INVESTIGATION NITRIDE LAYERS AND PROPERTIES SURFACES ON PULSED PLASMA NITRIDED HOT WORKING STEEL AISI H13

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Abstract: *Nitriding is a surface treatment technique used to introduce nitrogen into metallic materials to improve their surface hardness, mechanical properties, wear resistance and corrosion resistance. In this research, the effects of plasma nitriding parameters including frequency and duty cycle were investigated on samples with different grooves dimensions. Steel blocks prepared from DIN1.2344 hot working steel were plasma nitride at 500 °C under the atmosphere contents of %75H_2-%25N_2, the duty cycles of 40%, 60%, 80%, and the frequencies of 8, 10 kHz for 5 hours. Then characteristics and micro hardness's of the nitrided samples were investigated using SEM, XRD, and Vickers Micro Hardness method. The results of the experiments indicated that with increasing frequency, the duty cycle, and the thickness of the grooves, the roughness of the surfaces increased. With an increase in duty cycle from 40% to 80%, the hardness of the surface rose and the thickness of the compound layer built up. Hollow cathode effect occurred in the samples with small grooves and high duty cycle in plasma nitriding. This will result in over heating of the sample which leads to a decrease in the slope of hardness values from the surface to the core of the sample and also a decrease in the diffused depth of nitrogen. The compound layer of the treated samples consisted of γ΄: Fe4N and* **ε**: *Fe2-3N phases and the proportion of the ε to γ΄ increased with the decrease in the duty cycle. Increasing the frequency did not affect the proportion of phases and micro hardness of the samples.*

Keyword: *Pulse Plasma Nitride, Frequency, Duty Cycle, Hot Work Steel AISI H13.*

1. INTRODUCTION

Nowadays, AISI H13 is one of the most famous hot-work tool steels and dies, which is widely used as die casting and hot forming dies [1]. High toughness, high hardenability, good machinability, low distortion during heat treatment and low cost have made this steel as avery useful tool steel [1]. Plasma nitriding is an advanced surface modification technology that has experienced industrial components to improve their surface properties [2, 8]. Nitriding steel part of different shapes and size, such as for crankshafts, gears and injection nozzles, result in non-uniform nitrided layer for industrial to get good results necessary to carefully choose and secure the treatment condition [3, 7]. There are many kinds of plasma nitriding, such as pulsed plasma nitriding [4, 5], Low pressure, and plasma nitriding duplex treatments [6, 11]. Because of glow discharge in a gas mixture of N2 and H2, with the cathode at a special range of temperature, atomic nitrogen can form and penetrate in to surface steel. Depending on the concentration and type of alloying element and the process parameters, a diffusion zone is formed, when nitrogen penetrate in to the surface steel. Moreover a surface compound layer is also formed on top of diffusion zone. These compounds layer are called white layers because they appear white on surface [9, 10]. The hollow cathode discharge effect is a special glow discharge in a hole of a block. The important influences on this effect are the gas pressure, the size of the hole and the plasma density. The discharge can penetrate in to hole and the gas in hole will be overheated to extremely high temperature [12]. The pulsed source enables the temperature of the specimen to be controlled by simply adjusting the width of the pulse, ton, without changing the bias voltage. Even though plasma rectors equipped with a pulsed power supply have been available for over a decade, only recently have extensive studies been published regarding the effect of the pulse aspect on the final properties of the nitride layers [13].

The main purpose of the research is to study and survey the effect of nitriding parameters including frequency and duty cycle on samples with different grooves.

2. EXPERIMENTAL

Several sample assemblies including steel blocks with dimensions of 30× 40× 60 mm^3 were produced and in each block a rectangular groove with dimensions of 2, 4, 6, 8 and 10 (W) × 40 (H) × 20 (L) mm^3 was produced by machining. For each block a plate (substrate) from hot work steel AISI HI3, DIN1/2344, with dimensions of 10× 40× 60 mm^3 was prepared to cover the groove and to complete the sample assembly, Fig. 1. The surfaces of the substrates (Fig.1b) were mechanically polished before being placed in the chamber. To measuring surface temperature, a hole has been drilled on the sample for installation of thermocouple; Fig.1c. The surveyed surface in the research is the determined surface in section Fig. 1b. After preparing the samples, we were austenitized them by putting them in the furnace at the temperature of 1050 °C for 1 hour. Afterwards the samples were immersed in oil. Then, samples were tampered during of 1 hour at 530 °C . The surfaces of the samples were mechanically polished before being placed in the chamber. The samples were nitrided by a plasma nitriding system of pulsed–DC. Samples were plasma nitrided under an atmosphere of %75 H_2–%25N_2 in 500 °C for 5 hours with the frequencies of 8 and 10 kHz and in the duty cycles of %40, %60, and % 80. The properties of the nitrided sample were investigated by evaluating the phase composition, the thickness of the compound layer, the profile micro hardness, and case depth using Scanning Electron Microscopy (SEM), X-ray Diffraction (XRD) and Vickers Micro Hardness method. the samples were cut from the groove edge to the end of the groove with the distance of 1cm after nitriding treatment, the

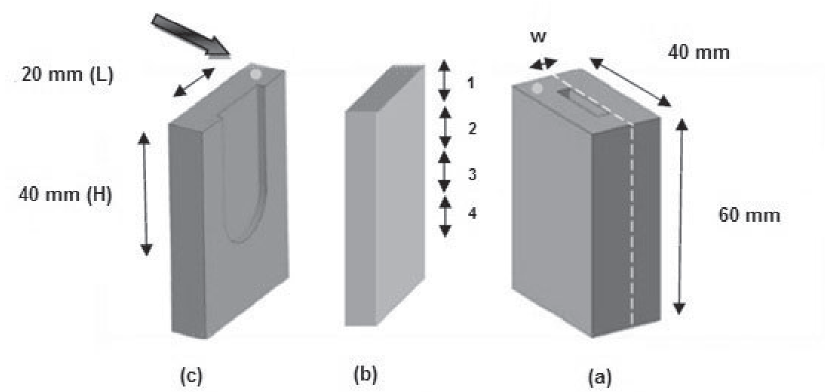

Fig. 1. (a) The sample assembly (b) investigated surface and (c) grooved part.

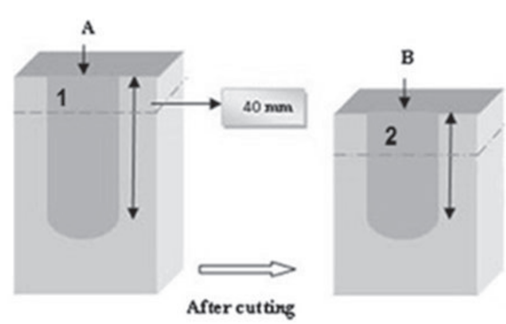

Fig. 2. Different investigated regions of the groove.

surface of the grooves are called (1), (2), (3), (4). Also, the cross surfaces are called surface A and surface B, as shown in Fig. 2.

3. RESULTS AND DISCUSSION

The layer thickness measurement data for samples, treated with different dimensions of the groove are presented in Table 1. From the data in Table 1, it can be understood that the thickness of the compound layer increases with increasing the width of the groove. The thickness of the compound layer is controlled by nitrogen concentration at a static temperature in the pulse plasma nitriding method [14]. The thickness of the compound layer increases by the increase in the nitrogen in grooves, which itself is caused by an increase in the penetration of nitrogen atoms under the layer; as shown in Fig. 3 [15]. The thickness of the compound layer in pulse plasma nitriding methods depends on the samples and the density of the activity like: nitrogen ion, radical ion, and the composition [16, 17]. The thickness of the compound layer of surface A is more than that of surface B; as shown in Fig. 2 [18]. As it is observed, the thickness of the compound layer increases by the increase of the duty cycle because more nitrogen atoms are ionized, and more N is produced and this results in more bombardment on the surfaces of grooves by more atoms. Also, According to Table 1 it has been observed that an increase in the frequency from 8 kHz to 10 kHz does not have much effect on the thickness of compound layer. The thickness of the compound layer rises slightly with increasing the frequency. The ranges of the layer thickness variation are 3.7-4.1 µm for the groove of 2 mm and 8.4-10.6 µm for the groove of 10 mm [19].

Fig. 4 (a-d) shows the SEM cross section images of the samples treated with the pulse

Table1. Thickness of the compound layer of different samples (µm)

Duty Cycle		80%				60 %				40%			
Frequency		10 kHz		8 kHz		10 kHz		8 kHz		10 kHz		8 kHz	
Surface		A	B	A	B	A	B	A	B	A	B	A	B
Width of the groove (mm)	2	3.8	0	3.7	0	4.4	0	4.2	0	4.1	0	3.9	0
	4	5.1	4.4	4.7	4.4	4.7	4.3	4.5	4.2	4.4	3.8	4.3	3.6
	6	6.8	6.3	6.7	6.1	….	….	…..	….	5.5	4.7	5.4	4.3
	8	9.8	7.8	9.5	7.4	….	….	….	…	8.1	7.3	7.8	7.4
	10	10.6	8.4	10.1	8.2	…	….	….	….	8.7	8.1	8.4	7.6

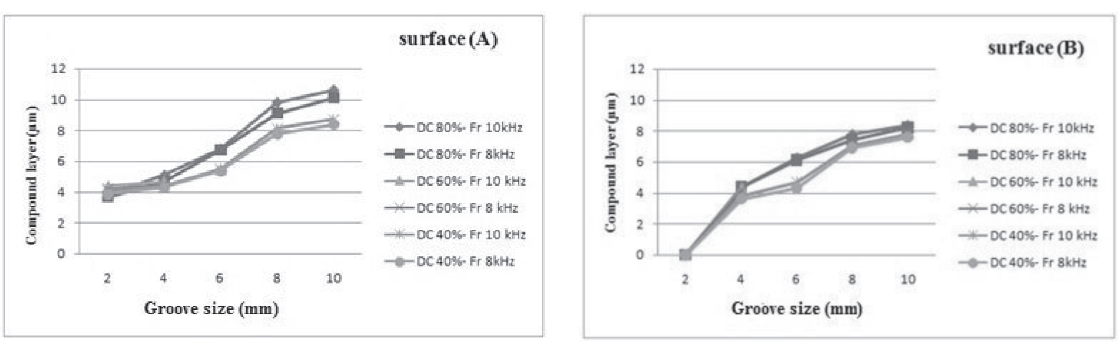

Fig. 3. Results the thickness of the compound layer with changing the width of the grooves at different conditions of nitriding plasma at the cross section surfaces A and B.

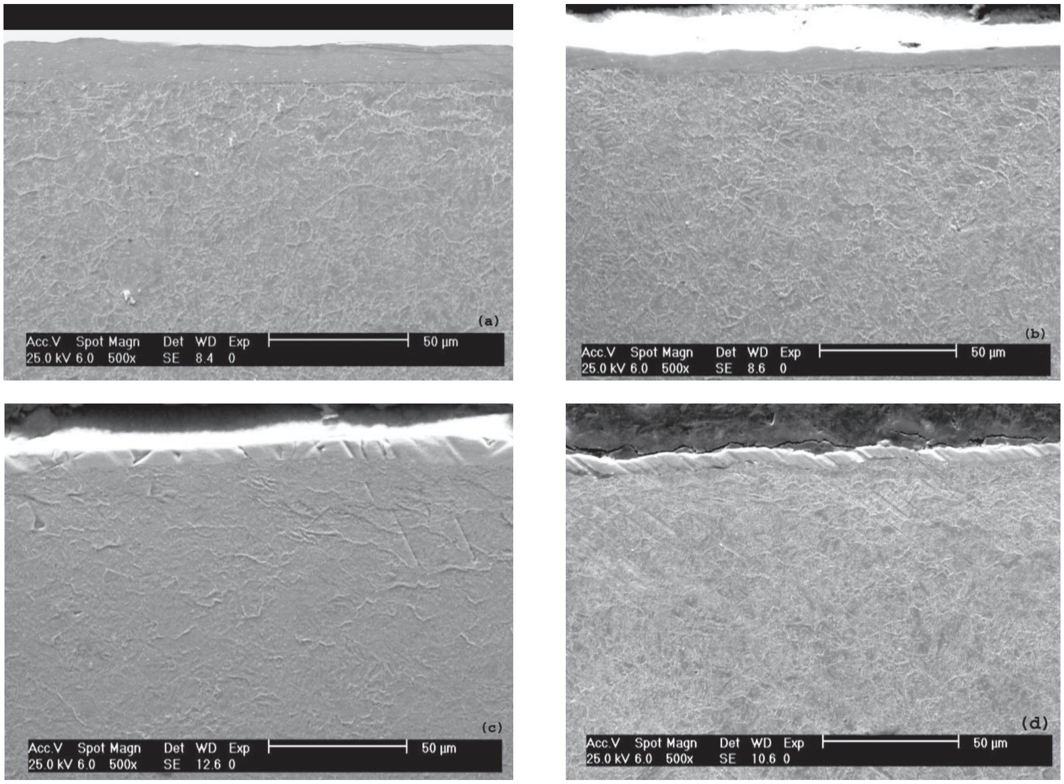

Fig. 4. SEM cross section images of samples with groove 4mm and treated plasma nitriding at (a) duty cycle 80%, frequency 10 kHz, (b) duty cycle 80%, frequency 8 kHz, (c) duty cycle 40%, frequency 10 kHz, (d) duty cycle 40%, frequency 8 kHz.

plasma nitriding method with dimension of the groove 4mm, at the temperature of 500 °C , duty cycles of 40%, 80%, and the frequencies of 8 kHz, 10 kHz that are shown in compound layer. According to Fig. 4 and Table 1, it can be concluded that increasing the sputtering of the surface of the samples is due to increasing the thickness of the compound layer [18].

Fig. 5 (a-d) shows the micro hardness profile of the nitriding samples at 500 °C for 5 hours in different frequencies and duty cycles. Micro hardness profiles obtained from cross-sections of treated specimens show presence of a slope interface between the case (nitride layer) and the core. All samples show high surface micro hardness values that drop decreasingly at the case/ core interface to the substrate micro hardness values [13]. The gradient is created by the penetration of nitrogen atoms from the surface to the steel core. The sudden decrease in the hardness shows the depth of nitrogen penetration [8]. It can be observed that hardness increases with increasing width of the groove. In the larger grooves, more nitride precipitate forms and more hardness increase occurs [18]. Also, Fig. 5 shows that frequency does not have much effect on the micro hardness cross section by increasing frequency just a bit, the micro hardness of the samples reduces from the surface to the core; and with an increase in the duty cycle from 40% to 80%, the amount of the surface hardness rises from 1160-1240 HV to 1280-1330, due to more bombardment of the surface by ionized nitrogen and increasing sputtering of the surface [19]. Considerable phenomena occur in the samples with the duty cycle of %80 and width of the groove 2 mm. This will result in over heating of the sample which leads to a decrease in the slope of hardness values from the surface to the core of the sample and also a decrease in the diffused depth of nitrogen in the duty cycle of 80% rather than the duty cycle of %60 and %40

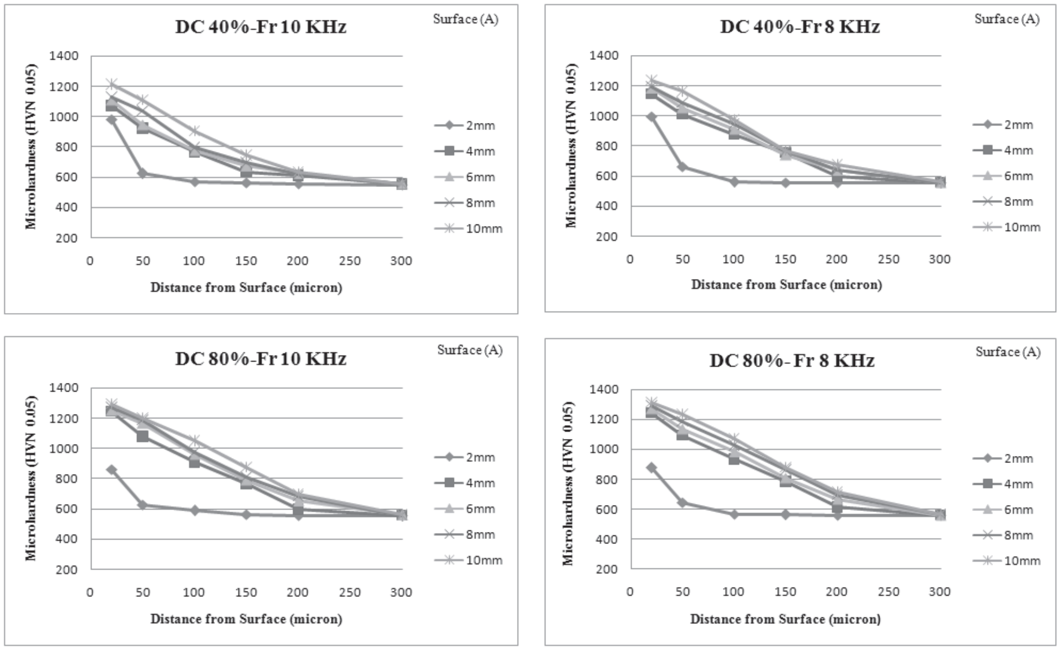

Fig. 5. Micro Hardness profiles of the plasma nitriding pulse treated samples from surface A.

at a groove thickness of 2 mm. The reason can be over heating under hollow cathode phenomena in which plasma has been overlapped in two sides of the groove, due to the groove getting thinner and this results in electrons getting caught and more ionization [20, 16].

In Fig. 6 (a-d), we can observe that the surface of the plasma nitriding samples is covered by cauliflower form of particles. This morphology has been observed and reported before by the researcher [14], [21]. These observations determined that the cauliflower form of the particles in plasma nitriding is due to sputtering of the surface during the process. Also, the size of the cauliflower form of the nitride particle is very coarse and large in treatment of the duty cycle of %80 and it is very small in the duty cycle of %40. Therefore, we can see that the nitride particles become larger, and the steel surface becomes rougher and uneven by an increase in the duty cycle; and by its reduction, the surface becomes smoother and the nitride particles become smaller. In addition, nitride particles become a bit course and the surface becomes rougher and more uneven by an increase in the frequency. As it is observed, the effect of frequency on nitride particles sizes on the surface is lower than duty cycle [10].

XRD patterns for the plasma nitriding treated samples at 500 ˚C are shown in Fig. 7 (a) and (b), present the existence of ε , the phase rich in nitrogen, and γ' phases in all compound zones on the surface of all the grooves. This figure indicates that while the width of the groove increase, the ratio of ε to γ' and the thickness of the compound layer increase. This can be due to the presence of nitrogen, increasingly penetrating into the groove because of the increased width of the groove. Also, the surface sputter increases by increasing the width of the groove and the percentage of the built-up nitride ε in the composition layer disappears due to the sputter [19].

XRD patterns for the plasma nitriding treated samples at 500 °C , in the duty cycles of 40% and 80%, the frequency of 8 kHz, with width of the groove of 4 mm are shown in Fig. 8 (a) and (b). As it is observed ε, the phase rich in nitrogen, decrease by an increase in the duty cycle, unlike the γ′ phase which increases due to the duty cycle increase which causes more bombardment of the surface by ionized nitrogen and an

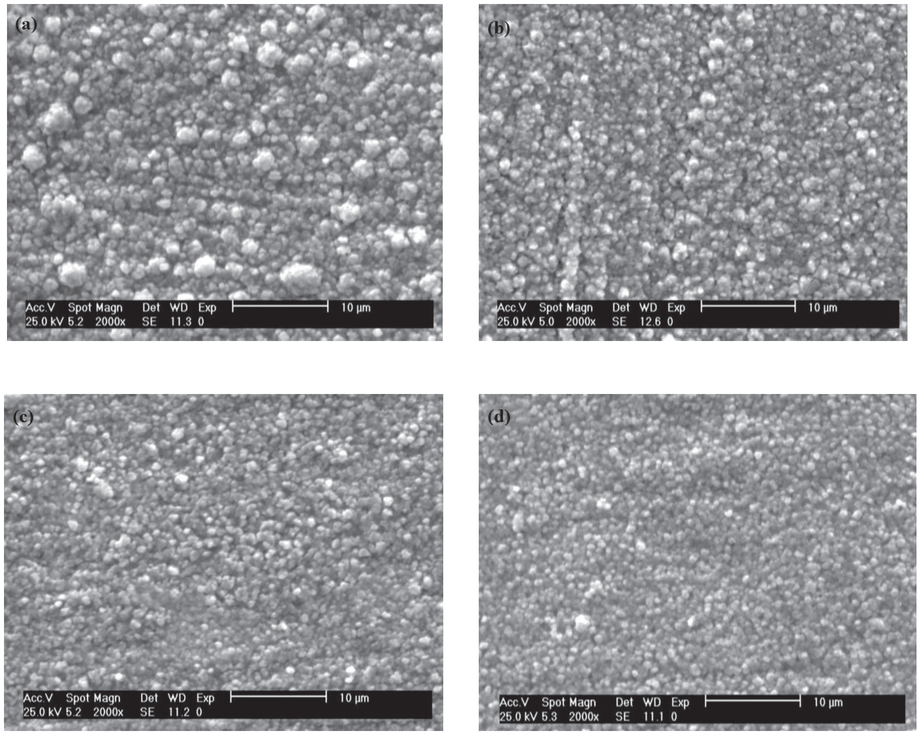

Fig. 6. SEM topography images of treated samples at (a) 80% duty cycle, 10 kHz frequency, (b) 80% duty cycle, 8 kHz frequency, (c) 40% duty cycle, 10 kHz frequency, (d) 40% duty cycle, 8 kHz frequency.

escalation in sputtering the surface [22]. Also carbon helps to form the ε nitride layer. In the lower duty cycles, surface decarburizing occurs to a small extent due to the low carbon penetration and there is sufficient carbon for the formation of ε. There is considerable surface decarburizing in the higher duty cycles that result in a decrease in surface carbon. Therefore, γ′ is formed in lower nitrogen density. The reduction of ε nitride in the compound layers of the operated samples in higher duty cycles can be attributed to the reduction carbon range on the surface under the sputtering process [19]. Also we can observe that frequency does not have much effect on the phases changing [13].

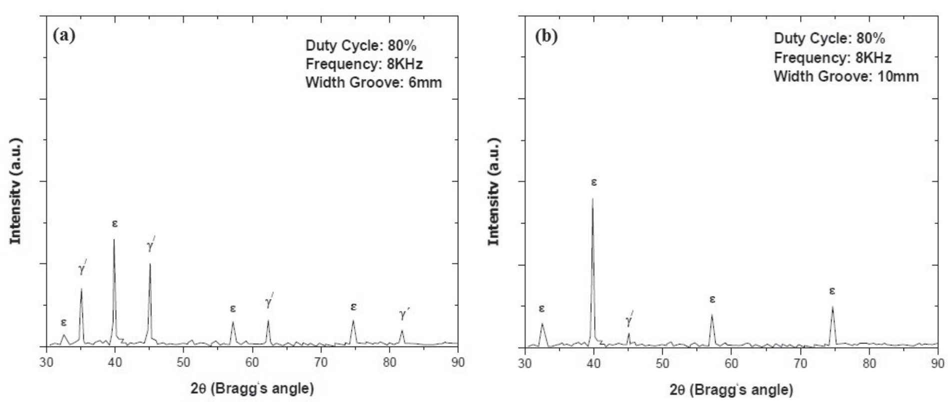

Fig. 7. XRD patterns of treated samples at duty cycle 80%, frequency 8 kHz and with different grooves (a) 6 mm and (b) 10mm.

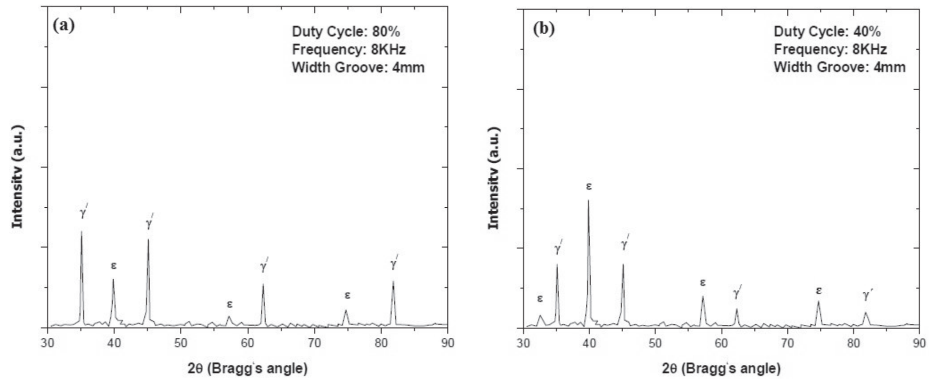

Fig. 8. XRD patterns of treated samples at frequency 8 kHz, with groove 4mm and at duty cycles (a) 80% and (b) 40%.

4. CONCLUSIONS

1. The thickness of the compound layer increases with increasing the duty cycle from 40% to 80 % and with increasing the thickness of the groove from 2 to 10 mm. Also it rises slightly by an increase in the frequency from 8 to 10 kHz.
2. Surface layer at the regions of the groove that are remote from the edge is thinner than that of closer regions.
3. The micro hardness and the case depth increase with increasing the duty cycle from 40% to 80% and with increasing the thickness of the groove from 2 to 10 mm. Also, a slight decrease may happen with increasing the frequency from 8 to 10 kHz.
4. Hollow cathode effect occurred at the duty cycle of 80% and with the groove of 2 mm in pulse plasma nitriding leading to a decrease in the micro hardness of the surface to the core and decreases the depth of nitrogen diffusion. Additionally, it increases the roughness of the surface.
5. The roughness of the surface increases with an increase in the duty cycle and frequency which, in turn, causes nitride particles to become course and large due to more roughness and unevenness of the surface.
6. The compound layer of the treated samples consisted of γ' and ε phases. The proportion intensity of γ' to ε phase increased by raising the duty cycle and the decreasing thickness of the grooves. Also, frequency does not have much effect on the phase ratios.

5. ACKNOWLEDGEMENT

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