Chapter 5

Plasma Nitriding of High Carbon Low Alloy Steel

5.1 Introduction

In this chapter the results of plasma nitriding on AISI 52100 ball bearing steel have been reported. The effect of heat treatments (quenching and tempering) on such steel after plasma nitriding is studied. Three different types of ball bearing races, i.e. annealed, annealed-quenched and annealed-quenched-tempered were selected for plasma nitriding. Plasma nitriding on such steel were performed with the variation of plasma nitriding process parameters such as; plasma nitriding process temperatures, process time, gas compositions and gas mixture to see the effect on surface properties.

A brief introduction to the high carbon low alloy steels, technical disadvantages of the AISI 52100 ball bearing steel, details about the chemical composition and samples preparation and obtained results are described in the subsequent sections. Introduction to the high carbon low alloy steel are discussed in section 5.2, while the technical disadvantages/ difficulties with plasma nitriding, pertaining to this particular ball bearing steel are discussed in section 5.3. The details of the chemical composition, different heat treatment processes, sample preparation and the experimental set up are described in section 5.4. The results of the effect of various process parameters on AISI 52100 ball bearing steel are discussed in section 5.5. Section 5.5.1 represents the effect of plasma nitriding process temperature on surface micro-hardness. Section 5.5.2 represents the effect of plasma nitriding gas compositions on surface micro-hardness. Section 5.5.3 shows metallurgical study and X-ray diffraction analysis of plasma nitrided ball bearing steel. The results of effect of heat treatments (quenching and tempering) on plasma nitrided ball bearing steel samples are presented in section 5.5.4. Overall conclusions of the study are given in section 5.6.

5.2 High Carbon Low Alloy Steel

Classification of steels was made on the basis of carbon content (in wt%) present in the component. If the carbon percentage in steel components varies from 0.6 wt% to 1.0 wt% such steels are known as high carbon steel. However, high carbon concentration reduces some other properties such as, ductility and toughness of the components. High carbon steel is generally used in hardened and tempered condition for various useful applications. For effective use of such steel, some other alloying elements such as, Chromium (Cr), Molybdenum (Mo), Tungsten (W), Vanadium (V), and Nickel (Ni) are intestinally added to increase the mechanical properties (wear resistance and hardness). High carbon steel is generally used to make drills, shear blades, hammers and different kinds of bearings. Some common bearing steels are known as WS 1·3505, EN 100 Cr 6, AISI 52100, AFNOR 100 C 6, CCR-1150 and En31 steel.

5.3 Technical disadvantages of AISI 52100 ball bearing Steel

AISI 52100 bearing steel in hardened and tempered condition with spheroidized carbides structure is the most commonly used material for bearing applications in rotating devices, machinery and automobiles sector [111]. This steel is broadly accepted for the above applications because it has immense combinations of low cost, high hardenability, high yield/tensile strength, high wear resistance, good machinability and formability [112]. However, high wear resistance enhances friction, generates noise/vibration and necessitates an early replacement, which causes premature failure.

For solving these problems, constantly extensive research work is going on to explore new bearing designs and suitable microstructure by changing the chemistry of bearing materials. Researchers are also considering the use of thin hard coatings to improve bearing performance and durability applications [113]. Implementation of surface hardening process on various steels surfaces, make it possible to exhibit stable quality, high wear resistance reliability and longevity during services in various media.

Plasma nitriding is a best suitable approach to resolve such problems [114]. But, such ball bearing steel is not very much suitable for plasma nitriding process. The reason behind this steel is its tempering temperature ~170–200 ^{0}C, which is much lower than the standard plasma nitriding processing temperature needed for the plasma nitriding treatment. In fact, the plasma nitriding is carried out usually on a quenched and tempered material at temperature ~ 460 ^{0}C – 600 ^{0}C. During the plasma nitriding process the process temperature is generally kept ~50 ^{0}C lower than that of the tempering temperature. Due to this fact the plasma nitriding of this specific ball bearing steel is not possible by conventional plasma nitriding mechanism.

5.4 Details of Sample Preparation

Material used for assessment in this study are commercially available AISI 52100 bearing steel races. The chemical composition of this standard steel is given in the Table 5.1.

Table 5.1: Chemical composition of selected ball-bearing steel.

C	Cr	Mn	P	Si	S	Mo	Cu	Ni	Fe
0.98-1.1	1.3-1.6	0.25-0.45	0.025	0.15-0.35	0.020	0.05	0.25	0.25	Balance

Ball-bearing races of diameters ranging from 7-15 cm made of AISI 52100 steel were used in this study. Before plasma nitriding, several types of heat-treatments were carried out on three types of ball-bearing steel races as listed in the Table 5.2.

To study the effect of heat-treatment on plasma nitrided annealed samples, they were heat treated (Q+T) after plasma nitriding. The process of heat treatment (quenching and tempering) was similar as given in the Table 5.2. Different samples were prepared by polishing with SiC emery papers of 240, 320, 400, 600 grit size followed with 1 μm diamond paste and alumina paste of 0.05 μm grain size. After polishing, the prepared samples were rinsed with acetone, washed and dried. The experimental set-up used for the plasma nitriding of ball bearing steel is same as earlier described in chapter 2 section

2.2. The typical process parameters for the plasma nitriding of ball bearing steel are shown in Table 5.3.

Table 5.2: Detail of various heat-treatment processes and corresponding microstructure and surface hardness.

Treatment type	Designation	Conditions	Microstructure	Initial Surface Hardness
Annealed	A	**(Conditions for Annealing)** Heating- 820^{0}C for 4 hrs -710 ^{0}C for 10hrs -680 ^{0}C for 2 hrs Cooling in furnace – till 500^{0}C Cooling in air – till room temperature	Spheroidized carbides	262 HV
Annealed + Quenched	A + Q	**(Conditions for Quenching)** Heating: ~850^{0}C Soaking time: 1 hour Quenching: oil	Martensite, retained austenite and carbides	874 HV
Annealed + Quenched + Tempered	A + Q + T	**(Conditions for Tempering)** Heating: 170-200^{0}C Soaking time: 90 minutes Cooling: up to room temperature	Tempered martensite, retained austenite and carbides	700 HV

Table 5.3: Plasma nitriding process parameters for ball bearing steel.

Expt. No.	**Temperature (°C)**	**Pressure (mbar)**	**Gas (%)**	**Time duration**
1	560 – 570	5	65 : 35 (N_2:H_2)	24 hour

2	560 - 570	5	95 : 5 (N_2:H_2)	24 hour
3	500	5	95 : 5 (N_2:H_2)	24 hour
4	580	3	25 : 75 (N_2:H_2)	4 hour
5	580	3	25 : 75 (N_2:Ar)	4 hour

5.5 Results and Discussion

The obtained results of microstructure study, diffusion depth and surface micro-hardness with variation of plasma nitriding process parameters such as; plasma nitriding process temperature, process time, plasma nitriding gas composition and effect of heat treatment (Q+T) after plasma nitriding are discussed below.

5.5.1 *Effect of Process Temperature on Hardness*

The effect of the process temperature on the hardness of AISI 52100 steel subjected to different heat-treatments is shown in the Figure 5.1.

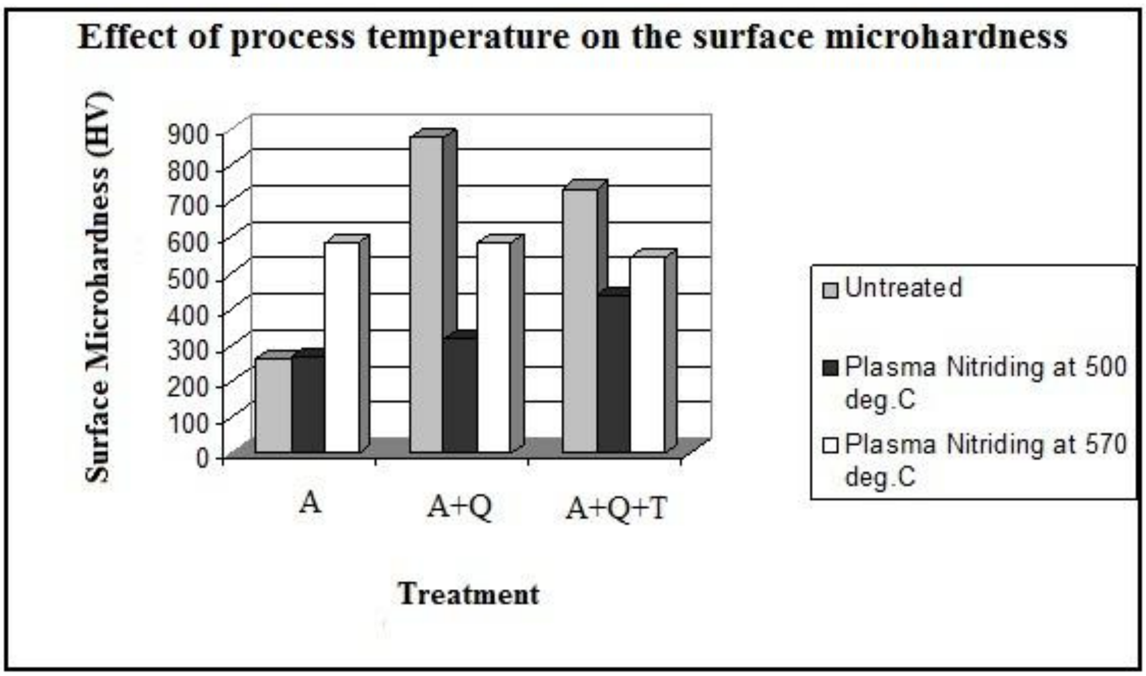


Figure 5.1: Effect of the process temperature on surface microhardness.

It is observed that the surface micro-hardness in the annealed samples increases significantly, from 262 HV to 585 HV when these were plasma nitrided at 570 ^{0}C while at 500 ^{0}C surface micro-hardness not increases significantly. The increasement in the surface micro-hardness at higher temperature as compared to lower temperature is related to the possibility of diffusion of nitrogen and the formation of iron-nitrides compound on the surface. On the other hand the surface micro-hardness decreases ~400-500 HV from bulk hardness ~700 HV in the annealed-quenched (A+Q) and annealed-quenched-tempered (A+Q+T) samples after plasma nitriding at the same processing temperatures 500 ^{0}C and 570 ^{0}C. Behrisch et al. [115] suggested that it could be due to the coarsening of carbides, recovery of dislocation structure and growth of ferrite grains.

5.5.2 Effect of Gas Composition on Hardness

The results of previous subsection clearly indicates that the surface hardness of AISI 52100 steel increases after plasma nitriding only on annealed samples. Therefore, annealed samples were explored for further study. The Figure 5.2 shows the effect of the gas compositions on the surface micro-hardness after plasma nitriding process of the annealed samples.

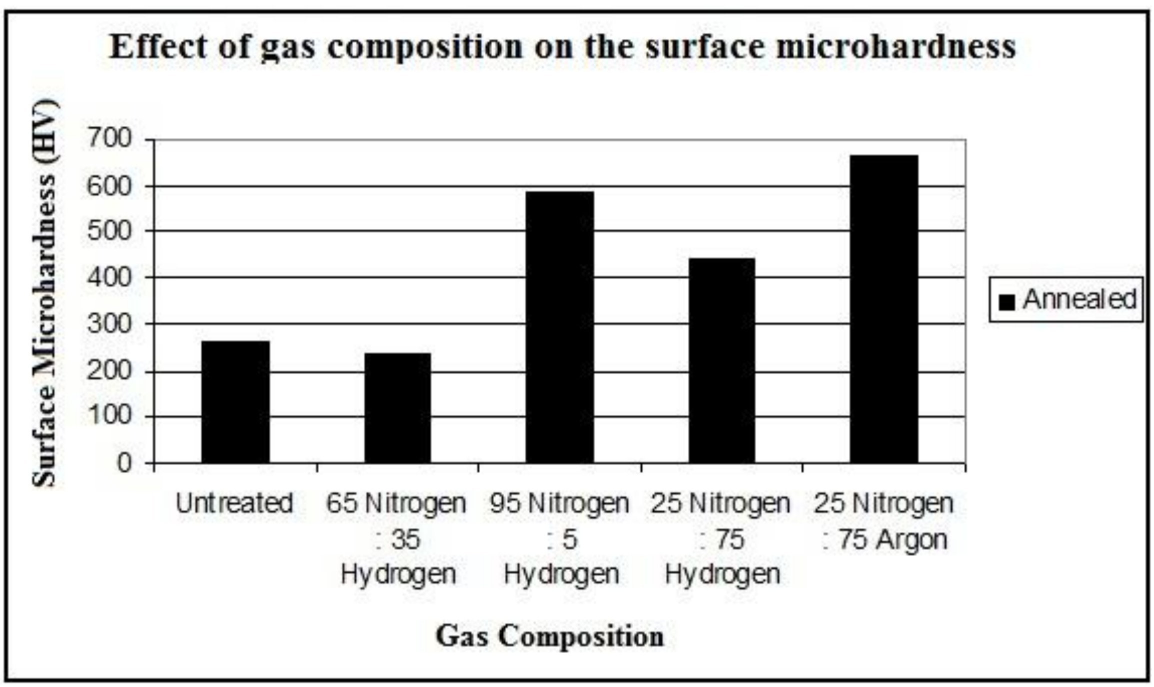


Figure 5.2: Effect of gas compositions on the surface microhardness.

It is observed that the surface hardness increased ~585 HV from 262 HV when samples were plasma nitrided with 95% Nitrogen and 5% Hydrogen gas. On the other hand surface hardness increased from 262 HV to 661 HV when samples are plasma nitrided with 25% Nitrogen and 75% Argon gas mixture. Meletis et al. [116] shows that, the Argon gas has higher sputtering yield as compared to hydrogen gas. Narendra et al. [117] found to Ar-N_2 discharge provide higher concentration of excited and ionized nitrogen on the surface that increase the formation of iron-nitrogen compound. Fancey et al. [118] suggested that, the gas temperature of discharge generally increases as percentage of Ar in Argon–Nitrogen mixture increases. Further, it is also reported that, the Argon gas improve the surface hardness and it may be beneficial in controlling white layer effects and/or modifying surface morphology.

5.5.3 Metallurgical Studies

Figure 5.3 shows the microstructure of untreated annealed sample, which showed the spheroidized carbides structure while figure 5.4 shows the microstructure after plasma nitriding with 25% Nitrogen and 75% Argon which showed a 7 µm thick white layer and 60 µm diffusion zone.

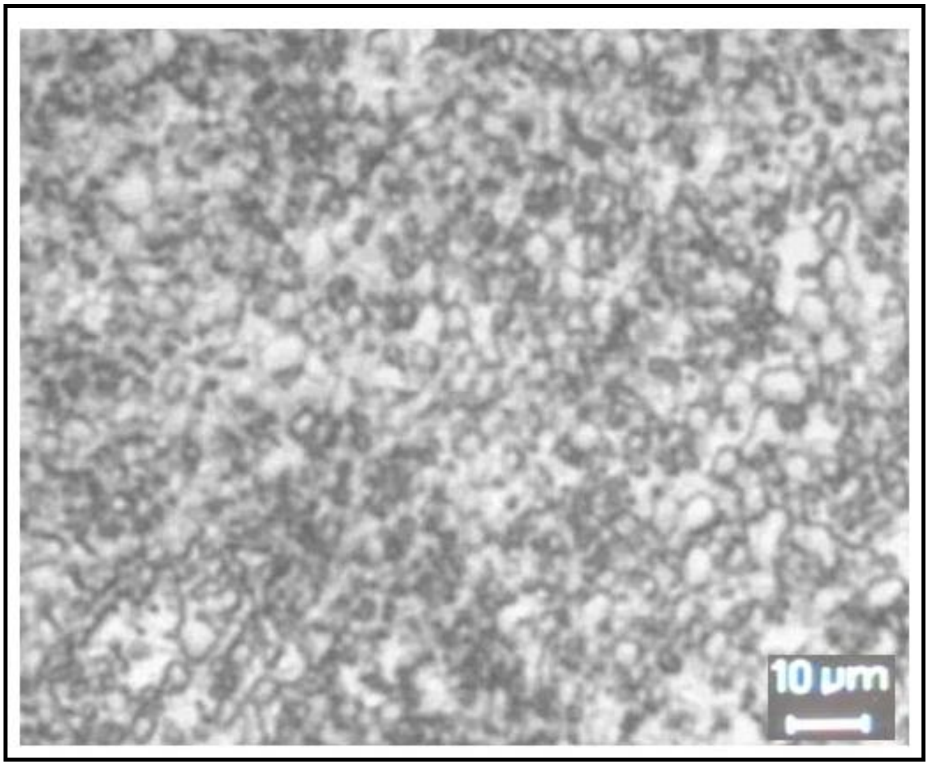


Figure 5.3 Microstructure of untreated annealed AISI 52100 steel sample.

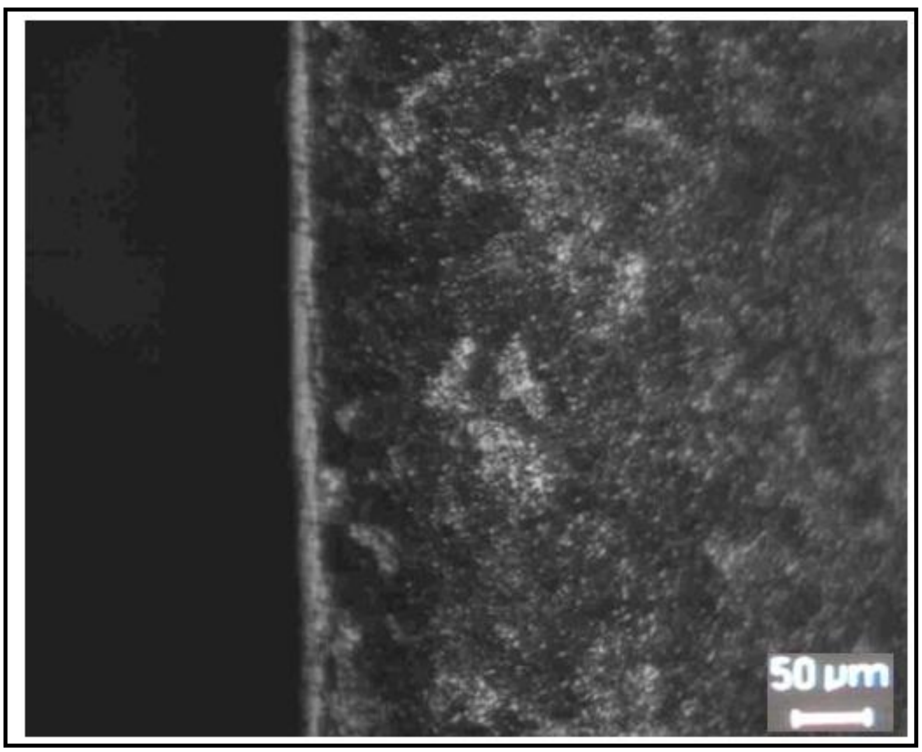


Figure 5.4: Microstructure of 25% Nitrogen and 75% Argon gas plasma nitrided sample.

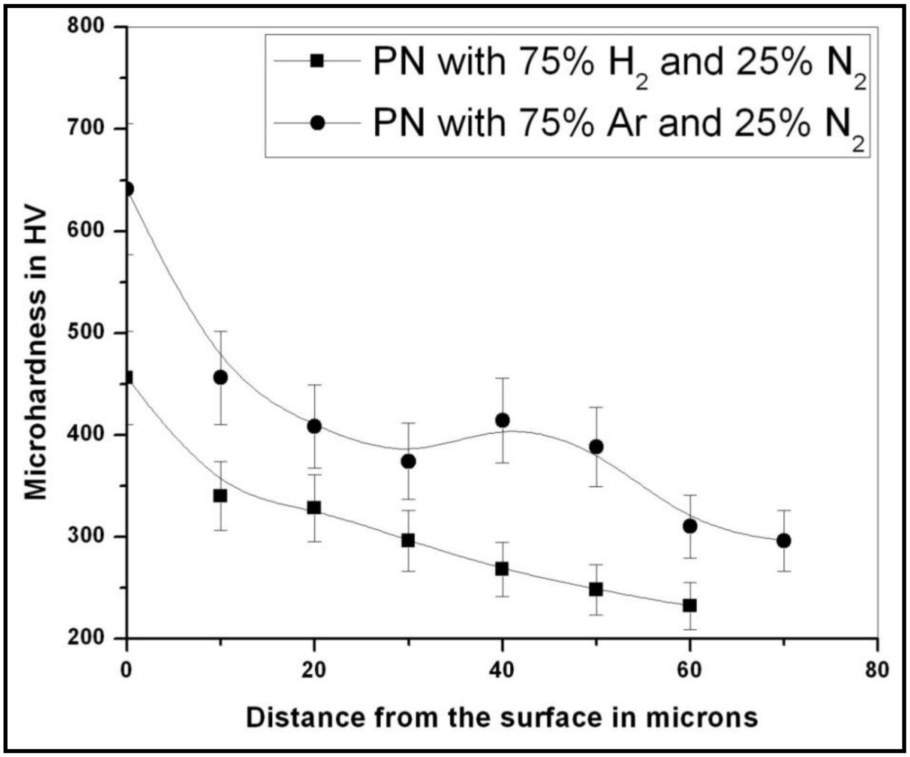


Figure 5.5: Micro-hardness profile with diffusion depth in plasma nitrided samples.

On the other hand when samples were treated with 25% Nitrogen and 75% Hydrogen gas mixture a 5 µm thick white layer and 40 µm thick diffusion zone is observed. The formation of white layer is due to the presence of Fe_4N and Fe_3N phases on the surface.

Figure 5.5 shows the micro-hardness depth profile of plasma nitrided samples. Micro-hardness is measured at every 10 µm distance from the surface towards the core which includes white layer. It is evident from the Figure 5.5 that Argon and Nitrogen gas mixture gives slightly larger diffusion region in comparison to the Hydrogen, Nitrogen gas mixture. X-ray diffraction analysis is also carried out to identify phases formed on the surface before and after the plasma nitriding process. The observed peaks on untreated and plasma treated annealed sample are shown in the Figure 5.6.

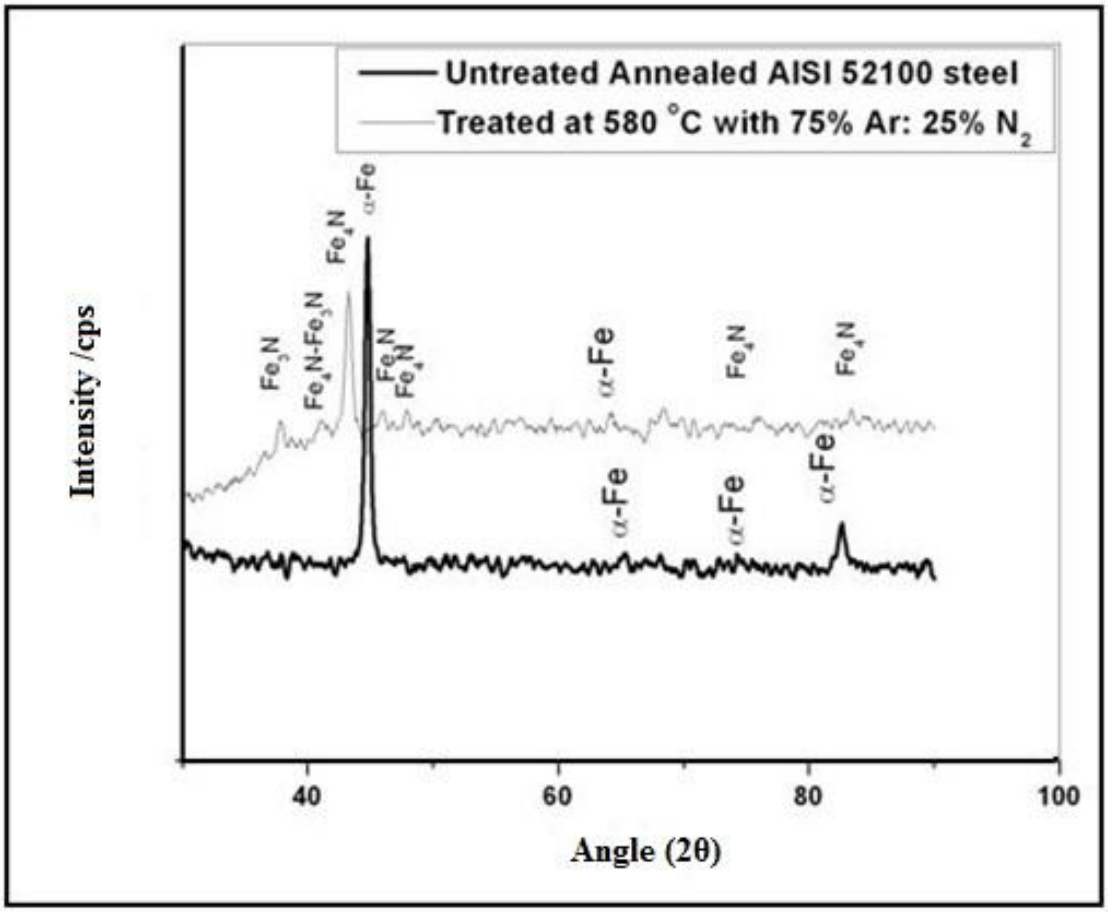
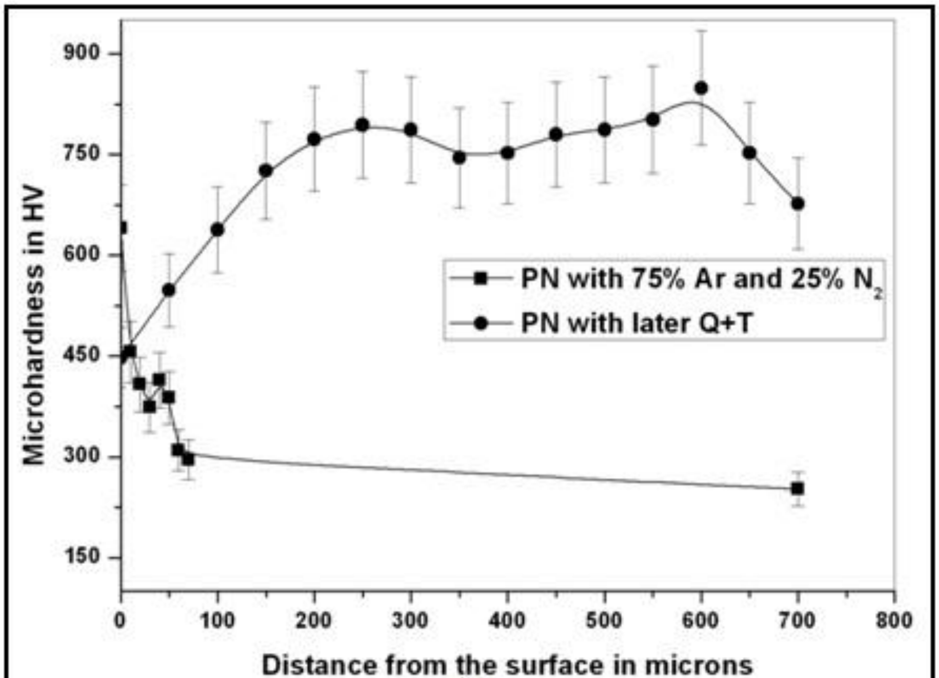


Figure 5.6: X-ray diffraction patterns of untreated sample and 25% Nitrogen and 75% Argon gas plasma nitrided sample.

The untreated sample showed broadened ferrite peaks whereas plasma nitriding led to the formation of iron nitride precipitates like Fe_4N and Fe_3N and their relative abundance depends on the treatment temperature. Since the chromium nitride peaks are absent, the chromium content of this steel is too low to form a significant amount of hard chromium nitrides. This might be the reason for lower hardness in the annealed samples.

5.5.4 Effect of Heat Treatment on Annealed Samples after Plasma Nitriding

As explained earlier, annealed races of AISI 52100 steel with two different gas compositions were plasma nitrided. It is observed that plasma nitriding with 25% Nitrogen and 75% Argon gives a case depth of 60 microns whereas plasma nitriding with 25% Nitrogen and 75% Hydrogen gives a case depth of 40 microns. The increase in the case depth and increase in the surface hardness is due to the presence of Argon gas. To move one step further, these plasma nitrided annealed races have been heat-treated (i.e. Q+T) later. The obtained micro-hardness with distance from the surface to core for both the cases (i.e. 75% Ar – 25% N_2 and 75% H_2 – 25% N_2 gas plasma nitrided) is shown in the Figure 5.7 (a & b) respectively.



(a)

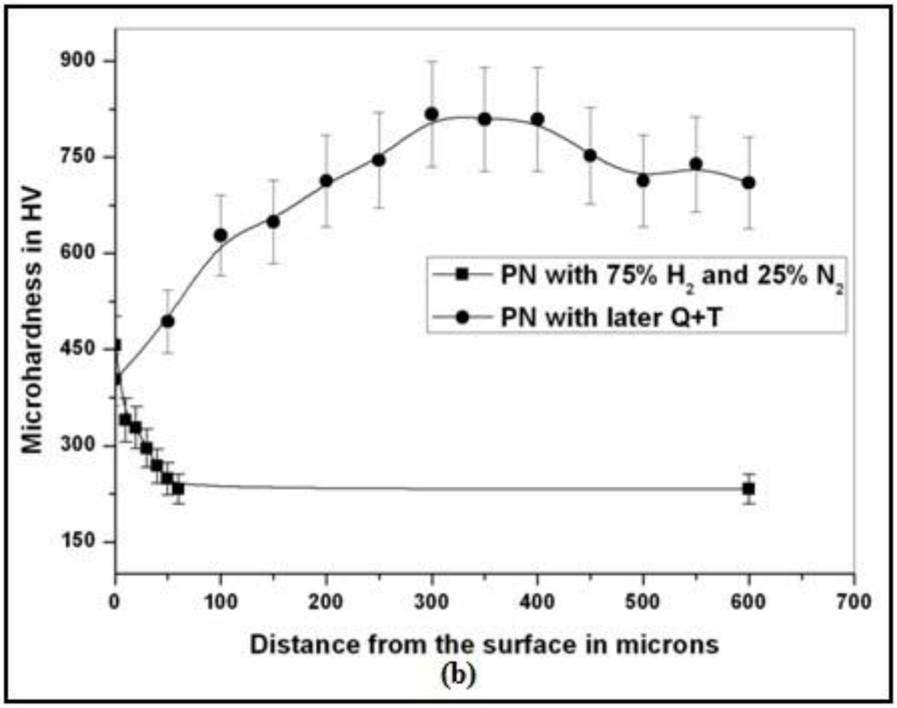


Figure 5.7: Micro-hardness profiles with diffusion depth in plasma nitrided samples after heat treatments (a) Plasma nitrided with 75% Ar – 25% N_2, (b) Plasma nitrided with 75% H_2 – 25% N_2.

This figure also shows the difference between the micro-hardness before and after the heat-treatment (Q+T) process of plasma nitrided races. The trends of the micro-hardness after heat-treatment is different from the non-heat treated plasma nitrided races.

The observed trends reveal that after the heat-treatment (i.e. Q+T) the surface micro-hardness has reduced in comparison to the plasma nitrided annealed sample. The lower surface micro-hardness of the nitrided and heat-treated (Q+T) sample is related to the decrease of nitrogen and carbon contents on the surface during the heat-treatment process. After certain distance from the surface the micro-hardness has increased (see Figure 5.7 (a) & (b)) indicating the formation of tempered martensite with carbides. The microstructure study of the plasma nitrided and heat-treated samples is shown in the Figure 5.8. It indicates that the nitrided layer which was formed in the nitriding process has been removed after heat-treatment process of the nitrided sample and should have diffused in the core. The removal of nitrided layer is also confirmed through the XRD analysis. The XRD analysis of the sample after heat-treatment has given only α – Fe peaks as shown in the Figure 5.9.

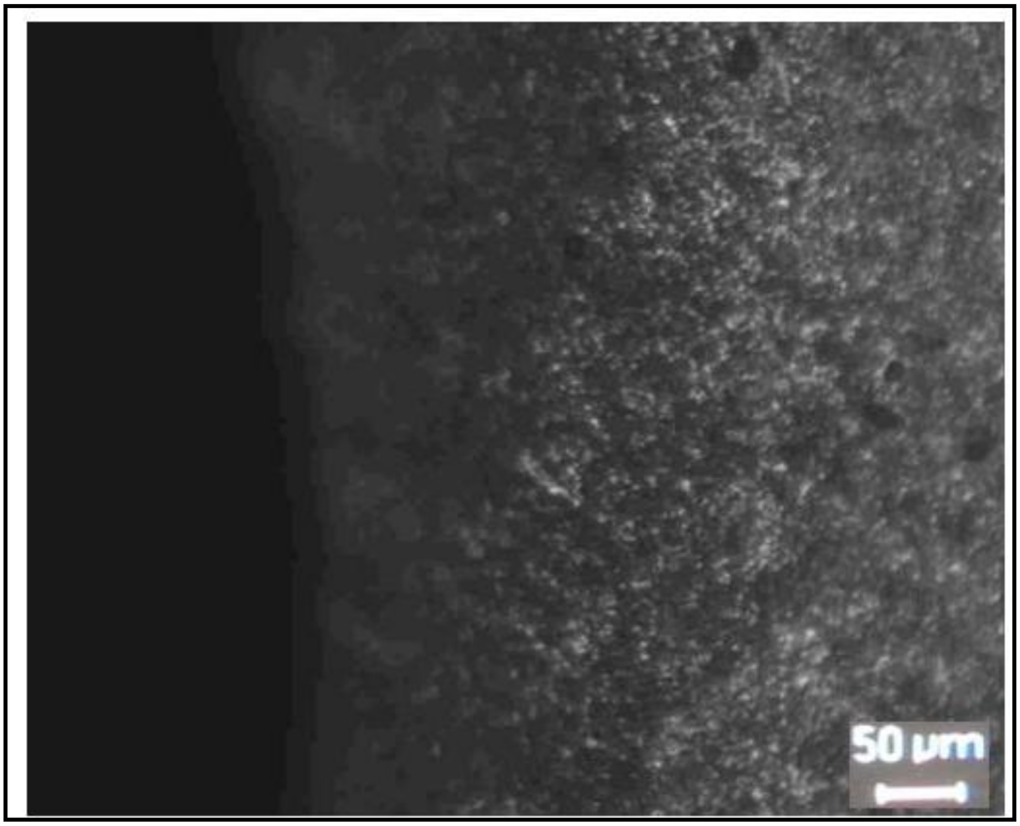


Figure 5.8: Microstructure of 25% Nitrogen and 75% Argon gas plasma nitrided annealed sample after quenching and tempering (Q+T).

It also confirms the removal of nitride from the surface that was formed in the nitriding process. As evident from Figure 5.7 (a) & (b), ~800 HV micro-hardness in ~200-600 μm region below 200 μm from the surface could be achieved through unconventional procedure. This hardness region is larger for the samples plasma nitrided with Argon-Nitrogen gas mixture. This 200 μm layer may be used for surface finish of the machined sample and the higher hardness region of 250-350 μm could be used to increase the life of the AISI 52100 ball-bearing steel. This step indicates that raising the temperature to higher temperature has removed the nitride layer indicating that there is an outward diffusion of nitrogen and the core got hardened. Hence plasma nitrided components should not be used at temperatures above 750-800 ^{0}C.

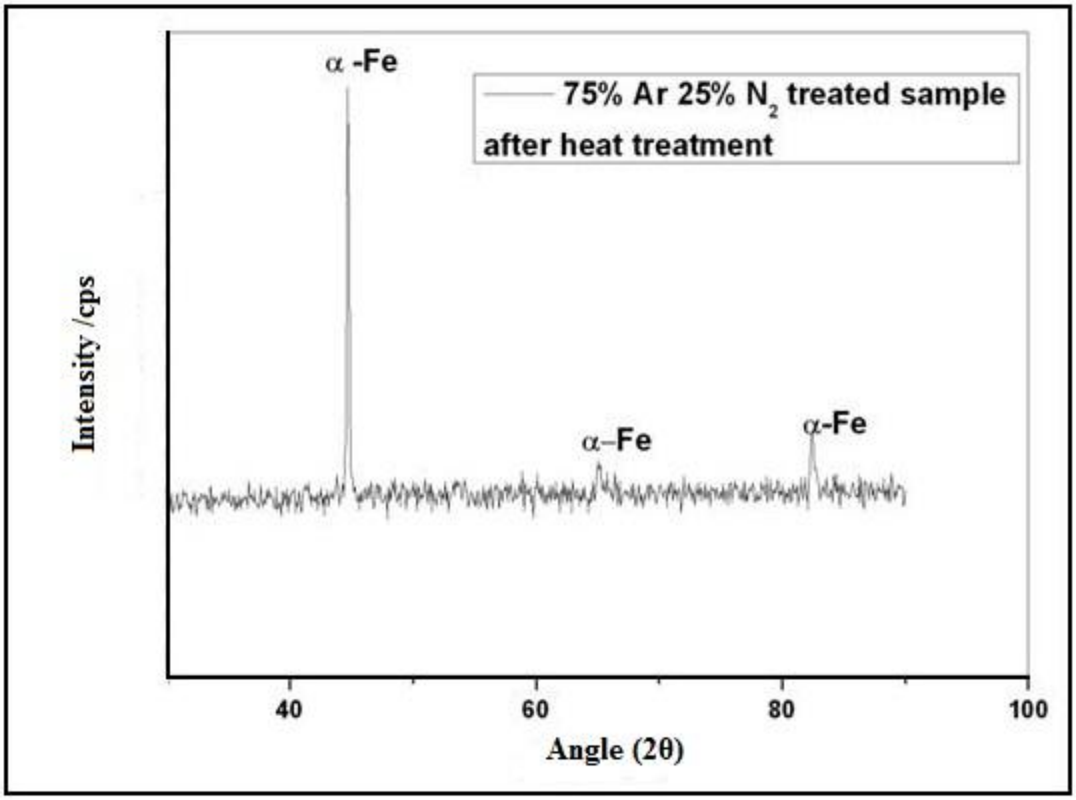


Figure 5.9: X-ray diffraction pattern of 25% Nitrogen and 75% Argon gas plasma nitrided sample after quenching and tempering (Q+T).

5.6 Conclusion

On the basis of the obtained results we can say that it is possible to plasma nitride AISI 52100 ball bearing steel in the annealed condition. Plasma nitriding of annealed sample is possible at higher temperature (>560 ^{0}C) whereas on the annealed-quenched (A+Q) and annealed-quench-tempered (A+Q+T) samples it reduces core hardness. The Argon and Nitrogen gas mixture gives higher surface hardness on the annealed samples. On doing quenching and tempering (i.e. Q+T) on the plasma nitrided annealed sample, we obtained a region ~200-600 µm of higher hardness ~800 HV below 200 µm region from the surface. The higher hardness region is much better and wider for Nitrogen and Argon gas treated samples. The obtained results are promising and a scope exists for further study and also to see the effects on the mechanical behaviour after plasma nitriding of AISI 52100 steel.