Influence of chamber pressure variations of plasma nitriding on surface hardness and micro structure of Al 6061

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Abstract. Aluminium 6061 is a material that is widely applied in industrial and automotive parts. The corrosion resistance and surface hardness of Al 6061 can be improved by using plasma nitriding. Plasma nitriding is the process of treating the surface of a material by inserting nitrogen in the material through the plasma, i.e. nitrogen gas was converted in the form of plasma before being inserted into the material. This research was conducted using experimental research methods by directly observing the plasma nitriding process. This research used aluminium alloy 6061 as a specimen, variations in chamber pressure of plasma nitriding 100 mTorr, 200 mTorr, and 300 mTorr, constant temperature of 150 °C, constant power input of 40 watts, and holding time for 2 hours. Hardness testing was carried out using a micro-Vickers hardness tester, whereas microstructure testing used SEM (Scanning Electron Microscope) and EDAX. Experimental results showed the highest surface hardness value of Al 6061 was 276.5 VHN. This condition occurred at a chamber pressure of 200 mTorr, temperature of 150 °C, the input power of 40 watts and a holding time of 2 hours. Based on composition and microstructure analysis using EDAX and Scanning Electron Microscope shown that the lowest surface hardness 179.4 VHN had a nitrogen content of 1.225% and this occurred at a chamber pressure of 300 mTorr.

Keywords: Al 6061, plasma nitriding, SEM, EDAX, surface hardness.

1. Introduction
Nitriding is the process of surface hardening by diffusing nitrogen to the metal surface. Plasma nitriding is one of the nitriding processes. Plasma nitriding is the process of treating the surface of a material by inserting nitrogen in the material through plasma. The surface hardness of aluminium could be increased by using plasma nitriding and the use of plasma nitriding as a promising surface treatment [1]. The use of aluminium as parts of motor vehicles replaced other types of metal with the same shape and function. This happens when the product must have good heat-conductive properties, have a low melting point, and have a lightweight. Based on those requirements, aluminium alloy 6061 was chosen as the material for the research.

Besides nitrogen, hydrogen and argon were also used in plasma nitriding. Hydrogen and argon were used before the nitriding process to clean the surface of the material to be nitrided. This cleaning procedure aimed to clean the oxidation layer and the solvent layer that was still contained on the surface of the specimen. This cleaning was useful to stabilize the plasma temperature when the plasma chamber was started and prevent minor changes in temperature.
during the nitriding process. In the nitriding process, hydrogen gas was useful for maintaining the surface of the material to avoid oxidation. A low-temperature plasma nitriding process was proposed as a surface treatment to improve the engineering durability of stainless steel mini-/micro-pipes and nozzles [2].

Plasma nitriding is actually a diffusion phenomenon. For example, is the diffusion of nitrogen into aluminium alloy 6061. Diffusion mechanisms of interstitial elements in FCC systems are generally well-known. In the case of a simple metallic system, e.g. aluminium, the diffusion of interstitial exhibits a wide variety of paths and mechanisms that depend on the specie [3]. The nitrogen content was significantly increased with nitriding time, along with the removal of oxides. Vickers hardness of the Al surface could increase more than thrice for 30 hours nitriding [4]. Plasma assisted nitriding of aluminum alloys offers a suitable alternative to the thermochemical treatment variants used to increase the wear resistance of this material primarily from an environmental effect [5].

The surface morphology of specimen was studied by means of macro scale electrochemical techniques and localized microscopic methods using Scanning electron microscope (SEM) with associated elemental analysis by energy dispersive spectrometer (EDS) [6].

2. Methods and Material

This experimental research was conducted using plasma nitriding process with chamber pressure variations of 100 mTorr, 200 mTorr, and 300 mTorr, constant temperature of 150oC, constant input power of 40 watts, and holding time of 2 hours. Specimens without plasma nitriding treatment were used as a comparison. Figure 1 shows the dimensions of the Al 6061 specimens used in the research. Figure 2 shown the Al 6061 specimens before plasma nitriding.

![Figure 1. Dimensions of specimen (unit: mm)](image1)

![Figure 2. Specimen Al 6061 before plasma nitriding](image2)

Aluminum is a metal that has a low modulus of elasticity, so it is not good when working in cold conditions. From a technical point of view, aluminum’s low elasticity could hardly be improved by means of alloying or heat treatment. Table 1 shows the properties of aluminium. After plasma nitriding was completed, a microstructure test was carried out using SEM and x-ray Microstructure analysis equipped with Energy dispersive X-Ray (EDX) composition analysis. Energy Dispersive X-Ray Analysis (EDX), is an x-ray technique used to identify the elemental composition of materials. X-ray microanalysis carried out in a scanning electron microscope equipped with energy dispersive X-ray spectrometry (EDS) is an essence technology for the microstructure characterization of materials [7]. After that, it was followed by surface hardness testing using the micro Vickers hardness test. The pressure load for the micro Vickers hardness test was 100 grf.
| Properties                                                                 | Purity Al (%)             |
|---------------------------------------------------------------------------|---------------------------|
| Density (kg.m\(^{-3}\)) (20°C)                                            | 2.698                     |
| Melting point (°C)                                                        | 660.2                     |
| Specific heat (Cal. g\(^{-1}•\)°C\(^{-1}\)) (100 °C)                     | 0.222                     |
| Electric conductivity (%)                                                 | 64.91                     |
| Electrical resistance temperature coefficient (°C\(^{-1}\))              | 0.004                     |
| The coefficient of expansion (M.°C\(^{-1}\)) (20-100 °C)                 | 23.86 x 10                |
| Crystal Type                                                              | FCC                       |

3. Research equipment

The plasma system consisted of parallel plates in a vacuum chamber. The electrode of the system was connected to a DC voltage where the anode was at the bottom. The plasma chamber was equipped with a heating system to heat the specimens. The chamber temperature was set at 150°C, then nitrogen was flowing into a vacuum chamber with a constant flow of 50 ml/s. Plasma was generated between parallel plates (electrodes). The following figure was a schematic of the DC Plasma system used in the experiment.

![DC plasma system for plasma nitriding](image)

4. Result and discussion

In this research, the surface hardness test of specimens was carried out for each vacuum chamber pressure. The content of the specimen composition was tested for each variation in the vacuum chamber pressure. A relation diagram was made between the surface hardness of specimens and pressure variations of the vacuum chamber. Vacuum chamber pressures were 100 mTorr, 200 mTorr, and 300 mTorr. Surface hardness test results can be seen in table 2.
| No. | Chamber Pressure (mTorr) | Repetition | Indentation Point | D1 (µm) | D2 (µm) | D avg (µm) | Surface Hardness VHN |
|-----|-------------------------|------------|-------------------|--------|--------|-----------|----------------------|
| 1   | 100                     | 1          | A                 | 20.70  | 19.50  | 20.10     | 224.8                |
|     |                         |           | B                 | 20.47  | 19.61  | 20.04     | 226.1                |
|     |                         |           | C                 | 19.53  | 21.05  | 20.29     | 220.7                |
| 2   | 200                     | 2          | A                 | 19.38  | 17.74  | 18.56     | 263.8                |
|     |                         |           | B                 | 18.54  | 17.46  | 18.00     | 280.3                |
|     |                         |           | C                 | 19.77  | 20.51  | 20.14     | 224                 |
| 3   | 300                     | 3          | A                 | 21.42  | 20.52  | 20.97     | 206.6                |
|     |                         |           | B                 | 21.32  | 20.50  | 20.91     | 207.8                |
|     |                         |           | C                 | 23.21  | 22.19  | 22.70     | 176.3                |

Table 2. Surface hardness test results
Figure 4. The relation between chamber pressure variations and the surface hardness of Al 6061 specimen

Figure 4 shown the relation between chamber pressure and surface hardness. The graph shown that the surface hardness increased at chamber pressure of 100 mTorr and 200 mTorr. At chamber pressure of 300 mTorr there was a decrease in surface hardness. Even so, the surface hardness of the treated specimen was still above the surface hardness of the untreated specimen.

The specimens without plasma nitriding treatment had a surface hardness of 169.1 VHN. Specimen with a chamber pressure of 100 mTorr had a surface hardness of 225.6 VHN. Figure 4 was specimen Al 6061 after plasma nitriding with chamber pressure 100 mTorr and Figure 5 shown a microstructure of specimen Al 6061 after plasma nitriding using chamber pressure 100 mTorr.

The specimen with a chamber pressure of 200 mTorr had a surface hardness of 276.5 VHN and a specimen with a chamber pressure of 300 mTorr had a surface hardness of 179.4 VHN.

The untreated Al 6061 specimen had an FCC crystal structure where there was still empty space between the atoms. This caused the specimen to have a low surface hardness of 169.1 VHN. In contrast, the Al 6061 specimen with chamber pressure variations of 100 mTorr, 200 mTorr, and 300 mTorr after diffusion of nitrogen at a temperature of 150°C and a power input of 40 W, where the atoms in the aluminum could open space so that nitrogen diffused and occupied the empty space on aluminum atoms. This could strengthen the aluminum surface. In this research, the initial Al 6061 specimen did not undergo plasma nitriding (nitrogen diffusion). The initial percentage of aluminum in specimen Al 6061 without plasma nitriding was 97.47%. The microstructure of the Al 6061 specimen without treatment had a brighter color and had a slightly black grain structure compared to the specimens with chamber pressure variations of 100 mTorr, 200 mTorr, and 300 mTorr. Figure 5 shown the microstructure of specimen Al 6061 before plasma nitriding.

When a chamber pressure of 100 mTorr and 200 mTorr was applied, the surface hardness of the Al 6061 specimen increased. After the addition of chamber pressure, the surface hardness of the specimen decreased. These were due to the fact that plasma nitriding produced substitution diffusion, not interstitial diffusion. These were evidenced by the reduction in the percentage of aluminum. In untreated specimens, the aluminum content of the specimens was 97.47% and in
specimens, with a chamber pressure treatment of 300 mTorr, the aluminum content was reduced to 73.511%.

![Figure 5](image1.png)

**Figure 5.** The microstructure of untreated specimens Al 6061 (1000x)

![Figure 6](image2.png)

**Figure 6.** (a) The microstructure of specimen Al 6061 after plasma nitriding with chamber pressure 100 mTorr (1000x), (b) Specimen Al 6061 after plasma nitriding with chamber pressure 100 mTorr

Figures 6(a) and 6(b) shown the Microstructure and Specimens of plasma nitriding at a chamber pressure of 100 mTorr, respectively. It was known that the color of the specimens without plasma nitriding looked brighter and has a slight black grain structure compared to specimens with chamber pressure variations of 100 mTorr, 200 mTorr, and 300 mTorr. These black grains were indicated as carbon caused by the heating process.
Figure 7. (a) The microstructure of Specimen Al 6061 after plasma nitriding with chamber pressure 200 mTorr (1000x), Specimen Al 6061 after plasma nitriding with chamber pressure 200 mTorr

Figure 8. (a) The microstructure of Specimen with chamber pressure 300 mTorr (1000x), (b) Specimen Al 6061 after plasma nitriding with chamber pressure 300 mTorr

Table 3. shown the percentage of alloys element in the untreated Al 6061 specimen.

| Element | Percentage (%) |
|---------|----------------|
| Al      | 97.47          |
| Mg      | 1.01           |
| Si      | 0.88           |
| Fe      | 0.22           |
| Cu      | 0.21           |

There was a decrease in surface hardness of the specimen with a chamber pressure of 300 mTorr. Its surface hardness was 179.4 VHN. Table 4 shown that nitrogen was only able to diffuse 1.225% into the specimen, while oxygen diffused more into the specimen with a percentage of 3.656%. This caused a decrease in the surface hardness of the specimen with a chamber pressure of 300 mTorr. This indicated that the surface hardness value of the specimen was low. A large
chamber pressure caused the atoms to shift to their new positions. Even though the pressure was removed, the atoms remained in new positions, forming an irregular atomic structure. This model of displacement of the atoms was called a slip.

Table 4. Percentage of elements in specimens Al 6061 with chamber pressure 300 mTorr

| Element | Percentage (%) |
|---------|----------------|
| C       | 20.241         |
| N₂      | 1.225          |
| O₂      | 3.656          |
| Mg      | 0.801          |
| Al      | 73.511         |
| Fe      | 0.335          |

![Spectrum 1](image)

Figure 9. Spectrum graph of Al 6061 specimen with chamber pressure 300 mTorr.

From the spectrum graph, it can be seen that there were low atomic shell energies in the elements Carbon, Oxygen and Nitrogen. So that the spectrum waves of these elements had low waves too. Aluminum element had the highest atomic shell energy, but there was a decrease in the percentage of aluminum content in the Al 6061 specimen with a chamber pressure of 300 mTorr.

5. Conclusion

There has been an increase in surface hardness of Al 6061 specimens with chamber pressures of 100 mTorr and 200 mTorr, but the surface hardness decreased in specimens with a chamber pressure of 300 mTorr. It could be predicted that this was due to the type of generated diffusion. The generated diffusion was vacancy/substitution diffusion rather than interstitial diffusion. A large chamber pressure caused the atoms to shift to their new positions. Even though the pressure was removed, the atoms remained in new positions, forming an irregular atomic structure. This phenomenon was commonly referred to as slippage. The evidence of it was the percentage reduction of aluminium in specimen Al 6061 from 97.47% in untreated specimens to 73.511% in specimens with a chamber pressure of 300 mTorr.
6. References

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