

Effect of Post Treatment in Argon Environment of Plasma Nitrided Local Disc Brake

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Abstract: Plasma nitriding (also known as ion nitriding, plasma ion nitriding, or glow-discharge nitriding) is a method of surface hardening using glow-discharge technology to introduce nascent (elemental) nitrogen to the surface of a metal part subsequent diffusion into the material. Because of the formation of high compressive residual stresses in the case region, increasing surface hardness and depth parameters cause remarkable improvement in the mechanical properties of steels. In this study, the properties of a nitrided local disc brake were investigated. For that purpose, the material was nitrided at 400°C of temperature, 1.6 mbar of pressure, and 4 hours of time then followed by post-treatment in Argon (Ar) environment for various of holding time such as 10, 20, 30, 40, and 50 minutes. At these conditions the hardness increased in the range 123.1 - 223.3 VHN, the wear rate decreased in the range 21.7×10^{-9} - 3.1×10^{-9} mm³/mm kg, while before being nitrided the hardness and the wear rates were 113.5 VHN and 22.8×10^{-9} mm³/mm kg, respectively. The optimum condition for holding time during post treatments was 20 minutes. Besides that, the effect of various of N₂: Ar mixture such as 90%: 10%, 80%: 20%, 70%: 30%, and 60%: 40% on the properties of local disc brake was studied. For various of gas mixing, the optimum hardness in the order of 252.7 VHN and wear rate in the order of 2.8×10^{-9} mm³/mm kg were achieved at 90%N₂: 10%Ar of a gas mixture.

Keywords: Local Disk Brake, Ion Nitriding, Plasma, Post-treatment

1. Introduction

A braking system is one of the most critical safety components of an automobile. It is mainly used to decelerate vehicles from an actual speed to the desired speed. Friction based braking systems are still a conventional device to convert kinetic energy into thermal energy, through friction between the brake pads and the rotor faces [1-3]. All braking system depends upon the frictional force to stop, to control or to prevent motion [4-5]. Because these components are always rubbing against the surface of other components, these components will wear out quickly so that their service life will also be reduced. To slow down the wear rate or to extend the fatigue life or service life, the surface of the component needs to be improved. There are many surface treatment methods

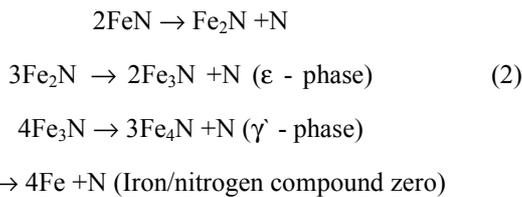
used to improve the surface quality such as carburizing, nitriding, carbonitriding, induction hardening, shot peening, physical vapor deposition (PVD) and chemical vapor deposition (CVD) coating, etc. These treatments form a hardened surface layer with compressive residual stress, and therefore, the fatigue properties are improved by the surface layer [6]. One of the chemical vapor deposition coating methods is plasma nitriding. Plasma nitriding is also called ion nitriding, glow discharge nitriding or plasma ion nitriding [7]. Plasma nitriding is a process of surface hardening using glow discharge technology to introduce nascent (elemental) nitrogen to the surface of a metal part for subsequent diffusion into the material [8]. In plasma DC nitriding process the components to be treated as a cathode, where the grounded wall of the reactor forms the anode.

The treated material is directly involved in the discharge

process [9]. Typically, the applied voltage between the anode and the cathode is 400 - 700 V. The positive ions produced by glow discharge are accelerated near the cathode surface and bombard the surface of the treated specimen. These ions bombardment heats the workpiece, cleans the surface and provides active nitrogen, and the nitrogen diffusion modifies the surface. During ion nitriding, three reactions will occur at the surface of the material being treated. In the first reaction, iron and other contaminants are removed from the surface of the sample by an action known as sputtering or by a reducing reaction with hydrogen. The impact of hydrogen or argon ions bombarding the sample surface dislodges the contaminants that will be extracted by the vacuum system. The removal of these contaminants allows the diffusion of nitrogen into the surface [10]. During the second reaction, and as a result of the impact of the sputtered ion atoms, case formation begins at the sample surface of iron nitrides, which follows equation;



During the third reaction, a breakdown of the FeN begins under continuous sputtering from the plasma. This one causes the instability of the FeN which begins to break down into the ϵ phase followed by γ' - phase and an iron-nitrogen compound zone which follows equation [10];



The schematic representation of plasma nitriding, indicating the different steps in the formation of the nitride film is presented in Figure 1 [11].

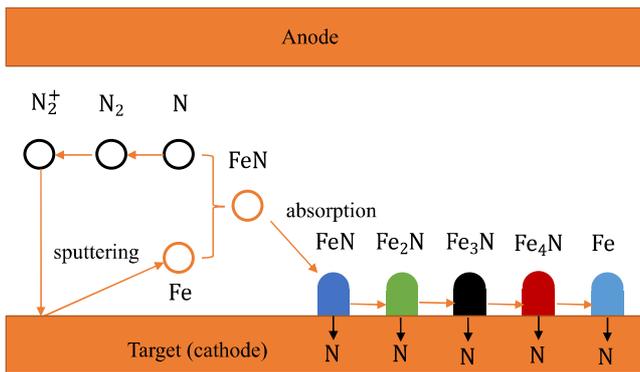


Figure 1. Schematic representation of plasma nitriding, indicating the different steps in the formation of the nitride film.

Figure 1 shows the target (cathode) is bombarded by positive ions (N_2^+), causing sputtering of the metallic atom (e.g., Fe), which react in the plasma to form FeN. The nitrides are deposited at the target, where they react further with the target (Fe) atoms, into Fe_2N and Fe_3N , until finally the stable Fe_4N is formed. The N atoms released in these reactions can diffuse into the target, to form the final nitride layer.

2. Methodology

2.1. Materials and Sample Preparation

The material used in this study was a local disc material with chemical composition as shown in Table 1.

Table 1. Chemical Composition of the Local Disk Material.

Element	Quantity (%)
Carbon	0.072
Silicon	0.060
Phosphor	0.004
Mangan	1.726
Nickel	0.011
Chromium	0.025
Molybdenum	0.007
Copper	0.012
Titanium	0.044
Tin/Sn	0.010
Aluminium	0.046
Niobium	0.052
Vanadium	0.006
Cobalt	0.002
Lead/Pb	0.006
Calcium	0.005
Zinc	0.010
Iron/Fe	97.90

This material was cut into specimens 3×1.4 mm size in a disk shape. The specimens were grounded with SiC papers from #80 up to #5000 grit and polished mechanically with $1 \mu\text{m}$ diamond paste. The polished specimens were washed with acetone in an ultrasonic cleaner and dried at room temperature.

2.2. Plasma Nitriding Process

Plasma nitriding process was carried out using homemade DC-glow discharge plasma (PSTA-BATAN) which usually used for carburizing and nitrocarburizing processes [11-16]. The photograph of the DC-glow discharge plasma is shown in Figure 2.



Figure 2. A DC-plasma glow discharge in PSTA-BATAN Yogyakarta.

In this research, the plasma nitriding process was conducted at 400°C of temperature, 1.6 mbar of pressure, and 4 hours of nitridation time. After the nitridation process then followed by post-treatment in Argon (Ar) environment for various of

holding time such as 10, 20, 30, 40, and 50 minutes. Besides that, plasma nitriding process was also conducted for various of Ar:N₂ mixture such as 90%:10%, 80%:20%, 70%:30%, and 60%:40%.

After the treatments, several parameters such as surface hardness, wear rate, corrosion rate, and phase material were tested using Matsuzawa MMT-X7 microhardness tester, Ogoshi High-Speed Universal Wear Testing Machine, potentiostat type PGS-201, and X-Ray Diffractometer (XRD), respectively.

3. Results and Discussion

3.1. Analysis of the Hardness and Wear Rate

The first experiment was conducted at 400°C of temperature, 1.6 mbar of pressure, 4 hours then followed by an Argon post-treatment process with the treatment results as shown the results as shown in Figure 3.



Figure 3. The samples after nitridation and Argon post-treatment. Labels 1, 2, 3, 4, and 5 indicate the post-processing times; 10, 20, 30, 40, and 50 minutes, respectively.

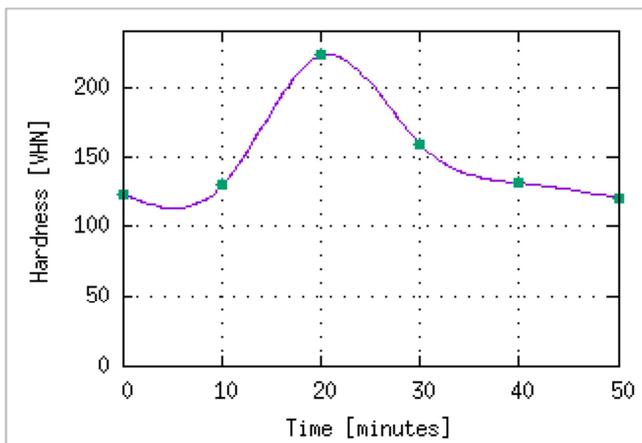


Figure 4. The hardness of the sample as a function of Argon post-treatment times. The optimum hardness was at 20 minutes of post-treatment.

After the treatments, we found the hardness was in the range 123.08 - 223.3 VHN, while the wear rate was in the range 21.7×10^{-9} - 3.1×10^{-9} mm³/mm kg as shown in Figures 4 and 5. Before the treatment, the hardness and wear rate were 113.48 VHN and 22.8×10^{-9} mm³/mm kg, respectively. The optimum values of hardness and wear rate were founded at 20 minutes of holding time. At this optimum condition, the hardness increased ~ 2 times, while the wear rate decreased ~7 times. This increase in hardness can be attributed to the addition of sputtered atoms. Increasing in sputtered atoms will cause the higher possibility to form a compound layer (FeN

phase system). For post-treatment, after 20 minutes the microhardness of steel suffered a decline. This decrease probably associated with the increase of alloy nitride precipitation which reduces the nitrogen held in solid solution and consequently the compressive residual stress in the crystal lattice.

Figure 5 shows that the wear rate decreases with the increasing of post-treatment time, and reached the lowest value in the order of 3.1×10^{-9} mm³/mm kg. This phenomenon in decreasing of wear rate is same as on the phenomenon in increasing in hardness. Increasing in hardness followed by decreasing in wear rate.

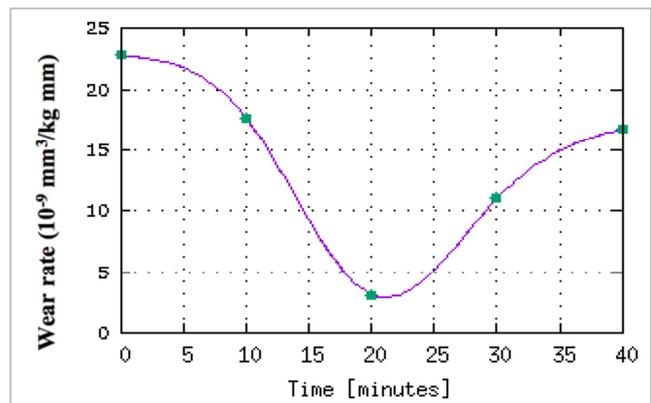


Figure 5. The wear rate of the sample as a function of Argon post-treatment times. The optimum wear rate was at 20 minutes of post-treatment.

The second experiment was conducted at 400°C of temperature, 1.6 mbar of pressure, and 4 hours nitridation for various % of Argon (Ar) such as 10%, 20%, 30%, and 40% with the samples conditions after treatments are presented in Figure 6.



Figure 6. The samples after nitridation with various % of Argon. Labels 1, 2, 3, and 4 indicate the percentages of Ar; 10%, 20%, 30%, and 40%, respectively.

The results of the second experiment are shown in Figures 7 and 8, the hardness was in the range 123.08 - 252.72 VHN, while the wear rate was in the range 26.4×10^{-9} - 2.8×10^{-9} mm³/mm kg. The optimum values of hardness and wear rate were at 90% N₂:10% Ar of a gas mixture. At this optimum condition, the hardness increased ~ 2 times, while the wear rate decreased ~ 8 times which is a significant improvement. With the increasing of Ar gas mixture percentage (%), the hardness increases and the wear rate decreases, caused by the higher possibility of sputtered atoms. The higher possibility of sputtered atoms will cause the higher possibility to form FeN compound layer system.

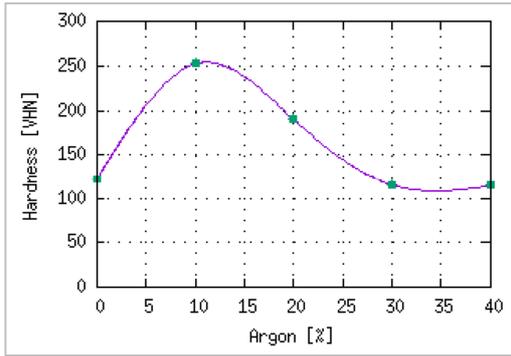


Figure 7. The hardness of the sample as a function of Ar percentage. The optimum hardness was at 10% of Ar.

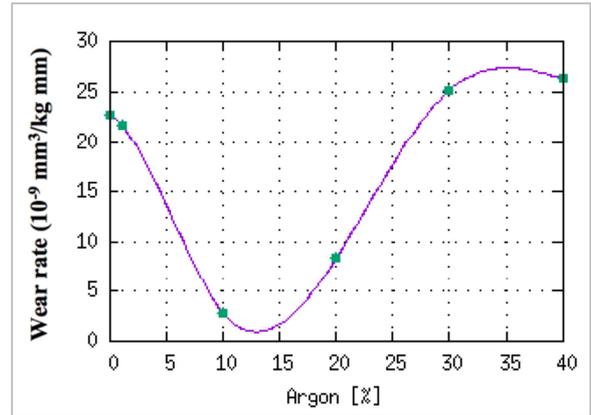


Figure 8. Wear rate of the sample as a function of Ar percentage. The optimum wear rate was at 10% of Ar.

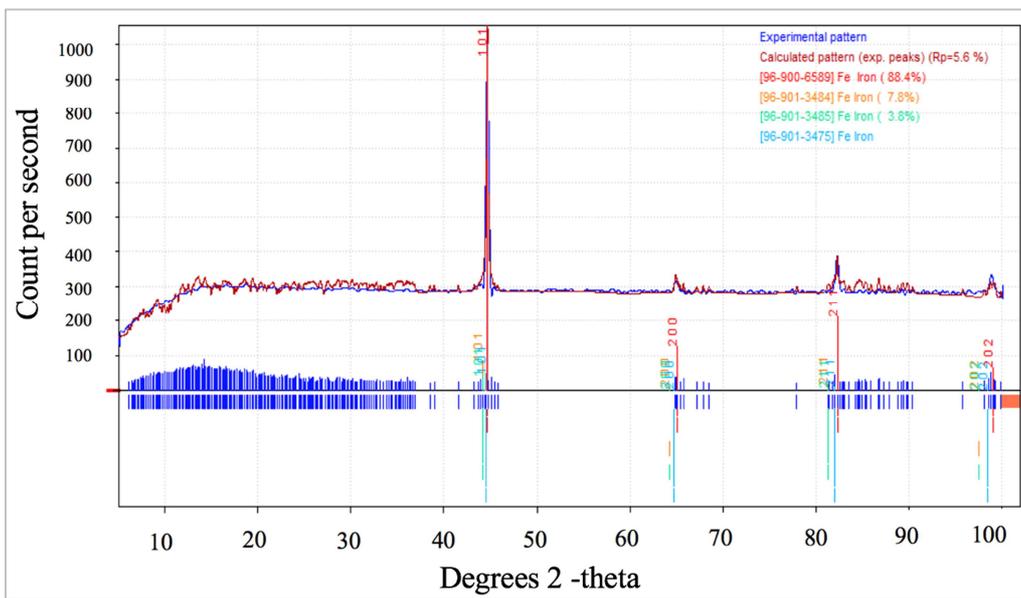


Figure 9. X-ray diffraction patterns of raw material.

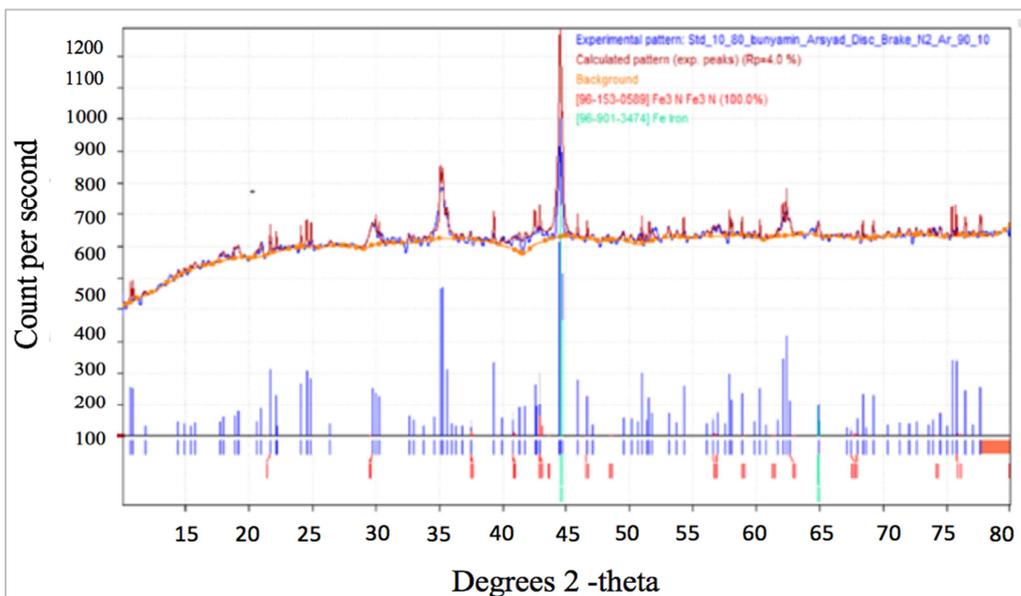


Figure 10. X-ray diffraction patterns of sample nitride at 90%N₂:10%Ar of a gas mixing.

3.2. XRD Analysis of Nitrided Samples

An XRD analysis results for raw and nitrided materials at 90%N₂:10%Ar gas mixing are presented in Figures 9 and 10. The XRD analysis found the Fe₃N phases that give the main contribution in increasing of hardness and decreasing of wear rate of the materials.

3.3. Analysis of Corrosion Resistance Test Results

Corrosion test results for raw material, pure N₂ treatment, 90% N₂ + 10% Ar gas mixture, and 20 minutes post-treatment samples in 3.5 % NaCl media are presented in Figure 11.

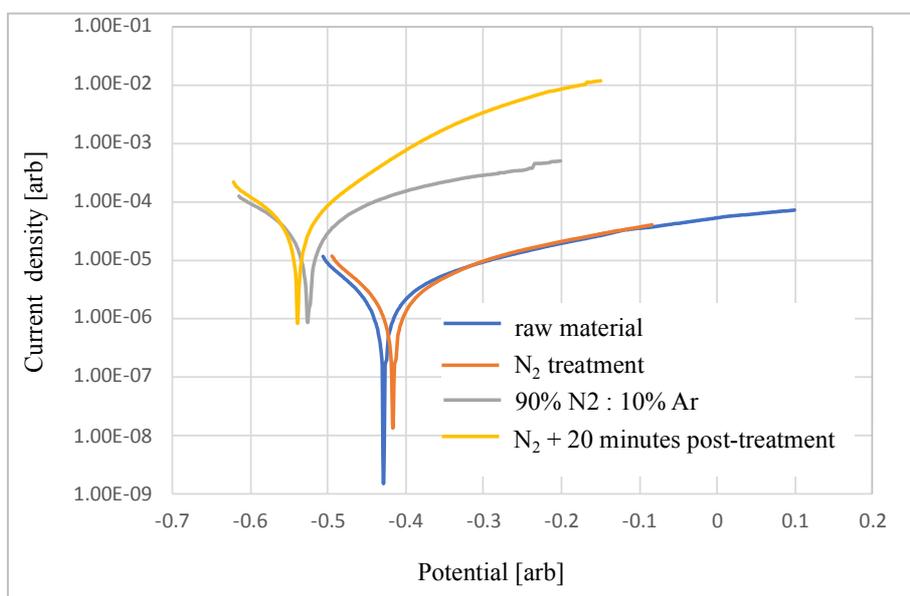


Figure 11. Potentiodynamic polarization curves of raw material, pure N₂ treatment, 90%N₂ + 10% Ar gas mixture, and 20 minutes post-treatment samples in 3.5 % NaCl media.

Further analysis using VersaStudio given data as shown in Table 2. These data show that the corrosion rate of raw material is 0.012 mm/year, and after being nitride with pure nitrogen it becomes 0.010 mm/year. Corrosion rate for nitrided samples at 90% N₂:10% Ar and nitrided samples

followed by 20 minutes post-treatment are 0.186 mm/year and 0.268 mm/year, respectively. These data show that the effect of Argon in the nitriding process and post-treatment deteriorate the corrosion properties.

Table 2. Corrosion Test Result.

No.	Treatment condition	I _{corr} (μA)	E _{corr} (mV)	mpy	mm/year
1.	Raw material	1.57	- 429.15	0.47	0.012
2.	Pure Nitrogen (N ₂)	1.36	- 419.25	0.41	0.010
3.	90 %:10% of N ₂ :Ar	24.48	- 525.26	7.32	0.186
4.	Post-treatment, holding time 20 minutes	35.37	- 538.60	10.57	0.268

4. Conclusion

We have described the effect of Argon post-treatment environment on plasma nitrided local disc brake, followed by the experiment of N₂:Ar of gas mixing. The main parameters to be observed were hardness, wear rate, and corrosion rate. The optimum conditions of time for post-treatment was at 20 increased ~ 2 times, while the wear rate decreased ~7 times. For various of gas mixing, the optimum values of hardness and wear rate were founded at 90%N₂:10%Ar of a gas mixture. At this optimum condition, the hardness increased ~ 2 times, while the wear rate decreased ~ 8 times which is a significant improvement. Based on the XRD, the Fe₃N form gave the main contribution in increasing in hardness and wear rate of

the materials. Corrosion rate for nitrided samples at 90 %N₂:10%Ar and nitrided samples followed post-treatment and holding time 20 minutes were 0.186 mm/year and 0.268 mm/year, respectively. These data show that the effect of Argon in the nitriding process and post-treatment deteriorate the corrosion properties.

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