Influence of Nitrogen Ion Implantation on the Disc Brake Material of Motor Vehicles Component

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Abstract: Weaknesses of local disc brakes are cover several conditions such as low hardness, wear, and corrosion resistance. To improve this weakness, it is necessary to modify the surface properties of the material. The aim of this research is to study the influences of nitrogen ion implantation on the surface properties of a disc brake material. The implantation process was carried out for various of ions dose such as $3.10^7$×$10^{16}$ ions/cm$^2$, $3.148$×$10^{16}$ ions/cm$^2$, $3.728$×$10^{16}$ ions/cm$^2$, $4.039$×$10^{16}$ ions/cm$^2$, $4.350$×$10^{16}$ ions/cm$^2$ at a certain energy and beam current of $60$×$10^{16}$ ions/cm$^2$, $30$ µA respectively. Hardness and wear properties were tested using microhardness tester and wear testing machine, respectively. Meanwhile, the crystalline structure for un-implanted (raw) and implanted materials at the optimum dose was analyzed using XRD. From the hardness test results, it can be obtained that the hardness of raw material is 59.82 VHN and after implantation it reached the highest value of 109.78 VHN or increases by factor 83%, while the wear test results is $22.9$×$10^{-9}$ mm$^2$/kg for raw material and after implantation it reaches the highest value of $2.5$×$10^{-9}$ cm$^2$/kg or decreases by factor 88%. These conditions were obtained at $3.728$×$10^{16}$ ions/cm$^2$ of dose. Based on the XRD analysis, 45.5% Fe$_2$N and 54.55% Fe$_3$N compounds are formed.

Keywords: Ion Implantation, Disc Brake, Hardness Test, Wear Test, XRD

1. Introduction

A braking system is one of the 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 [1-4]. All braking system depends upon the frictional force to stop, control or prevent motion [5, 6]. Because these components are always rubbing against the surface of other components, it will wear out quickly so that their service life will also be reduced. To reduce the wear rate or to extend the fatigue life or service life, the surface of the component needs to be improved. Several surface treatment methods are used to improve the surface quality such as carburizing, nitriding, carbonitriding, induction hardening, shot peening, physical vapor deposition (PVD) and chemical vapor deposition (CVD), as well as an ion implantation technique [7, 8]. These treatments form a hardened surface layer with compressive residual stress, and therefore, the fatigue life is improved by the surface layer [9, 10].

Ion implantation is a surface modification technique by which atoms and molecules are ionized, accelerated in an electrostatic field, and implanted into the near-surface of a substrate. This technique produces a modification in the structure of metals by the formation of new crystalline phases, metastable or amorphous, and thus to improve the surface properties [11]. A great advantage is the negligible effect of ion implantation on the dimensions of the treated element; hence, the process can be applied in the final stage of manufacturing of products that already have their final dimensions [12]. Besides the improvement of tribological properties, ion implantation contributes to an increase in mechanical strength. This is associated with an increase in the microhardness of the implanted samples. The implantation process is accompanied with the appearance of compressive stresses and inclusions of nitrides, carbides, and borides.
implantation-induced hardening process depends on the type and dose of implanted ions and the temperature of the implanted material. In most of the applications for surface treatment, implanted elements are nitrogen, carbon and boron that harden the surface alloy as a consequence of fine particle formation by precipitation. The introduction of new atoms in the crystal lattice is not the only effect of ion implantation, but also the damage originated in the crystal structure of the target by the energetic collision cascades must also be taken into account [13]. As each ion penetrates the target, it undergoes a series of collisions displacing host atoms along the way. Both the ion and dislodged target atoms can continue and cause further damage, and so the energy is spread over many moving particles. Therefore, after implantation of high doses of ions, an initially crystalline target will be so perturbed that it will have changed to a highly disordered state [14]. The amount of crystallographic damage can be enough to cause the partial amorphization of the metal surface, depending on the dose, energy, temperature (governing self-annealing that can occur to repair some or all of the damage as it is generated) and ion species (heavy ions displace a greater volume of target atoms per ion) [15]. Finally, the implantation of a high dose of ions induces significant compressive stress that can contribute to the blocking of the fissures and close the channels of corrosion [16].

2. Methodology

The material that is used in this study was a local disc material, the type of component is presented in Figure 1. In this experiment, local disc component was cut into specimens 4×14 mm in size in a disc shape using a water jet cutting. The specimens were grounded with SiC papers from 80 up to 5000 mesh and polished mechanically with 1 µm diamond paste. The polished specimens were washed with acetone in an ultrasonic cleaner and dried at room temperature. The chemical compositions of the samples are listed in Table 1. The samples were implanted by using 150 keV/2mA ion implanters. Implantation of the samples performed at dose 3.107×10^{16} ion/cm^2, 3.148×10^{16} ion/cm^2, 3.728×10^{16} ion/cm^2, 4.039×10^{16} ion/cm^2 and 4.35×10^{16} ion/cm^2 at 60 keV of ions energy. The increase of the temperature of the samples was ensured solely by the incoming ion beam without any additional heating.

### Table 1. Chemical Composition of local disc brake.

| Element | Quantity (%) |
|---------|--------------|
| C       | 0.0720       |
| Si      | 0.0606       |
| P       | 0.0041       |
| Mn      | 1.7266       |
| Ni      | 0.0113       |
| Cr      | 0.0252       |
| Mo      | 0.0073       |
| Cu      | 0.0121       |
| Ti      | 0.0446       |
| Sn      | 0.0011       |

3. Result and Discussion

3.1. Analysis of the Hardness and Wear Rate

The effect of nitrogen ion implantation in micro-hardness properties of samples was tested by using Vickers Tester Microhardness Tester type MTX7, while the wear properties were tested using the Ogoshi High Speed Universal Wear Testing Machine with the result as shown in Figure 2. It concluded that the hardness and wear rate of samples improved after nitrogen ion implantation and extend of improvement increases with the dose. The maximum hardness is appearing at a nitrogen ion dose of 3.728×10^{16} ion/cm^2. In this condition, the hardness increase from 59.77 VHN (raw material) to 109.78 VHN (83% higher), while the wear rate decreases from dose 3.107×10^{16} ion/cm^2 to 3.728×10^{16} ion/cm^2 or reduce by a factor 88%. Over this dose the hardness decreases while the wear rate increases. This may be caused by defects due to the excess nitrogen ions implantations.

### Table 2. Peaks positions and interplanar spacings for un-implanted local disc brake materials.

| 2Θ (degree) | d-spacing (Å) | Intensity | hkl  |
|-------------|---------------|-----------|------|
| 44.67       | 2.0271        | 10.00     | 101  |
| 64.91       | 1.4354        | 39.57     | 200  |
| 82.25       | 1.1712        | 130.47    | 211  |
| 98.72       | 1.0066        | 52.16     | 202  |

Figure 1. A type of local disc component.
Figure 2. Effect of nitrogen dose on the hardness and wear rate of implanted samples (local disc material).

### 3.2. XRD Analysis of Implanted and Un-implanted Samples

The phase composition of nitrogen-implanted samples was analyzed using X-rays in the Θ–2Θ mode and $\lambda = 1.548$ Å of CuKα radiation. The presence of nitride in implanted samples at optimum conditions was revealed by the spectrum in Figure 4, Tables 3, and 4. Figure 3 and Table 2 show the XRD patterns of un-implanted sample material.

The pattern in Figure 3 (un-implanted material) shown (101), (200), (211), and (202) peaks with cubic structures at $2\Theta = 44.67^\circ$, 64.91°, 82.25°, and 98.72° respectively. Those peaks are related to the low alloys steel substrate with iron (Fe) as the main element. A Crystallography Open Database (COD), entry number 96-411-3942 code show that the phase is $\alpha$ Fe (alpha-iron). The XRD pattern for implanted material with nitrogen ions at $3.728 \times 10^{16}$ ion/cm$^2$ of ions dose is shown in Figure 4. A COD, entry number 96-411-3942 code show that the phases are 54.5% of Fe$_3$N and 45.5% of Fe$_2$N.
Figure 4. XRD pattern of implanted samples with nitrogen ions at energy of 60 keV for 60 minutes of implantation time or $3.728 \times 10^{16}$ ions/cm$^2$ of ions dose (45.5% of Fe$_2$N phase and 55.5% of Fe$_3$N phase).

Table 3. Fe$_2$N phase peaks positions and interplanar spacings for local disc brake materials.

| $2\theta$ (graph) | $2\theta$ (cal) | d-spacing (Å) | Intensity | hkl |
|------------------|----------------|--------------|-----------|-----|
| 20.01            | 19.57          | 4.4101       | 15.77     | 001 |
| 37.60            | 37.44          | 2.3851       | 14.64     | 11  |
| 40.74            | 40.72          | 2.2081       | 19.34     | 002 |
| 42.93            | 42.81          | 2.1027       | 21.60     | 111 |
| 67.76            | 67.68          | 1.3802       | 16.59     | 300 |
| 82.20            | 82.14          | 1.1730       | 72.34     | 302 |
| 83.65            | 83.51          | 1.1549       | 18.94     | 221 |
| 88.39            | 88.21          | 1.1054       | 19.04     | 204 |
| 94.10            | 93.94          | 1.0527       | 23.85     | 222 |

Table 4. Fe$_3$N phase peaks positions and interplanar spacings for local disc brake materials.

| $2\theta$ (graph) | $2\theta$ (cal) | d-spacing (Å) | Intensity | hkl |
|------------------|----------------|--------------|-----------|-----|
| 21.97            | 21.9           | 4.0566       | 16.64     | 100 |
| 30.11            | 30.04          | 2.9629       | 17.42     | 101 |
| 38.54            | 38.52          | 2.3335       | 17.95     | 110 |
| 41.35            | 41.34          | 2.1722       | 16.24     | 002 |
| 43.96            | 43.92          | 2.0535       | 35.37     | 111 |
| 57.81            | 57.88          | 1.5904       | 14.94     | 112 |
| 64.57            | 64.54          | 1.4413       | 16.40     | 211 |
| 69.73            | 69.97          | 1.3484       | 16.13     | 300 |
| 82.61            | 82.59          | 1.1673       | 14.43     | 220 |
| 84.43            | 84.22          | 1.1475       | 20.64     | 302 |
| 86.20            | 86.62          | 1.1267       | 14.28     | 221 |

4. Conclusion

Based on the experiments, it can be concluded as follow:

1. The hardness of un-implanted materials is $59.77$ VHN, after being implanted for various of time or ions dose the hardness increased and reached optimum in order of $109.78$ VHN or increase by factor $83\%$, while the wear rate decreased from $22.9 \times 10^{-9}$ mm$^2$/kg to be minimum in order $2.5 \times 10^{-9}$ mm$^2$/kg or decreased by factor $88\%$. This optimum condition was achieved at 60 minutes of implantation time or at $3.728 \times 10^{16}$ ions/cm$^2$ of ions dose.

2. Based on XRD analysis and after being analyzed using Crystallography Open Database (COD) code entry number 96-411-3942 it’s observed that for un-implanted samples the observed phase is $\alpha$ Fe, after being implanted at optimum conditions the formed phases are $54.5\%$ of Fe$_3$N and $45.5\%$ of Fe$_2$N. The formation of these phases are causing in increasing the hardness or reducing the wear rate.

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