



STUDY OF MICROSTRUCTURAL AND CORROSION PROPERTIES OF ALUMINIUM ALLOY 7075 AFTER PLASMA NITRIDING

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ABSTRACT

STUDY OF MICROSTRUCTURAL AND CORROSION PROPERTIES OF ALUMINIUM ALLOY 7075 AFTER PLASMA NITRIDING. Plasma nitriding is a treatment process of metals by depositing nitrogen into metal that considered to be nitrided by mean of increasing the mechanical, physical, and chemical properties of the metal. This treatment will form a hard layer compound of Al-N on the surface of the sample. In this study, aluminium alloy 7075 was nitrided which the application of it to structural part of aircraft makes it vulnerable to not only corrosion and wear attack but also decreasing the hardness of the material. One method to overcome these issues is plasma nitriding. This research uses ammonia gas (NH₃) in the treatment process. The purpose of this research is to do the characterizations of plasma nitrided aluminium alloy 7075 regarding its microstructure, mechanical, and chemical properties. The characterizations that had been done were microhardness Vickers testing, SEM-EDX, and electrochemical corrosion testing Potensiostat. The hardness of the sample increased 55% from 75,88 VHN (raw material) to 117,68 VHN (at optimum parameter). The depth of the white layer of plasma nitriding is approximately 6 µm, while the EDX result reported carbon, oxygen, and nitrogen presence. Corrosion testing showed that the highest corrosion rate is on the raw material, 0,15393 mpy. While the optimum one is 0,07184 mpy.

Keywords: Microstructure, Corrosion, Aluminum alloy, Plasma, Nitriding

ABSTRAK

STUDI MIKROSTRUKTUR DAN SIFAT KOROSI PADUAN ALUMINIUM DENGAN NITRIDASI PLASMA. Nitridasi plasma merupakan proses treatment terhadap logam guna meningkatkan sifat mekanik, sifat fisis, dan kimia dengan mendeposisi nitrogen ke dalam material logam sehingga menghasilkan lapisan tipis senyawa Al-N yang keras pada permukaan logam aluminium. Sampel yang dinitridasi plasma adalah aluminium paduan 7075 yang secara luas digunakan pada struktur pesawat terbang yang membuatnya vulnerable untuk tidak hanya korosi dan serangan keausan tetapi juga penurunan kekerasan material. Salah satu metode untuk mengatasi masalah ini adalah nitridasi plasma. Penelitian ini menggunakan gas amonia (NH₃) dalam proses treatment. Tujuan penelitian ini adalah melakukan karakterisasi terhadap sifat struktur mikro, mekanik, dan kimia sampel setelah dinitridasi. Karakterisasi yang telah dilakukan adalah pengujian microhardness Vickers, SEM-EDX, dan pengujian korosi elektrokimia Potensiostat. Kekerasan sampel meningkat 55% dari 75,88 VHN (bahan baku) menjadi 117,68 VHN (pada parameter optimal). ion nitrogen berdifusi ke dalam sampel diperkirakan hingga 6 µm, sementara hasil EDX menunjukkan adanya tambahan unsur karbon, oksigen dan nitrogen. Pengujian korosi menunjukkan bahwa laju korosi tertinggi ada pada raw material, 0,15393 mpy. Sedangkan untuk sampel dengan parameter optimum, laju korosinya adalah 0,071804 mpy.

Kata kunci: Mikrostruktur, Korosi, Paduan aluminium, Plasma, Nitridasi

INTRODUCTION

Aluminium alloy 7075 is one of the aluminium 7xxx series which was elaborated for the structural material part of aircraft [1]. This alloy is included as aluminium alloy which has the highest strength. Spar is one of the examples of aircraft part which uses aluminium alloy 7075 as its material [2]. It is located in wing of aircraft, and due to tremendous environment exposure during aircraft operation, it could be susceptible to corrosion attack [2,3]. And because of it, not only the lifetime of the aircraft part shortens but also its strength dwindles. That's why a material engineering treatment to improve the properties of aluminium is required. Since the first damage of material happens on surface of material, surface treatment has existed in order to improve mechanical, chemical, and physical properties of material [4]. One of the surface treatment methods that could be applied to elevate aluminium's properties is plasma nitriding [4,5].

Plasma nitriding has various parameters that can be used to do the treatment, such as substrate temperature, processing times, processing chamber pressure, gas composition, and input voltage [6], [7]. Plasma nitriding is more economical than other surface treatment methods and it needs shorter time of processing treatment [6]. Plasma nitriding itself is a surface hardening process of a metallic material involving the deposition of nitride layer and nitrogen diffusion into the metallic material in a vacuum chamber by making nitrogen into a plasma using high voltage between two electrodes (cathode and anode), so that the deposition and diffusion of nitrogen occurred on the surface of the material [8], [9]. Plasma nitriding of aluminium was chosen because aluminium needs low temperature and pressure to form nitride layer on the surface of aluminium, while the conventional nitriding can treat steel only which has higher melting point than aluminium [7]. Although it is slightly hard to do plasma nitriding treatment of aluminium, this treatment apparently has an excellent result of its properties, such as high hardness, good resistance of corrosion and high resistance of wear [5,10].

Plasma nitriding can generate a nitride compound on the surface of the material, which has high hardness properties as the result of plasma process in the vacuum chamber. This nitride compound that formed AlN, not only generate high hardness on the surface of material, but also has high resistance of corrosion. This is due to the properties of nitrogen as the barrier of corrosion process, although AlN compound is hard to form [7-9].

In this study, we report the result of the depth of nitrogen diffusion and corrosion rate of aluminium alloy 7075 after plasma nitriding which used temperature of substrate and processing time parameters, and we explore the correlation among microstructural, corrosion

and hardness of the samples after plasma nitriding process. The motive of some phenomenons in this study is also investigated. this research can provide benefits in terms of reducing the cost of preventive maintenance of the aviation industry using this alloy, as a substitute for damaged parts that have been treated with plasma nitriding techniques.

EXPERIMENTAL METHOD

Aluminium alloy 7075 was used with chemical compositions, after composition testing, were Zn 7,5 %, Mg 2,6 %, Cu 2,1 %, and Al 87,47 %. In this nitriding process using ammonia gas (NH₃), nitriding is carried out in a furnace at a temperature between 373-433 K. This material was initially a sheet which was then cut to disc shape using water jet cutting machine, with the diameter of 14 mm and the thickness of 5 mm, while samples for SEM-EDX was cut cross-sectionally in order to investigate the depth of nitride layer. Samples were then polished using abrasive paper from mesh number 800 up to 2400 until the surface of the samples were mirror-like. After that, the surface of the samples was wiped down using autosol and velvet on the polishing machine. The purpose of this process was to vanish wrinkles and debris on the surface of the samples which was formed after polishing process. The next process was to lave the samples using liquid soap and then alcohol in the ultrasonic cleaner machine for 5 minutes and 20 minutes respectively.

Plasma nitriding used is the design and assembly of the Center for Science and Accelerator Technology of the National Nuclear Energy Agency. Plasma nitriding process was carried out in two steps of the process, the first one was process temperature variation and the second one was process time variation, while pressure was made constant at 1,2 mbar. To regulate the temperature, the voltage regulator was used. While regulating the pressure, the nitrogen gas generated in the vacuum chamber was measured. The temperature variation was 373 K, 398 K, 423 K and 433 K. After the temperature variation process, the hardness of the samples was characterized using microhardness tester Matsuzawa MMT-X7. At a certain temperature, the hardness of a sample was the highest one, so this temperature was used to do the next process variation, process time variation of plasma nitriding. The process time variation was set up from 2 hours, 3 hours, 4 hours to 5 hours. After that, the hardness of the samples was characterized to determine the highest hardness and the optimum parameter of the plasma nitriding process.

The microstructural properties of the sample were characterized using SEM-EDX Oxford Instrument. In this study, the microstructural properties that were investigated were only the depth of the nitrogen diffusion and the comparison chemical elements of the samples before and after process. The corrosion properties of

the sample were characterized using electrochemical method with the instrument of potentiostat Princeton Applied Research Versastat 4. In this study, the corrosion property that was analyzed was only the corrosion rate with the unit of the corrosion rate is mpy (mils per year).

RESULTS AND DISCUSSION

SEM-EDX Analysis

In this characterization, two samples were characterized. The first one was sample with non-optimum parameter which was at temperature 100° C, pressure 1,2 mbar, and time 3 hours. The second one was a sample with optimum parameter which was at temperature 125° C, pressure 1,2 mbar and time 4 hours. As shown in Figure 1, the surface of the sample with the optimum parameter looked whiter than the sample with non-optimum parameter. This is due to the nitride layer presence which is indicated by white layer on the surface [6,11]. This layer has high hardness characteristic and high corrosion resistance [5,7]. The issue was the depth of this layer could not be determined well using SEM, due to the deeper diffusion of the nitrogen ion into the material. But the depth of the nitrogen ion was approximately 6 μm. The white layer on the surface of the sample was suspected not only nitride layer, but also oxide layer. The oxide layer could be formed because of the preparation process in which a lot of oxygen left on the surface and could not be released even after plasma nitriding [7,12]. The other reason why oxide layer might be formed was that the impurity of the nitrogen

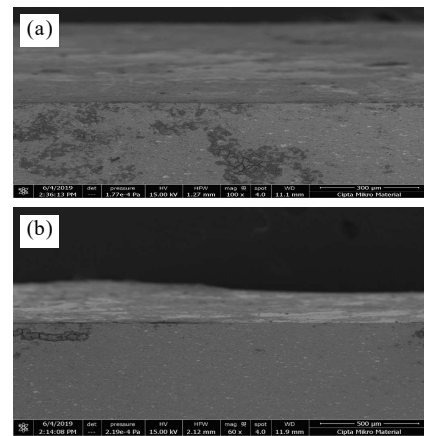


Figure 1. (a). SEM image of sample with non-optimum parameter, (b). SEM image of sample with optimum parameter.

gas used during plasma nitriding which then supported the oxide-forming on the surface of the material [6]. The device that could be used to determine the depth of the nitrogen ion diffusion into the material more precise is Linescan.

EDX result reported addition chemical elements after plasma nitriding. Those chemical elements are oxygen, carbon, and nitrogen, those elements presence was existed both on the sample with non-optimum parameter and optimum parameter but with different percentage. Figure 2 shows the graph of EDX result for both samples. From those figures, we can see that the oxygen percentage on the surface of the sample with non-optimum parameter (100 °C, 3 hours, 1,2 mbar) is 15,7 % while on the surface of the sample with optimum

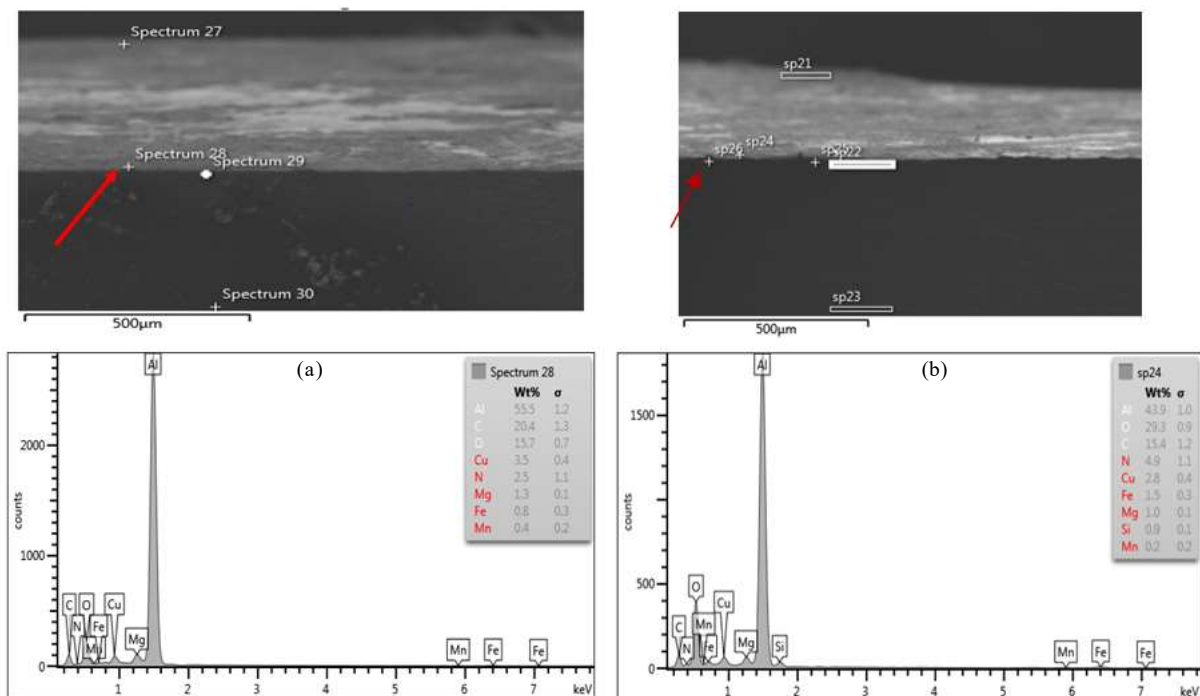


Figure 2. (a) EDX graph of a sample with non-optimum parameter, (b) EDX graph of sample with optimum parameter.

parameter (125 °C, 4 hours, 1,2 mbar) is 29,3 %. The presence of oxygen after plasma nitriding was due to preparation process and the impurity of the nitrogen gas used during plasma nitriding [5]. Another reason why oxygen existed was that aluminium is one of the metallic material which is easy to merge to produce an oxide compound. This process could lead to the formation of the oxide layer which was known as Al_2O_3 [13]. This oxide layer has some properties such as high hardness, and corrosion resistance. That's why the hardness of the sample with higher percentage of oxygen (optimum parameter) is higher than the sample with lower percentage of oxygen (non-optimum parameter). Microhardness measurement in the direction perpendicular to the surface of the sample. The hardness of the sample with optimum parameter is 117,9 VHN, while the hardness of the sample with non-optimum parameter is 99,48 VHN. This will be explained more on hardness test result later. Hassan et al., also stated that plasma nitriding process is followed by oxidation process due to the impurity of gas that is used and the environment of the chamber that probably some oxide particles were existed [6]. Moreover aluminium is easy to form oxide compound than other metals [13].

Carbon was also one of the chemical element that was existed on the surface of the sample after plasma nitriding. Carbon existed because the plasma nitriding machine was used by turns with DLC process (Diamond-Like Carbon) which used carbon and nitrogen as the main gas compositions. The percentage of carbon on the surface of sample with non-optimum parameter (100 °C, 3 hours, 1,2 mbar) is 20,4 % (Figure 2.a), while the percentage of carbon on the surface of sample with optimum parameter (125 °C, 4 hours, 1,2 mbar) is 15,4 % (Figure 2.b). Here, carbon is considered as the impurities of plasma nitriding process. The process of DLC left carbon on cathode which is the place where the sample was put on. Then, carbon was sputtered during plasma nitriding process, and patched on the surface of samples.

The main gas of this process is nitrogen so it is not astonishing that nitrogen existed on the surface of samples. The EDX result (Figure 2.a) shows that the percentage of nitrogen on the surface of the sample with non-optimum parameter (100 °C, 3 hours, 1,2 mbar) is 2,5 %, while on the sample with optimum parameter (125 °C, 4 hours, 1,2 mbar) is 4,9 % (Figure 2.b). Nitrogen presence is because of melting process at the bottom of the sample due to bombardment of the nitrogen simultaneously. Then, samples' atoms were sputtered and combined with nitrogen ion and formed a nitride compound which was then adsorbed on the surface of the sample which is then considered as a white hard layer. The percentage of nitrogen and oxygen was influenced by the time duration of the process. The result reported that sample with non-optimum parameter (3 hours process) has lower percentage of nitrogen and oxygen than the sample with optimum parameter (4 hours process). Furthermore, the percentage of oxygen on both type of samples is higher than the percentage of nitrogen. This is due to the impurity of the gas used and the oxygen that was patched during preparation process which was hard to decompose if it had been combined with aluminium and then formed Al_2O_3 compound. This compound interfered the formation of AlN compound by preventing the diffusion of nitrogen ion [6,14]. Another reason why the percentage of nitrogen is lower than oxygen on the surface of samples is that the dimension of AlN crystal is very small which is only 10 nm, that's why the diffusion process of nitrogen could be interfered by Al_2O_3 compound [14]. From this exposition, the white layer of SEM image of samples mainly consisted of AlN and Al_2O_3 compound.

Microhardness Analysis

The hardness of the sample was measured using microhardness testing device Matsuzawa MMT-X7. There were three types of samples that were

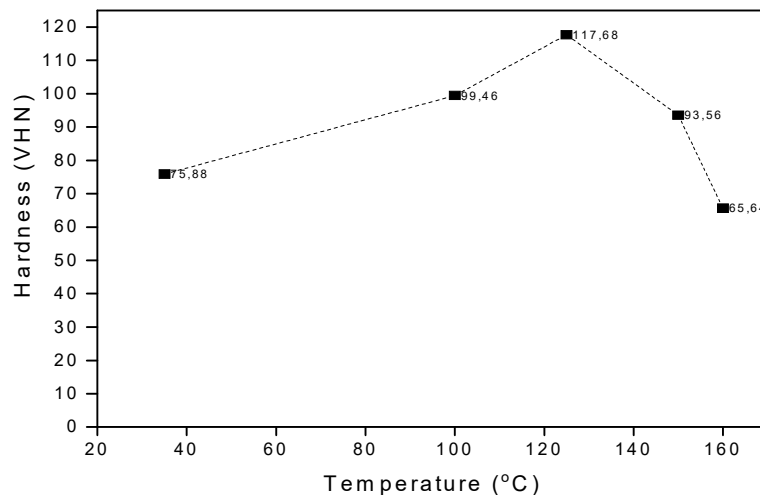


Figure 3. Hardness graphic after temperature variation.

characterized, they were raw material, which was untreated, samples which were treated after temperature variation and samples which were treated after time variation. The load of the hardness testing was 10 gf (gram force), and this load was applied to all samples, so there was no hardness load variation. During temperature variation, time and pressure were constant, for 3 hours and at 1,2 mbar. These parameters were obtained from precedented research of plasma nitriding of aluminium. Figure 3 shows the hardness graphic of samples after temperature variation, from this graphic it can be seen that the hardness of raw material was only 75,88 VHN, but there was an anomaly of this result. After the hardness of the sample got the highest value, the hardness of the sample decreased as the temperature increased, even at the highest temperature 160 °C, the hardness of the sample is lower than raw material which was only 65,64 VHN. The peak hardness of the sample was 117,68 VHN at temperature of 125 °C.

The temperature of 125 °C was considered as the optimum temperature because, at this point, the highest hardness of the sample was obtained. The decreasing hardness of the sample after the highest hardness was obtained at 125 °C was due to supersaturated solid solution strengthening phenomenon. This phenomenon occurred because the grain size of the sample expanded, Ebisawa et al. said that the grain size has inversely proportional correlation with the hardness of the sample. The bigger the grain size, the lower the hardness of the sample. Another reason why this occurred was that aluminium had reached the peak of its maximum temperature for plasma nitriding process. Visuttipitukul et al. stated that the maximum temperature of material for plasma nitriding process was 1/3 – 2/3 from material’s melting point. On the other hand, Goodfellow catalogue, provider data about some properties of some materials, indicate that the melting point of aluminium alloy 7075 is 447 °C – 635 °C. So that the maximum temperature of aluminium alloy 7075 for plasma nitriding process is

around 149 °C up to 211,6 °C. That’s the reason why at temperature 150 °C and 160 °C, the hardness of the sample decreased [17].

Time variation was done using optimum temperature of 125 °C, while the pressure was kept constant at 1,2 mbar. The phenomenon that occurred during time process variation was the same with temperature variation, the hardness of the sample decreased after the highest hardness reached. Figure 4 shows the graphic of hardness of samples after time variation at temperature of 125 °C and at pressure of 1,2 mbar. From the graphic it can be seen that the hardness of the sample decreased after 4 hours, and 4 hours is considered as the optimum time duration of plasma nitriding process of aluminium alloy 7075, because at this point, the highest hardness was obtained which is 117,9 VHN.

Corrosion Analysis

Corrosion testing was held using the instrument of potentiostat Princeton Applied Research Versastat 4, the liquid media instead of NaCl 0,5 mol/L as inhibitor. In this characterization, three samples were used, they were raw material, sample with non-optimum parameter (100 °C, 1,2 mbar, 3 hours) and sample with optimum parameter (125 °C, 1,2 mbar, 4 hours). This corrosion analysis is only focused on the corrosion rate, not type of corrosions. Corrosion rate unit which used is mpy (mils per year, 1 mils= 0,0254 mm/year). From this characterization, we got a graph of E_{corr} vs I_{corr} which was then extrapolated on the software Versastudio, this extrapolation process would produce the real I_{corr} used for corrosion rate calculation. The Equation (1) is to calculate corrosion rate is given below:

$$CR (mpy) = 0,13 \frac{I_{corr} \times EW}{\rho} \dots\dots\dots (1)$$

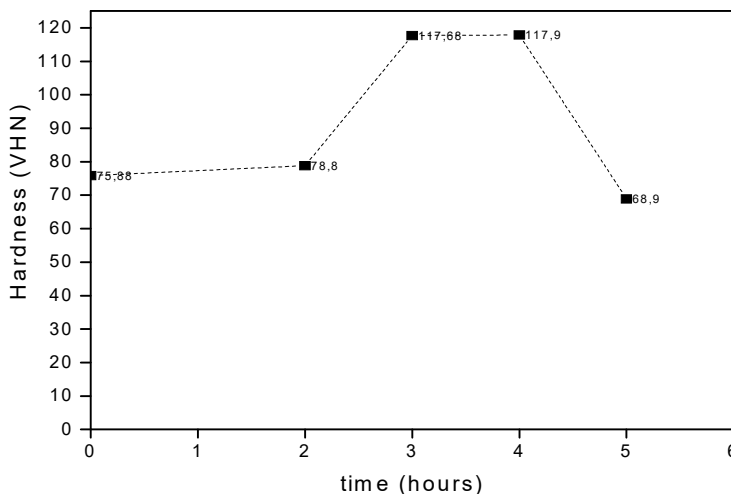


Figure 4. Hardness graphic after time variation

where CR is corrosion rate which stated in mpy (mils per year), I_{corr} is corrosion current density in $\mu A/cm^2$, EW is the equivalent weight of sample in gr/mol, \bar{n} is density of sample in gr/cm^3 [15]. I_{corr} of each samples were 0,4336206 $\mu A/cm^2$ for raw material, 0,2187606 $\mu A/cm^2$ for sample with non-optimum parameter (100 °C, 1,2 mbar, 3 hours), and 0,202272 $\mu A/cm^2$ for sample with optimum parameter (125 °C, 1,2 mbar, 4 hours). EW for aluminium alloy 7075 that was used in this study was 9,8 gr/mol, while \bar{n} of the sample is 2,817 gr/cm^3 . By using these data and Equation 3.1, we could calculate the corrosion rate of each sample. The corrosion rate of raw material is 0,15393 mpy, corrosion rate of sample with non-optimum parameter (100 °C, 1,2 mbar, 3 hours) is 0,077638 mpy, and corrosion rate of sample with optimum parameter (125 °C, 1,2 mbar, 4 hours) is 0,071804 mpy.

Raw material has the highest corrosion rate, 0,15393 mpy, which means that untreated sample is the fastest to be corroded of other samples. It is surely because there was no treatment or improvement process on to the surface of the sample. While the slowest sample to be corroded is sample with optimum parameter (125 °C, 1,2 mbar and 4 hours) which is 0,071804 mpy. This is due to high concentration of nitrogen after plasma nitriding process (4,9%), furthermore the SEM image of the sample shows that the surface of the sample is whiter than other sample, which indicated the presence of nitride and oxide layer as explained before [6], [14]. This is suspected to be the protection layer against corrosion attack, which was then resulted in the lowest corrosion rate. This condition of sample with non-optimum parameter (100 °C, 1,2 mbar and 3 hours) is inversely proportional to sample with optimum parameter. The corrosion rate is higher than the corrosion rate of sample with optimum parameter which is 0,077638 mpy. Although the difference is not quite fair, the concentration of nitrogen also influenced the corrosion rate of both samples. The sample with non-optimum parameter (100 °C, 1,2 mbar and 3 hours) has concentration of

nitrogen lower than sample with optimum parameter. Non-optimum one has only 2,5 % of nitrogen while the optimum one (125 °C, 4 hours, 1,2 mbar) has 4,9 % of nitrogen. This evidence makes clear that the concentration of nitrogen also influenced the corrosion resistance of the samples. The hardness of the sample has correlation with the corrosion resistance as well. The hardness of the sample with non-optimum parameter (100 °C, 1,2 mbar and 3 hours) 99,46 VHN, which has higher corrosion rate, is lower than the sample with optimum parameter (125 °C, 4 hours, 1,2 mbar) 117,98 VHN. Aziz et al., stated that the correlation between corrosion rate and hardness is inversely proportional, which means the higher the hardness, the lower the corrosion rate [16].

The corrosion testing of potentiostat method process which include current and voltage, has influenced the result of the corrosion rate and give evidence regarding the corrosion rate of each sample. Figure 5 shows I_{corr} vs E_{corr} graphic of corrosion testing, that the sample with optimum parameter (125 °C, 1,2 mbar, 4 hours), the black line, is more positive (most right) in voltage. While sample with non-optimum parameter (100 °C, 3 hours, 1,2 mbar), the blue line, has almost the same voltage with sample with optimum parameter and has the lowest current of all samples. The yellow line is graph for untreated material (raw material), which has the most negative voltage of all samples.

CONCLUSIONS

The study of plasma nitriding of aluminium alloy 7075 using temperature and time variation of the process and its characterizations had been done. The presence of nitrogen onto the surface of the samples after plasma nitriding had proved that this process is able to improve hardness and decrease corrosion rate. The presence of nitride layer on the sample was shown by white layer on SEM image. Nitride layer was not the only cause of

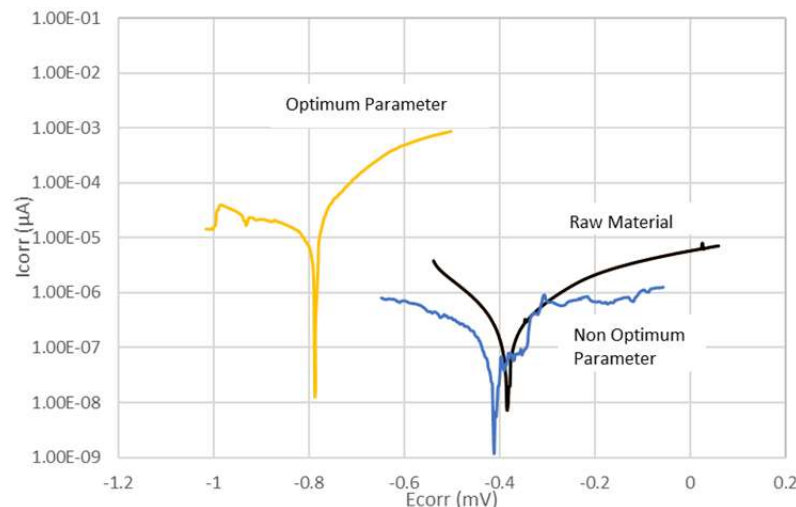


Figure 5. I_{corr} vs E_{corr} graphic of corrosion testing.

Table 1. Correlation among characterizations

Samples	EDX result		Hardness	Corrosion rate
	Nitrogen (%)	Oxygen (%)	VHN	mpy
Raw material	0	0	75,88	0,15393
Non-optimum sample (100 °C, 1,2 mbar, 3 hours)	2,5	15,7	99,46	0,077638
Optimum sample (125 °C, 1,2 mbar, 4 hours)	4,9	29,3	117,9	0,071804

hardness improvement and corrosion rate decrease but the presence of native oxide layer on the surface of the sample also contributed to the hardness improvement, corrosion rate decrease and white layer on the surface of samples. This evidence is proved by EDX result which showed a high percentage of oxygen. The correlation among these characterizations can be seen on Table 1 below. From the table, we can understand that the presence of nitrogen and oxygen had influenced the hardness and corrosion rate of samples. The highest hardness and the lowest corrosion rate was obtained when the percentage of nitrogen and oxygen is the highest one at the optimum parameter, while the lowest hardness and the highest corrosion rate was when there were no nitrogen and oxygen at raw material.

The highest hardness, 117,9 VHN, was obtained at the parameter temperature of 125 °C, pressure 1,2 mbar, and time 4 hours which is then called as an optimum parameter of plasma nitriding process of aluminium alloy 7075. The increasing percentage of hardness from raw material is approximately 55 %.

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