried out using other methods such as gas nitriding or nitriding in the salt bath. With the advent of plasma nitriding, which is based on a plasma environment or a glow discharge, the nitriding process was revolutionized. The advantages of plasma nitriding over conventional methods include a decrease in gas and energy consumption and a decrease or complete elimination of environmental pollution. The application of plasma surface modification technology using a glow discharge has been practiced in various countries over the past 30 years [1-4]. This method is used to improve the corrosion resistance, wear resistance and fatigue strength of steel components. In this process, the nitrided surface consists of compound and diffusion layers; the compound layer being composed of ε-$Fe_{2-3}N$ and γ'-Fe_4N phases, and the diffusion layer being composed of fine and dispersed precipitates of iron nitride or other alloying elements [4]. In spite of the advantages that the common plasma nitriding method offers over conventional nitriding methods, it has been hindered due to some deficiencies, such as: the heterogeneous temperature of parts with different dimensions and geometrics, limitations concerning the processing of specimens with complicated shapes, the formation of a non-uniform layer on the treated surface, the hollow cathode defect, the edging effect and arc damage to the components. Efforts to overcome these deficiencies have resulted in the development of conventional plasma nitriding, as well as the creation of Through Cage Plasma Nitriding (TCPN). This method is based on the use of an active screen, so that plasma is not necessarily formed on the surface of the specimen but instead on the screen surface [4-8]. The TCPN procedure is similar to the conventional plasma nitriding method. It differs, however, in that the screen is placed in the cathodic potential [4,7] and thus the production of active nitrided species, as well as the heating of the specimens, is conducted by the screen [4,5,7-9]. The plasma generated on

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the screen (rather than on the specimens) heats the screen, and radiation emitted from the screen supplies the heat required for treatment [1,4,10]. To date, few studies have been conducted as to the effect of different parameters of a through cage method on the properties of the nitrided layer on DIN 1.6580 low alloy steel, which is widely used in structural components for heavy demand, and seamless circular steel tubes for mechanical and general engineering purposes [1,2,4-10]. Thus, the present study is an attempt to investigate these parameters.

MATERIALS AND METHODS

DIN 1.6580 low alloy steel disc specimens of 20 mm diameter and 10 mm thick were used in the study. The chemical composition of the steel used in the study has been shown in Table 1.

The surface of the samples to be treated was ground and polished using standard metallugraphic procedures to yield a mirror finish before treatment. The through cage plasma nitriding treatment was carried out in a 5 KW conventional DC plasma nitriding unit, designed and created at Amirkabir University of Technology (Figure 1) using a nitriding screen produced on a laboratory scale (Figure 2). The active screen used was a mesh cylinder of 70 mm diameter and 50 mm height made of a 0.8 mm thick perforated st37 structural steel sheet, which had equally distributed round holes of 6 and 8 mm diameter. The top lid used in the study was detachable with two shapes: screen and iron plate. The samples were placed on the worktable, which was disconnected by ceramic insulation from negative potential (cathode) (Figure 2). Then, the screen was degreased and pickled in a frokilin solution, dried and, finally, placed on the worktable. The distance between the sample surface and the top lid was 12 mm and the nearest distance between the sample edge and the screen wall was almost 10 mm. The temperature was measured by a K type thermocouple, which was inserted into a 2.9 mm diameter hole at the center of the sample holder. The experiment was carried out at 550 and 580°C for 5 hr under a working pressure of 500 Pa.

The treatment gas was a mixture of 75% N_2-25% H_2 and 25% N_2-75% H_2. Following the experiment, the samples were cut from the middle for metallugraphic examination. The specimens were then placed in a Watts bath for nickle plating (in order to preserve the produced layer and prevent the destruction of the treated surface in metallugraphical procedures). The cut and plated samples were then wet ground and

Table 1. Chemical composition of DIN 1.6580 steel (in wt%).

Element	Fe	C	Cr	Mn	Mo	Ni	Si	P	S
Wt %	Balance	0.26-0.34	1.8-2.2	0.3-0.6	0.3-0.5	1.8-2	0.3-0.4	Max 0.035	Max 0.04

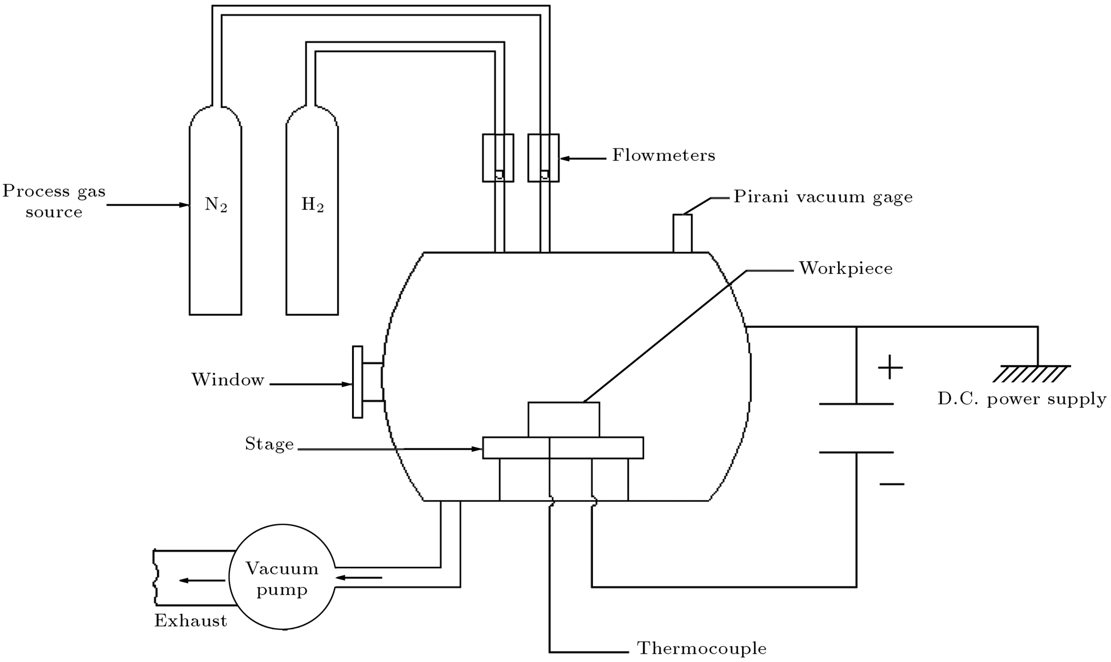


Figure 1. Schematic diagram showing the plasma nitriding system.

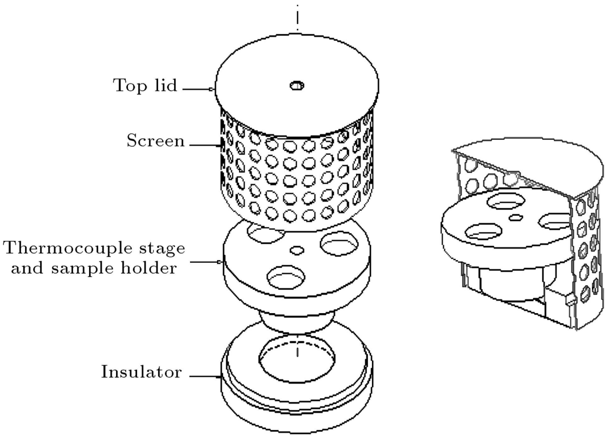


Figure 2. Schematic diagram showing the laboratory through cage plasma nitriding set-up.

fine polished using standard metallugraphic techniques; 2% Nital was used as the etch metallographic reagent. Various techniques were used to characterize the microstructures and properties of the treated samples. A microstructure analysis was carried out using both optical microscopy (OM:Olympus) and scanning electron microscopy (SEM, Philips-XL30). X-Ray Diffraction (XRD) analysis (Philips, Xpert X-ray diffractometer) using Cu-k$_\alpha$ radiation ($\lambda = 1.540\text{Å}$) with a scanning angle (2θ) ranging from 40 to 100° was applied for phase identification. The surface hardness of the treated samples was measured by a Vickers micro-hardness tester at a load of 10 gf.

RESULTS

The X-ray diffraction profile of surface layers under different temperature and top lid type (screen and iron plate) conditions, with a gas mixture of %75 N_2-%25 H_2, for screens of 6 and 8 mm diameter have been shown in Figure 3. Also, Figure 4 shows the X-ray diffraction patterns of treated specimens at 550°C, with a gas mixture of %25 N_2-%75 H_2, and a screen top lid for screens of 6 and 8 mm diameter. Based on X-ray diffraction results, the nitrided layer obtained for through cage nitrided samples is composed of ε and γ' iron nitrided phases.

Figure 3 shows that at 550°C regardless of top lid type, the intensity of the ε phase in the surface layer increases with the diameter size of the screen hole. Furthermore, it is seen from Figure 3 that at 550°C the relative intensity of ε, as expressed by $I_\varepsilon^{\text{screen-toplid}}/I_\varepsilon^{\text{iron-plate-toplid}}$, is 2.9 for a screen set-up with a 6 mm hole diameter, and 2.5 for an 8 mm hole diameter. Also, it can be observed that, at 580°C, $I_\varepsilon^{\text{screen-toplid}}/I_\varepsilon^{\text{iron-plate-toplid}}$ is 3.2 for a screen set-up with a 6 mm hole diameter. This indicates that the ε-phase in the compound layer for the screen set-up with a screen top lid is higher than that for the screen set-up with an iron plate top lid; as well as at higher treatment temperatures, the peak intensity of ε-phase is increased. Figures 3 and 4 show that, in the ASPN process, using a screen top lid, regardless of treatment gas mixture type and screen hole diameter, the intensity of the γ'-phase is much lower and the compound layer shows a tendency toward the formation of an ε-phase, whereas, under iron plate top lid conditions, the intensity of the ε-phase in the compound layer is reduced.

Figures 5-7 show the optical and scanning electron microscopy images of ASPN treated samples under various conditions for 6 and 8 mm diameter screens with screen and iron plate top lids. From Figures 5b and 5c, it is seen that under the same conditions the compound layer thickness increases with temperature and at a constant temperature; increasing the diameter size of the cylindrical screen holes has no significant effect on the compound layer thickness (Figures 5a and 5b). A comparison between Figures 5 and 6 reveals that, at a constant temperature, the screen set-up with a screen top lid generates a thicker compound layer, in comparison with the screen set-up with an iron plate top lid. Figure 6 shows that the compound layer thickness at a constant temperature under iron plate top lid conditions increases with screen hole diameter. Figures 7a and 7b indicate that an increase in the %volume of nitrogen in the gas mixture will lead to an increase in the compound layer thickness and that, with a screen top lid, an increase in the screen hole diameter under the same conditions, has little effect on the compound layer thickness (Figures 7b and 7c).

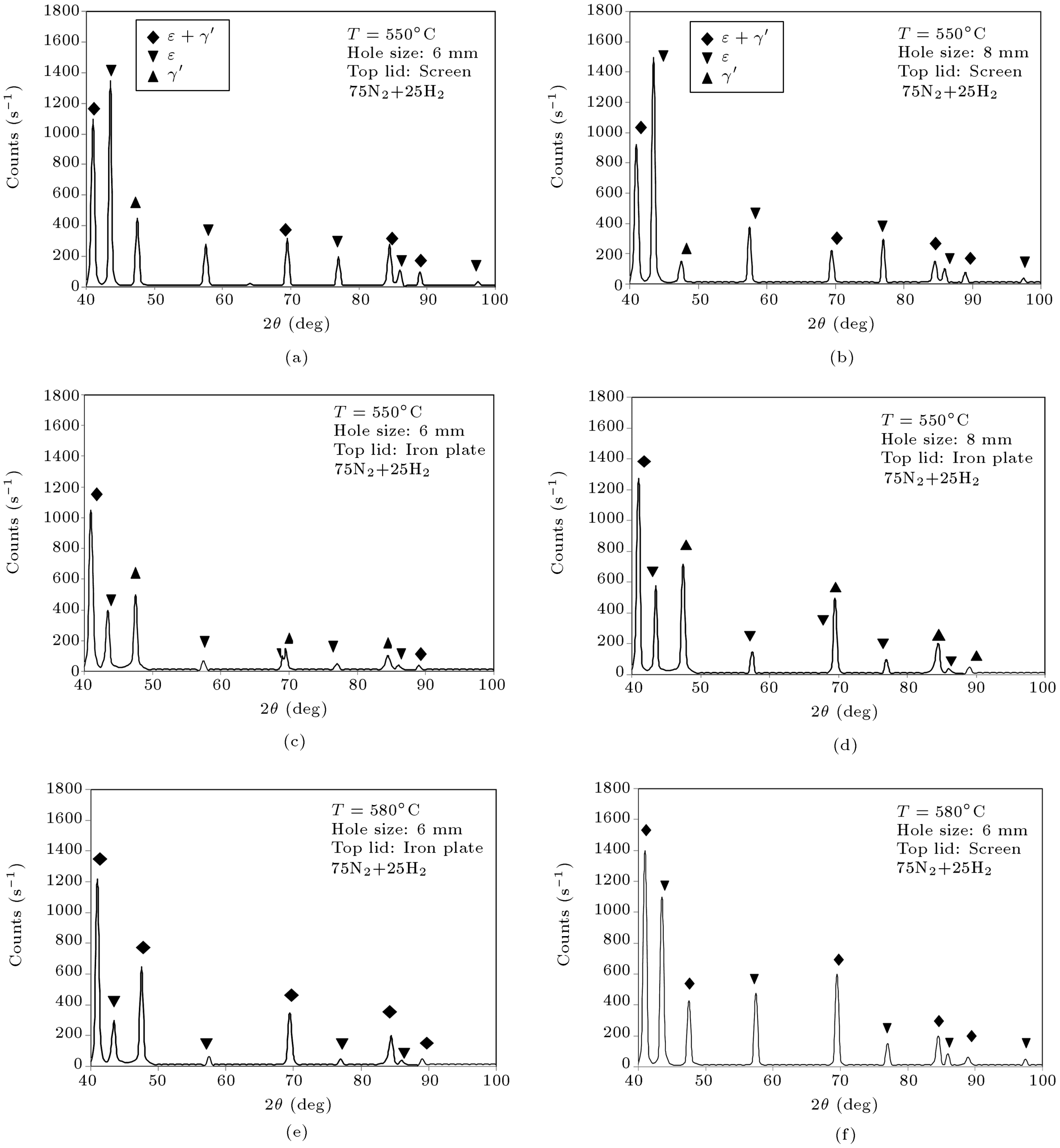
Compound layer thickness profiles of some of the treated specimens under various treatment and screen set-up conditions are presented in Table 2.

Figures 8 and 9 show the micro-hardness profiles of some of the samples under various conditions for 6 and 8 mm diameter screens with iron plate and screen top lids.

The results obtained from micro-hardness tests show that the hardness, as well as the hardened layer thickness, increases with temperature and %volume of the nitrogen in the gas mixture. It has been found that the top lid type (iron plate or screen) has little effect on the micro-hardness of the sample surface. The micro-hardness values for treated samples with a screen top lid are relatively higher than those for treated samples with an iron plate top lid (Figures 8 and 9).

DISCUSSION

In plasma nitriding, the structure of the layers can be varied by changing the treatment gas composition. In the presence of nitrogen or a mixture of nitrogen-hydrogen in the plasma atmosphere, the obtained



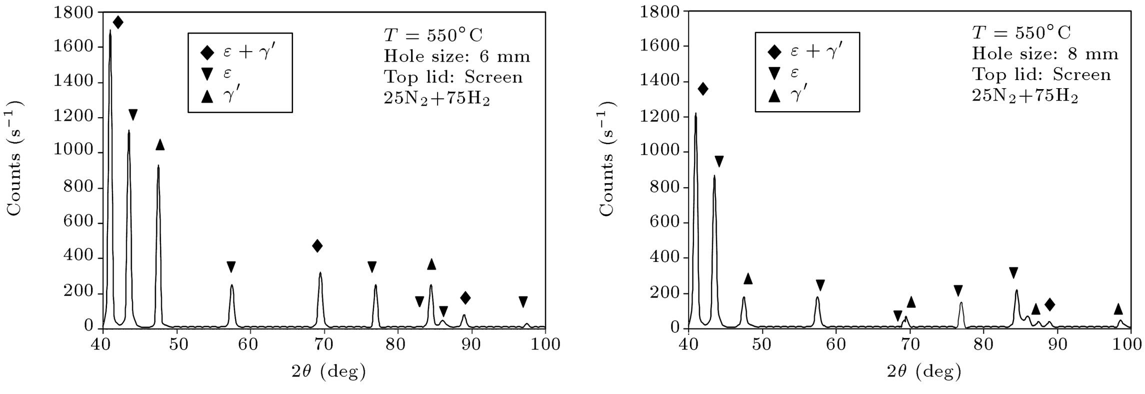
(a) (b)

(c) (d)

(e) (f)

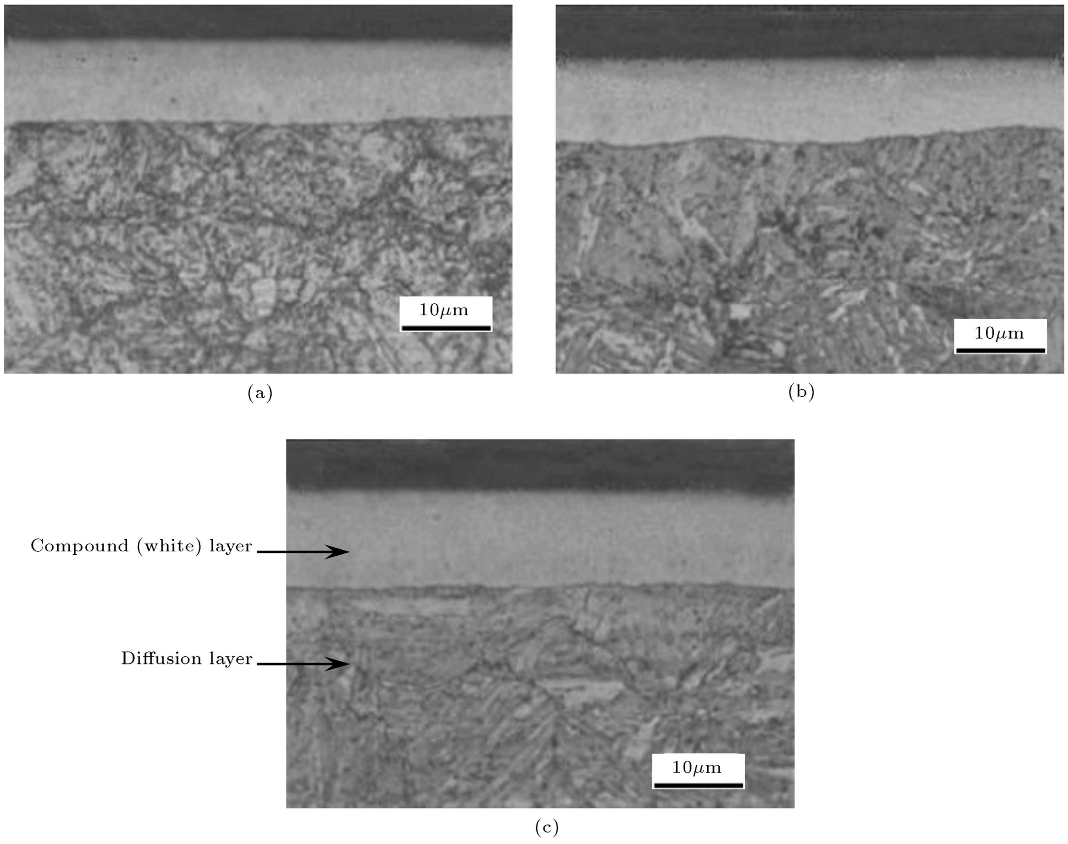
Figure 3. X-ray diffraction patterns of through cage plasma nitrided samples at 550 and 580°C for 5 hr for screens with (a, c, e, f) 6 mm diameter and (b, d) 8 mm diameter with (a, b, f) screen top lid and (c, d, e) iron plate top lid for %75 N_2 in gas mixture.

structure is according to a binary Fe-N phase system. The studies by B. Edenhofer show that the compound layer generated under these conditions must consist of ε and γ' phases [11]. Thus, both ε and γ' phases have been observed in X-ray diffraction analysis. These patterns (Figures 3 and 4) show that screen set-up parameters can be effective in facilitating of active particle transition toward the interior of the screen set-up and around the samples, and an increase in temperature may directly increase the sputtering rate (which causes an increase in nitrogen ions and nitride active species) on the screen cylinder. On the other hand, since the ε phase solves more nitrogen in comparison with the γ' phase with the temperature and nitrogen volume percentage increasing to facilitate the diffusion process [11], the intensity of the ε phase is more than the γ' phase in XRD patterns. An increase in compound layer thickness will increase the peak intensity of the ε phase due to the greater formation of this phase, and its improved detectability by X-ray (Figures 3 and 4). On the other hand, the comparison of phase variations observed in the results of the



(a) (b)

Figure 4. X-ray diffraction patterns of through cage plasma nitrided samples at 550°C for 5 hr for screens with (a) 6 mm diameter and (b) 8 mm diameter with screen top lids for %25 N_2 in gas mixture.



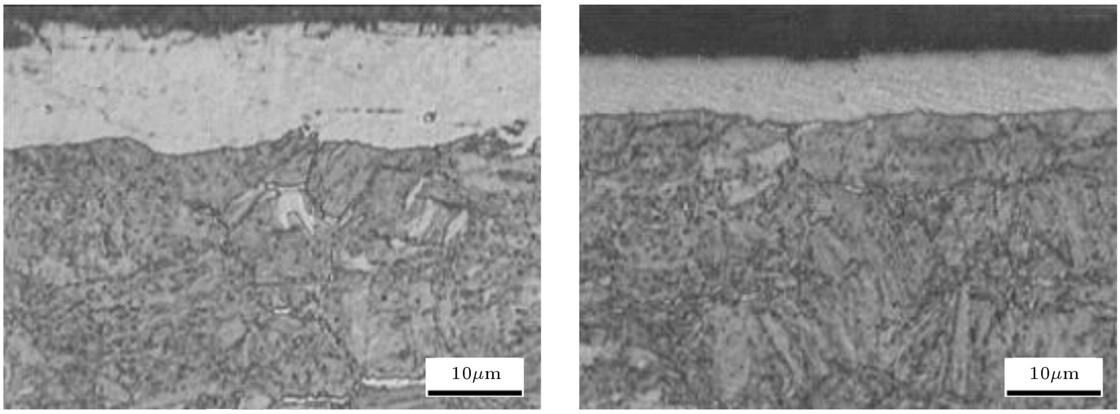
(a) (b)

(c)

Figure 5. Optical microstructures of treated samples in condition with %75 N_2 in gas mixture at 550°C and screen top lid for screens with (a) 8 and (b) 6 mm diameter and also (c) treated sample at 580°C and iron plate top lid by screen with 6 mm diameter.

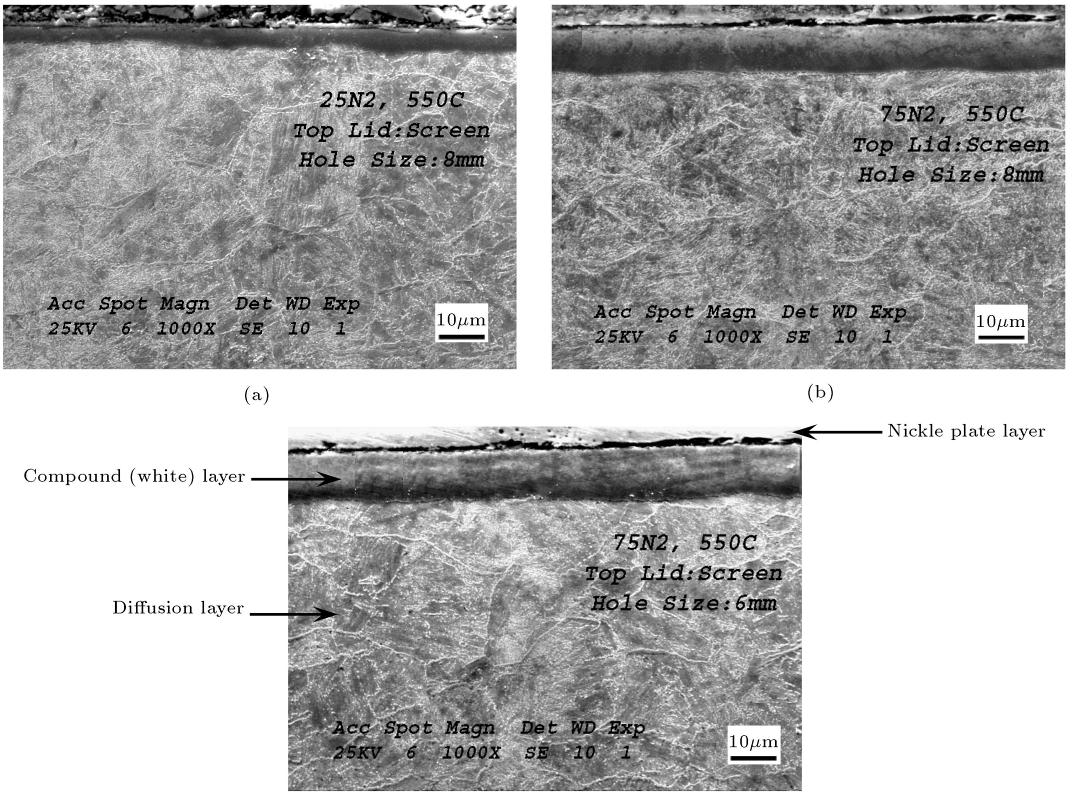
XRD analysis in Figures 3 and 4 confirms the results obtained from microscopic images. One of the proposed mechanisms in the formation of the compound layer is the sputtering and recondensation mechanism [12]. When the energetic ions present in the plasma hit the screen surface around the specimen, first the iron atoms are sputtered from the screen surface and, then, an iron nitride phase (FeN) is formed and recondensed on the screen surface due to a clash between iron ions and nitrogen ions around the surface. Some of these iron nitrides are precipitated on the screen surface, some others pass through the cylindrical screen holes and also through screen top lid holes and are subsequently precipitated on the surface of the treated specimen (Figure 10) [4,13].

Under these conditions, since the sputtering pro-



(a) (b)

Figure 6. Optical microstructures related to the treated samples at 550°C, %75 N_2 in gas mixture with iron plate top lid for screens with (a) 8 and (b) 6 mm diameter.

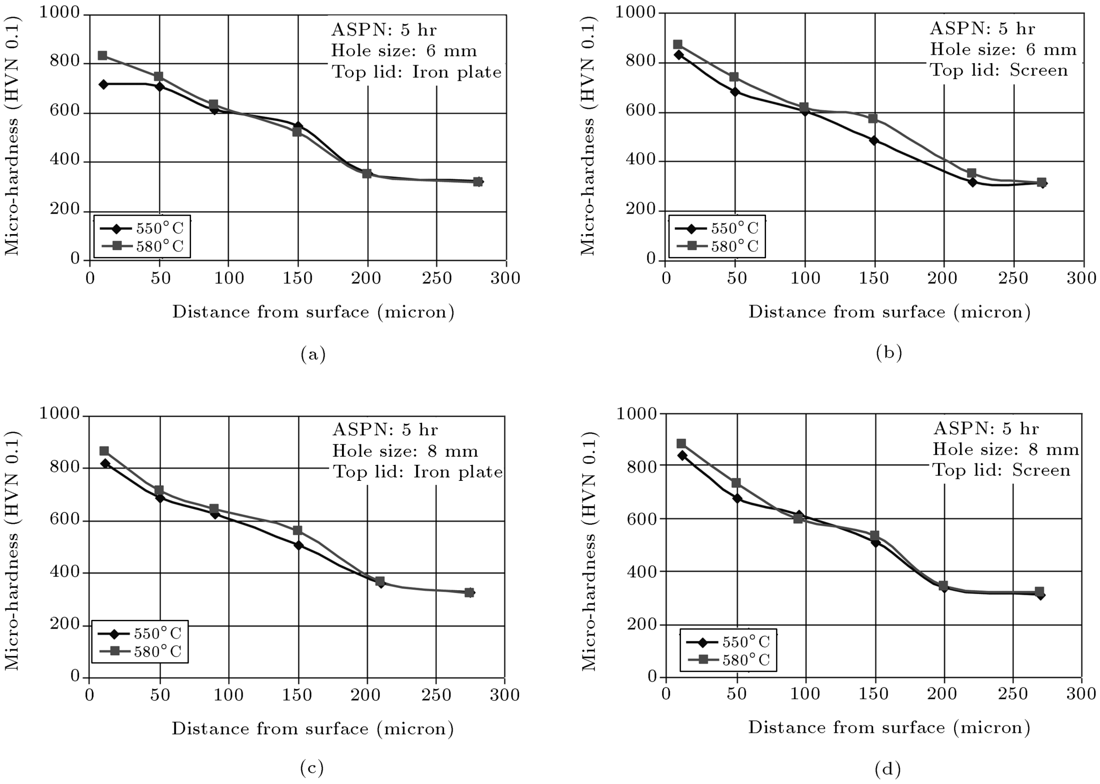


(a) (b)

(c)

Figure 7. SEM images of treated samples at 550°C with %25 and %75 N_2 in gas mixture for screens with (a, b) 8 and (c) 6 mm diameter with screen top lid.

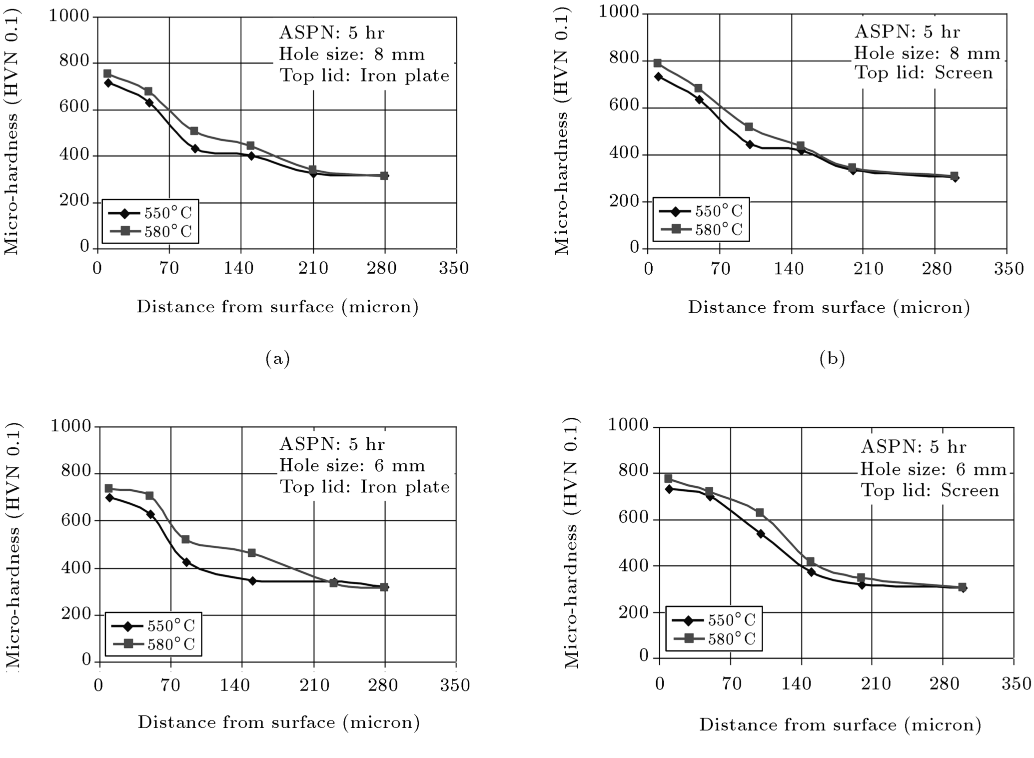
cess does not occur continuously on the sample surface, the concentration of iron nitrides on the sample surface increases. These particles (the iron nitrides (FeN) produced in the plasma) have a theoretical nitrogen content of 20.05 percent by weight. The FeN nitride, which is condensed on uncooled cathodic workpiece surfaces (550 and 580°C), is unstable and decomposes into the lower nitrides Fe_2N, Fe_3N, Fe_4N [11] and, thus, the atomic nitrogen that is liberated in this process will diffuse into the workpiece [14]. On the other hand, due to the reprecipitation of the iron nitride phase (FeN) on the sample surface during the process, the particles created from the sputtering on the screen surface consist of iron nitrides, Fe_xN ($x > 2$), rather than iron atoms [4,15]. Zhao et al. [15] observed N^+ ions and neutral nitrogen molecule



(a) (b)

(c) (d)

Figure 8. Micro-hardness profiles of treated samples at 550 and 580°C in %75 N_2-%25 H_2 for screens with (a, b) 6 and (c, d) 8 mm diameter with iron plate and screen top lids.



(a) (b)

(c) (d)

Figure 9. Micro-hardness profiles of treated samples at 550 and 580°C and %25 N_2 in gas mixture for screens with (a, b) 8 and (c, d) 6 mm diameter with iron plate and screen top lids.

Table 2. Compound layer thickness as a function of temperature, gas mixture and screen set-up parameters.

Temperature (°C)	**Screen Set-up Parameters**		**Compound Layer (μm)**	
	Top lid Type	**Screen Hole (mm)**	**For $N_2/H_2 = 3/1$**	**For $N_2/H_2 = 1/3$**
550	Iron plate	6	6	2.5
550	Iron plate	8	13	3
550	Screen	6	10	4
550	Screen	8	10	5
580	Iron plate	6	13	7
580	Iron plate	8	13.5	10.5
580	Screen	6	14	11.5
580	Screen	8	14.5	12

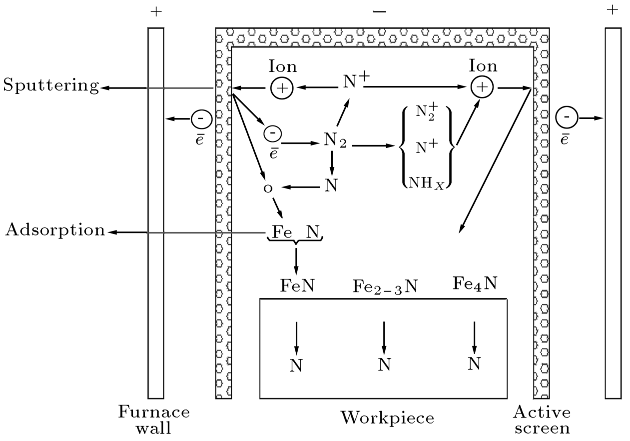


Figure 10. Schematic mechanism of mass transfer and interactions in through cage plasma nitriding process at the first of the treatment.

lines inside the steel screen by Optical Emission Spectroscopy (OES) examination (inserted diagram at Figure 11) [10].

Their OES results indicated that NH and neutral Fe may not be critical species in through cage plasma nitriding. Accordingly, with an increase of N_2 in the gas mixture and/or an increase in treatment temperature, increasing the amount of neutral species inside the screen and, subsequently, increasing the adsorption of active nitrogen atoms on the metastable sputtered FeN, these species may change to stable Fe_xN ($x > 2$) particles around the sample, and rich particles deposited on the sample surface. Iron nitrides (Fe_xN) sputtered from the screen surface absorb the atomic nitrogen released in the plasma, both physically and chemically, and are translocated to the specimens in the form of nitrogen-rich iron nitrides. Following a clash of these nitride particles with the surface of the sample that possesses a higher surface heat, some atomic nitrogens physically absorbed by the iron nitrides are separated and diffused toward the lower levels of the sample and, then, due to interaction with iron atoms and other alloy elements are precipitated as fine dispersed iron nitrides or alloy elements thus forming a diffusion layer. Other free active atomic nitrogens are chemically absorbed by iron nitrides, bind with them, and form iron nitrides, $Fe_{2-3}N$ and Fe_4N, on the sample surface [15,16] (Figures 11 and 12). Figure 12 shows the electron microscopy images of the surface of the treated samples, as well as the formation of hexagonal ε-$Fe_{2-3}N$ nitrides, which have homogeneous boundaries [4,16] due to the lack of a continuous sputtering process.

It is worth noting that an increase in screen hole diameter or the use of a screen top lid will lead to an improvement in the free movement of active plasma species with N_2^+ ions, neutral nitrogen molecules and atoms [6,13] and, especially, nitrogen-rich iron nitrides, Fe_xN, from the lateral surface of the screen and the upper surfaces of the top lid toward the specimen surface [16]. On the other hand, the development of the compound layer depends on the sputtering intensity on the screen surface. The sputtering intensity on the screen surface correlates positively with temperature due to the diffusion possibility, as well as greater interaction between active nitrogen atoms and screen iron atoms (owing to the diffusible nature of the plasma nitriding process) and also due to an increase in the %volume of nitrogen in the gas mixture, which is itself due to the greater atomic mass of nitrogen compared to that of hydrogen. For this reason, active nitrided species show a more effective movement toward the samples placed in the active screen. Compared to the γ' phase the ε phase solves more nitrogen content.

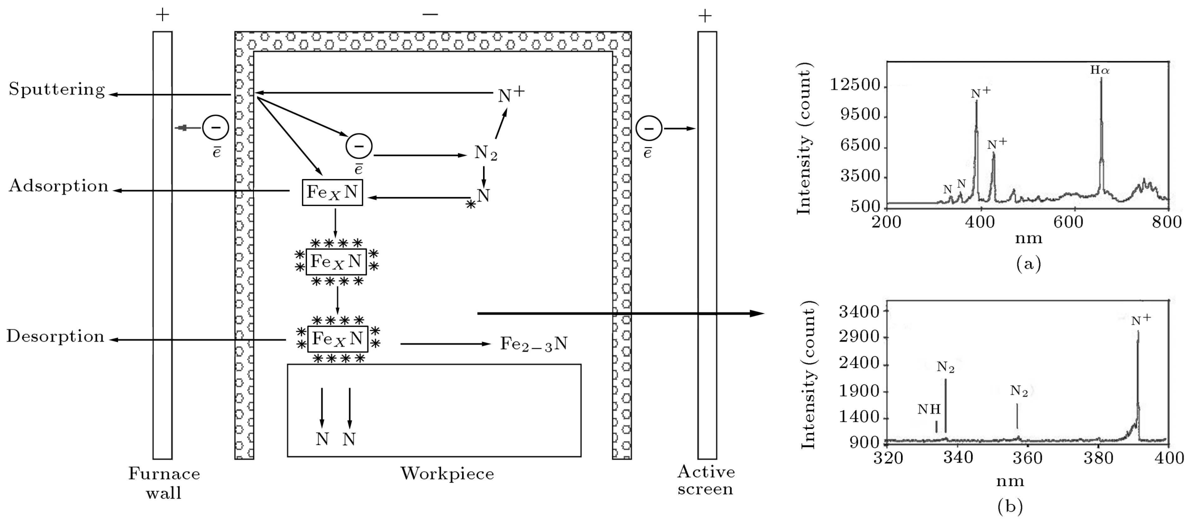
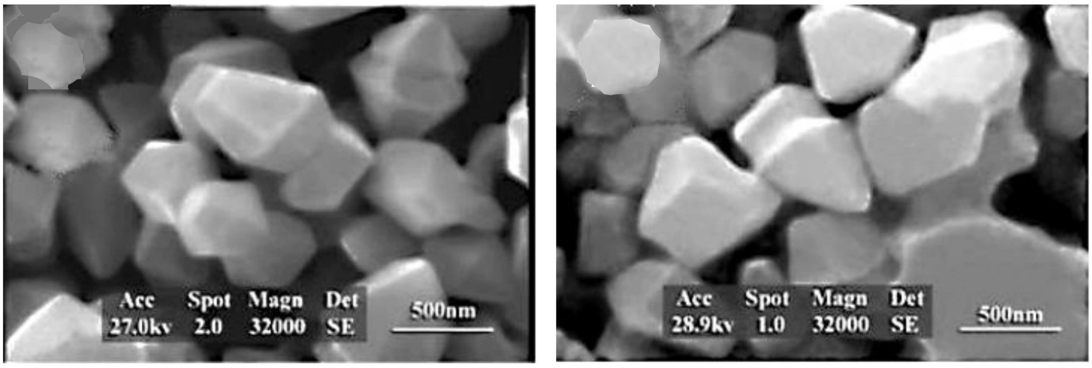


Figure 11. Schematic mechanism of mass transfer and interactions during through cage plasma nitriding process.



(a) (b)

Figure 12. Morphologies of through cage nitrided sample surfaces: 550°C in %75 N_2-%25 H_2 for screen with 6 mm, with (a) screen and (b) iron plate top lid.

Therefore, with an increase in screen hole diameter, the use of a screen top lid, an increase in temperature and, finally, an increase in %volume of nitrogen, XRD results show a higher concentration for the ε phase in the compound layer (Figures 3 and 4). An increase in ε-iron nitride phase concentration, and formation of these hexagonal nitrides with regular distribution and well-defined boundaries, will increase the compound layer thickness owing to the fact that there is a positive correlation between the compound layer thickness and the values of the generated nitride phases (Figures 5-7 and Table 2). On the other hand, there is a positive correlation between the hardness of the nitrided surface and the values of the nitrided phases in the compound layer (especially the ε phase having a h.c.p crystallographic structure and limited slip systems, which microstructurally lies in the upper surface of the sample above the γ' layer) (Figures 11 and 12) [11]. Therefore, under these conditions, the surface hardness tends to increase (Figures 8 and 9). An increase in %volume of nitrogen in the treatment gas composition results in a higher nitrogen diffusion into an iron lattice, since both the concentration of nitrogen atoms near the surface and the concentration slope go up. Also, because the nitriding process is temperature-dependent, it follows that an increase in the temperature will lead to a higher movement of atoms and to a more rapid nitrogen diffusion into the iron lattice. Therefore, when these two parameters are increased due to a greater nitrogen diffusion into the α iron lattice and the higher nitrogen solubility in this lattice, interstitial compounds are formed; leading to a distortion in the structure of the α iron lattice [17]. All this, together with the formation of dispersed carbonitride precipitates results in an improvement in the hardness and thickness of the α iron lattice. It is, therefore, seen that by increasing the temperature of the treatment and also by increasing the nitrogen volume in the gas mixture, the slope of the hardness profile is reduced from the nitrided surface toward the core. It should be noted that the trend of hardness variations is influenced by alloy elements, the intensity of interaction among them and also by the nitrogen volume in the steel [1]. However, with regard to the low percentage of alloy elements in the steel under investigation in the present study, and also with respect to less interaction between these elements and nitrogen, the slope of the hardness profile has declined steadily and slowly (Figures 8 and 9).

CONCLUSIONS

1. Owing to the presence of nitrogen in the treatment chamber, the phases generated during through cage nitriding are γ' and ε iron nitride phases.
2. The value and intensity of the ε phase peak with larger screen diameters and under screen top lid conditions are higher due to the free and more effective movement of active plasma species from lateral and top screen surfaces and the greater solubility of nitrogen in the ε phase compared to that of the γ' phase.
3. Increasing the temperature and nitrogen percent in the treatment gas will increase the compound layer thickness because of diffusivity and greater interaction between active nitrogen atoms and iron atoms.
4. An increase in the screen hole diameter under iron plate top lid conditions will lead to an increase in the compound layer thickness, but increasing this parameter under screen top lid conditions has little effect on the compound layer thickness. This is due to the improved and greater movement of active plasma species toward the samples, following an increase in screen hole diameter.
5. The surface hardness of the treated samples in ASPN under screen top lid conditions is slightly higher than that under iron plate top lid conditions. This is due to the greater movement of iron nitrides from the top lid holes and their greater precipitation on the sample surface.
6. An increase in the treatment temperature and %volume of nitrogen in the gas mixture will lead to an increase in surface hardness. This is due to an increase in the diffusion of nitrogen into the iron lattice following the formation of nitride precipitates and interstitial compounds.

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