Study of the hardness and microstructure profiles of SKD61 steel plunger tip (local material) after plasma nitriding

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Abstract. The Hardness and Microstructure Profiles of SKD61 Steel Plunger Tip (local material) after Plasma Nitriding have studied. To improve SKD61 quality for high temperature use, a plunger tip can be hardened through surface treatment using a plasma/ion nitriding technique. To test the success of the surface treatment, the nitriding temperature was varied from 350ºC to 550ºC with step for 50 ºC for the nitriding process completed for 10 hours. The results, it can be seen that the optimum surface hardness was achieved when the nitriding temperature was 500ºC with Vickers hardness (HV) was 955.29 HV with a depth of up to 100 μm from the surface of the specimen. At such a nitriding temperature, the hardness increased 1.79 times in comparison with that after hardening only, and it increased 2.076 times in comparison with that of the initial hardness. From the results of the study, it can be concluded that plasma nitriding temperature affects the amount of nitrogen concentration diffused into the substrate material. Nitride layer formed was possibly of Fe N, Cr₂N, VN, δ Fe, and γ Fe.

Keywords: Plasma/Ion Nitriding, SKD61 Steel, High Pressure Die Casting.

1. Introduction

A plunger tip is an important component in a high pressure die casting of cold chamber type that serves as an aluminum fluid booster in the cold sleeve to fill in the mold cavity. The plunger tip is always rubbing against the inner wall of the cold sleeve or in direct contact with the aluminum metal fluid poured in the cold sleeve or chamber. To keep the expansion steady, there is a cooling water circulation inside the plunger tip. The water circulation goes through a small pipe connecting the plunger tip and the hydraulic cylinder.

![Figure 1. A 65 x 90 mm Plunger Tip](image_url)

With a high pressure, aluminum fluid is pushed by the plunger tip in to the mold cavity/dies. Friction between the surfaces results in wear and tear. Besides the wear problem due to friction between metallic surfaces, the plunger tip also warms up due to direct contact with aluminum fluid during operation. For a durable plunger tip, it needs a material that is strong, wear-resistant, and it does not experience a decrease in hardness at high temperatures. Typically, a plunger tip in a high pressure die casting machine is made of hardened SKD61 steel with a hardness of 457.6 - 499.2 HV. For high temperature uses, hardening treatment alone is not enough. An additional surface treatment needs to be done on the plunger tip material to improve its quality at high temperature operation. A research had been conducted to observe...
the effect of plasma nitriding on the hardness and the microstructure profiles of SKD61 steel as a plunger tip component of high pressure die casting machines.

2. Research methodology
The specimens used in this research were SKD61 steel, each had a size of 22 x 20 mm. The steel was the material for a 65 x 95 mm plunger tip obtained from PT ASSAB Steels Indonesia. SKD61 steel is equivalent to ASSAB 8407 2M (a standard material of PT ASSAB Steels Indonesia) which has the following composition: C 0.39 - Si 1.0 - Mn 0.4 - Cr 5.3 - V 0.9 - Mo 1.3. The ASSAB 8407 2M material has an initial hardness of 460 HV. At such hardness, the process of forming or cutting the metal is easier. Generally after the cutting or machining is done, the material undergoes hardening process to achieve the standard level appropriate for a plunger tip. The typical hardness of SKD61 material is 499.2-572 HV. In this research, each specimen was cleaned with distilled water. After that, each one was cleaned with acetone. The natural oxide layer on the surface of the specimen was etched with HF for one to three minutes. Each specimen was cleaned again with distilled water. Then all specimens were sprayed with nitrogen gas. After that, the specimens were hardened on their surface with DC plasma nitriding [see Fig. 2] at temperatures of 350ºC, 400ºC, 450ºC, 500ºC, and 550ºC for 10 hours. Nitrogen gas used in the experiment was of high purity. Then specimens were cut, mounted, polished, and etched for microstructure observation with an optical microscope. The surface hardness of the specimens were tested with Vickers AKASHI MVK micro-machine - E load (g) 10 - 1000. The load was 100 gram-force.

3. Results and Discussion

3.1. Initial SKD61 Composition and Hardness Test

Based on the supplier's material certificate issued by PT ASSAB Steels Indonesia No: 076/8407 2M/13 on 14 January 2013, the composition of SKD61 or ASSAB 8407 2M was as follows:
C 0.39 - Si 1.0 - Mn 0.4 - Cr 5.3 - V 0.9 - Mo 1.3. The material hardness was 460 HV.

3.2. Hardness Testing of SKD61 Specimens

The hardness test was carried out using Vickers AKASHI MVK micro-machine - E load (g) 10 - 1000. The load was 100 gram-force. Vickers testing was carried out on each specimen after hardening and plasma nitriding at 350ºC, 400ºC, 450ºC, 500ºC, and 550ºC.

3.2.1. SKD61 Hardness after Hardening Process.
Hardness testing with Vickers technique for SKD61 specimens after hardening was carried out by tracking at depths of ranging from 0.02 mm to 2 mm from the specimen’s surface and using 100 gram-force load as shown in Figure 3.

For the hardness test, ten observation points were determined, i.e. at the depth of: (1) 0.02 mm, (2) 0.05 mm, (3) 0.10 mm, (4) 0.15 mm, (5) 0.20 mm, (6) 0.30 mm, (7) 0.50 mm, (8) 1.00 mm, (9) 2.00 mm, and (10) beyond 2.00 mm (core). Measurement results showed that the hardness of SKD61 steel after the plasma nitriding increased from the initial hardness of 460 HV to 531.32 HV. Such a difference in hardness was due to the hardening process carried out in vacuum where the cooling was done with N₂ medium. At the surface that is the first observation point, the hardness was 531.32 HV, while at the depth of 2.00 mm (core), the hardness was 460.27 HV.

3.2.2. **SKD61 Hardness after Plasma Nitriding at 350°C.**
Hardness testing with the Vickers technique for SKD61 specimens after plasma nitriding was carried out by tracking at the depth from 0.02 mm to the 0.3 mm from the surface and using 100 gram-force load as shown in Figure 4.

For the hardness test, ten observation points were determined, i.e. at the depth of: (1) 0.02 mm, (2) 0.03 mm, (3) 0.04 mm, (4) 0.05 mm, (5) 0.06 mm, (6) 0.08 mm, (7) 0.10 mm, (8) 0.20 mm, (9) 0.30 mm,
and (10) beyond 0.30 mm (core section). Measurement results showed that the hardness of SKD61 steel after plasma nitriding increased from its hardness after hardening of 531.32 HV to 818.20 HV.

3.2.3. SKD61 Hardness after Plasma Nitriding at 400ºC.

Hardness testing with the Vickers technique for SKD61 specimens after plasma nitriding was carried out by tracking at the depth from 0.02 mm to the 0.4 mm from the surface and using 100 gram-force load as shown in Figure 5.

![Figure 5](image)

Figure 5. Hardness of the specimen after 10-hour plasma nitriding at 400ºC

The hardness at the surface (at 0.02 mm depth) of the specimen treated with plasma nitriding at 400ºC was lower than that at 350ºC, that was from 818.20 HV to 776.70 HV. However, the hardness at 400ºC nitriding temperature was more evenly distributed than that at 350ºC nitriding. This is probably due to the accumulation of nitrogen atom layers on the surface of the specimen resulting in decreased hardness. The effect of the buildup of metal nitride layers on the surface can lead to a decrease in hardness. This phenomenon can occur if there is no equilibrium between the diffusion rate of N atoms on the surface and the deposition rate. The diffusion rate of N atoms on a metal surface is strongly influenced by the temperature of the substrate while the nitrogen ion deposition rate is affected by nitriding pressure. If the deposition rate is too large but diffusion and reaction of nitrogen atom (N) with substrate (Fe, Cr, Al, Mo, W, and Ti) cannot follow, it is possible to accumulate nitrogen atoms on the surface causing surface hardness to rise but will go down [1]. The results showed that the hardness decreased along with the depth until it reached the initial hardness of the SKD61 specimen. The optimum hardness was reached at a depth of 0.04 mm or 4μm. This indicates that the deeper the concentration of nitrogen at a lower concentration and the possibility of the occurrence of metal nitride compounds, the fewer the resulting violence decreases. On the surface, the hardness was 776.70 HV, while at a depth of 0.40 mm the hardness was 485.40 HV.

3.2.4. SKD61 Hardness after Plasma Nitriding at 450ºC.

Hardness testing with the Vickers technique for the SKD61 specimens after plasma nitriding was carried out by tracking the depth from 0.02 mm to the tracking depth of 0.4 mm from the surface and 100 gram-force load as shown in Figure 6.
For the hardness test, ten observation points were determined, i.e. at the depth of: (1) 0.02 mm, (2) 0.03 mm, (3) 0.04 mm, (4) 0.05 mm, (5) 0.06 mm, (6) 0.08 mm, (7) 0.10 mm, (8) 0.20 mm, (9) 0.30 mm, and (10) beyond 0.40 mm (core section). Measurement results showed that at the surface, the hardness of SKD61 steel after 450°C plasma nitriding increased from its hardness after 400°C plasma nitriding, that is from 776.70 HV to 911.58 HV. The results showed that the hardness decreased along with the depth until it reached the initial hardness of the SKD61 specimen. The optimum hardness was reached at a depth of 0.03 mm or 3 μm. This indicates that the deeper the concentration of nitrogen at a lower concentration and the possibility of the occurrence of metal nitride compounds, the fewer the resulting violence decreases. On the surface, the hardness was 911.58 HV, while at a depth of 0.40 mm the hardness was 485.42 HV. This indicates that the change in hardness at 450°C nitriding includes up to 0.40 mm depth until the hardness is the same as the initial hardness of the material before hardening.

3.2.5. SKD61 Hardness after Plasma Nitriding at 500°C.
Hardness testing with the Vickers technique for the SKD61 test sample after the plasma nitriding process was carried out by tracking the depth from 0.02 mm to the tracking depth of 0.4 mm from the sample surface and 100 gram-force load as shown in Figure 7. For the hardness test, ten observation points were determined, i.e. at the depth of: (1) 0.02 mm, (2) 0.03 mm, (3) 0.04 mm, (4) 0.05 mm, (5) 0.06 mm, (6) 0.08 mm, (7) 0.10 mm, (8) 0.20 mm, (9) 0.30 mm, and (10) beyond 1.0 mm (core section).

Figure 6. Hardness of the specimen after 10-hour plasma nitriding at 450°C.

Figure 7. Hardness of the specimen after 10-hour plasma nitriding at 500°C.
Based on the results of the measurement of the hardness shown in the graph above, the measurement results of SKD61 material after the plasma nitriding process increased from the previous hardness at nitriding temperature of 450°C which was 911.58 HV to 955.29 HV. Based on the test results show that the deeper the surface of the hardness value decreases until it reaches the same hardness as the initial material of the SKD61 specimen. The optimum hardness value is reached at 0.05 mm or 5μm depth. This indicates that the deeper the concentration of nitrogen at a lower concentration and the possibility of the occurrence of metal nitride compounds, the fewer the resulting violence decreases. On the surface edge of the first tracking sample it obtained hardness of 955.29 kgf/mm², while at a depth of 1.00 mm (core) the hardness value obtained was 485.42 kgf/mm². This indicates that the resulting hardness change in the nitriding process temperature of 500°C covers a depth of 1mm until the resulting hardness is the same as the initial hardness of the material before hardening, which is 460 HV at a depth of 2 mm from the metal surface. Based on the results of the measurement of the hardness shown in the graph above, the measurement results of SKD61 material after the plasma nitriding process increased from the previous hardness at nitriding temperature of 450°C which was 911.58 HV to 955.29 HV. Based on the test results show that the deeper the surface of the hardness value decreases until it reaches the same hardness as the initial material of the SKD61 specimen. The optimum hardness value is reached at 0.05 mm or 5μm depth. This indicates that the deeper the concentration of nitrogen at a lower concentration and the possibility of the occurrence of metal nitride compounds, the fewer the resulting violence decreases. On the surface edge of the first tracking sample it obtained hardness of 955.29 kgf/mm², while at a depth of 1.00 mm (core) the hardness value obtained was 485.42 kgf/mm². This indicates that the resulting hardness change in the nitriding process temperature of 500°C covers a depth of 1mm until the resulting hardness is the same as the initial hardness of the material before hardening, which is 460 HV at a depth of 2 mm from the metal surface.

3.2.6. **SKD61 Hardness after Plasma Nitriding at 550°C.**

Hardness testing with the Vickers technique for the SKD61 test sample after the plasma nitriding process was carried out by tracking the depth from 0.02 mm to the tracking depth of 0.4 mm from the sample surface and 100 g tracking load as shown in Figure 8.

![Figure 8. Hardness of the specimen after 10-hour plasma nitriding at 550°C](image)

Based on the results of the hardness test, it can be seen the hardness value of each tracking. Namely: 1) 1st tracking at 0.02mm, 2) 2nd tracking at 0.03mm, 3) 3rd tracking at 0.04mm, 4) 4th tracking at 0.05mm, 5) tracking 5th at 0.06 mm, 6) 6th tracking at 0.08 mm, 7) 7th tracking at 0.10 mm, 8) 8th tracking at 0.20 mm, 9) 9th tracking at 0.30 mm, and tracking above 1.0 mm is the 10th tracking (core / core section). The optimum hardness value is reached at a depth of 0.1 mm or 100μm. This indicates that the nitrogen diffusion zone is getting better inside Based on the results of the measurement of the hardness shown in the graph above, the measurement results of SKD61 material after the plasma
nitriding process at a temperature of 550ºC decreased from the previous hardness at nitriding temperature of 500ºC which was 955.29 HV to 911.58 HV. However, if we see the spread of nitriding hardness of 550ºC more evenly than nitriding at a temperature of 500ºC, this can be seen from the difference in hardness value at 0.3mm depth on the nitriding test specimens of 550ºC higher than the hardness at nitriding temperature 500ºC with the depth of hardness same. While the decrease in hardness on the surface of nitriding samples at a temperature of 550ºC is due to an imbalance between the rate of deposition to the rate of diffusion or the reaction of nitrogen atoms (N) so that accumulation occurs. Deposition rate is influenced by nitriding pressure while deposition rate is influenced by substrate temperature [1].

Based on the test results show that the deeper the surface of the hardness value decreases until it reaches the same hardness as the initial material of the SKD61. Based on the test results show that the deeper the surface of the hardness value decreases until it reaches the same hardness as the initial material of the SKD61 specimen. The optimum hardness value is reached at a depth of 0.1 mm or 100μm. This indicates that the nitrogen diffusion zone is getting better into the surface to produce the optimum hardness value. At the surface edge of the first tracking sample it obtained a hardness of 911.58 kg f/mm², while at a depth of 1.00 mm (core) the hardness value obtained was 485.42 kg f/mm². This indicates that the resulting hardness change in the nitriding process temperature of 550ºC covers up to a depth of 1mm until the resulting hardness is the same as the initial hardness of the material before hardening which is 460 HV at a depth of 2 mm from the metal surface. From the results of measurements and testing of hardness of SKD61 specimen material after the temperature nitriding process of 350ºC, 400ºC, 450ºC, 500ºC, and 550ºC a data analysis can be taken as shown in Table 1.

| Depth (mm) | Hardness Vickers at Nitriding Temperature |
|-----------|-----------------------------------------|
|           | 350 ºC       | 400 ºC       | 450 ºC       | 500 ºC       | 550 ºC       |
| 0.02      | 818.20       | 776.70       | 911.58       | 955.29       | 911.58       |
| 0.03      | 783.41       | 726.20       | 911.58       | 955.29       | 911.58       |
| 0.04      | 579.11       | 720.30       | 870.79       | 946.30       | 870.79       |
| 0.05      | 565.55       | 675.10       | 803.98       | 903.19       | 847.63       |
| 0.06      | 516.34       | 634.00       | 750.84       | 894.93       | 840.11       |
| 0.08      | 498.81       | 624.30       | 638.94       | 702.80       | 832.69       |
| 0.10      | 488.72       | 601.10       | 619.58       | 638.94       | 825.37       |
| 0.20      | 472.56       | 570.70       | 570.66       | 531.04       | 570.66       |
| 0.30      | 472.56       | 565.50       | 531.04       | 509.22       | 531.40       |
| 0.40      | 472.56       | 485.40       | 485.42       | 485.42       | 485.42       |

From figures above, it can be seen that the optimum hardness value is achieved when the hardness of 0.1 mm or 100 μm with the lowest hardness is reached at 350ºC and the highest hardness is reached when the nitriding temperature is 550ºC.

3.2.7. EDS (Energy Dispersive Spectroscopy) Testing.

The sample was cut using diamond saw so as not to damage the surface profile. In this study the SEM tool used is the JEOL type JSM-6390A brand which is equipped with EDS (Energy dispersive X-Ray Spectroscopy). EDS is used to determine the amount of concentration of element N which diffuses into
the surface of the material. At this stage the position of observation is taken at the cross section of the sample surface. The results of this test are shown in Table 2.

| Element | Mass Percentage (%) for temperature process of DC Plasma Nitriding |
|---------|---------------------------------------------------------------|
|         | 350 °C  | 400 °C  | 450 °C  | 500 °C  | 550 °C  |
| N⁴      | 0.36    | 1.19    | 1.28    | 1.28    | 1.75    |
| O       | 4.33    | -       | 4.34    | 4.34    | -       |
| V       | 1.04    | 1.14    | 0.42    | 0.42    | -       |
| Cr      | 6.54    | 4.13    | 5.03    | 5.03    | 4.60    |
| Fe      | 87.72   | 93.36   | 88.92   | 88.92   | 93.65   |

From the results were obtained that after nitriding at 350°C has a nitrogen content of 0.36% mass. The appearance of O atoms in the EDS data is caused by the presence of oxygen trapped inside the nitriding chamber during the nitriding process. Other compounds that may be formed are Cr₂N, and VN so on EDS data elements Cr and V are detected [12, 16]. From the test results it was found that for the nitrided area after being at 400°C it had nitrogen content of 1.19% mass. From the test results it was found that after being nitrided at 450°C it had nitrogen content of 1.28% mass. From the test results it was found that after being nitrided at a temperature of 500°C it had a nitrogen content of 1.54% mass. From the test results it was found that after nitriding at a temperature of 550°C it had a nitrogen content of 1.75% mass. Seeing from the results of testing the qualitative analysis of plasma nitriding temperature 350°C, 400°C, 450°C, 500°C, and 550°C for 10 hours that the influence of temperature affects the amount of diffuse nitrogen concentration or deposited on the surface of SKD61 material test specimens. The higher the nitriding temperature of plasma, the higher the nitrogen content obtained. The nitrogen element that exists when compared to the FeN diagram [17] is likely to form compounds Fe₂N, Fe₃N, Fe₄N, AlN, VN, Cr₂N. The properties of the nitriding compounds are very hard. However, for SKD61 material types classified as low carbon steel (<0.8% C) the ideal nitrogen element content is 11.2% mass so that Fe₂N compounds that have very hard properties can be obtained. The effect of plasma nitriding temperature on nitrogen concentrations obtained in SKD61 steel is shown in Tables 3 and Figure 9.

| Nitridation Temperature (°C) | Nitrogen Concentration (Mass%) |
|-----------------------------|--------------------------------|
| 350                         | 0.36                          |
| 400                         | 1.19                          |
| 450                         | 1.28                          |
| 500                         | 1.54                          |
| 550                         | 1.75                          |
Metallographic testing with Scanning Electron Microscope.

Metallographic tests are needed to determine the microstructure of SKD61 material especially after the nitriding process. This test was carried out using SEM whose results are shown in Figure 10.a for nitriding temperature of 350ºC, Figure 10.b for nitriding temperature of 400ºC, Figure 10.c for nitriding temperature of 450ºC, Figure 10.d for nitriding temperature of 500ºC, and Figure 10.e for nitriding temperature of 550ºC.
Figure 10. Microstructure Nitriding plasma at (a) 350ºC, (b) 400ºC, (c) 450ºC, (d) 500ºC, and (e) 550ºC, process for 10 hours

From the observation of microstructure using SEM the hard layer of Fe₄N and Fe₂N nitride compounds cannot be clearly seen, this is due to the nitrogen concentration obtained 0.36% N. If compared to the Fe-N diagram the nitrogen content below 1% is likely to dissolve in the Fe atom which forms the δFe phase [15]. The phase formed by delta iron (δFe) is a phase of the diffusion zone in which nitrogen atoms dissolve interstitially in ferrite matrix [17]. In Figure 10.b, SEM observations can be seen that the hard layer of nitride is formed, possibly the hard nitride formed is FeN, δFe, but the nitrogen element is still soluble in the Fe atom [15]. From the results of SEM nitriding observations on 450ºC temperature specimens, it was very clearly seen the hard nitride layer, when compared to the Fe-N diagram, the possible phase formed was Fe because the nitrogen elements obtained are 1.28%, however this type of phase was not harder than the phase Fe₂N, Fe₄N, and ε phase [15]. From the results of SEM nitriding observations on 500ºC temperature specimens, it is clear that the nitride hard layer, when compared to the Fe-N diagram, is likely to be formed γFe because the nitrogen elements obtained are 1.75% [15], however the phase concentrations obtained are still more a lot compared to the nitriding result of 450ºC. Apart from the δFe phase, other hard nitrides may also be formed such as Cr₂N, VN.

Figure 11. View of diffusion zone of Nitriding at 550ºC plasma for 10 hours

From the results of SEM nitriding observations in 550ºC temperature specimens, it is very clear that the nitride hard layer, when compared to the Fe-N diagram, is likely to form δFe because the nitrogen element obtained is 2.68% mass [15], however the phase γFe concentration obtained is still more than the
nitriding result of 500ºC. Other compounds formed are like VN, Cr2N if you look at the EDS data obtained.

From Figure 11, the results of the nitriding SEM observations at a temperature of 550ºC with a magnification of 100 X, it can be seen that the diffusion zone is the phases where nitrogen elements dissolve in the Fe atom. According to ASM vol 4 p. 946, the nitride layer formed from nitriding results with nitrogen concentrations below 5% is a diffusion zone, while compound layer zones are zones where hard nitride phases such as Fe4N, Fe2N are formed when the nitrogen concentration is above 5% mass [15].

Overall, if you see the results of SEM nitriding of SKD61 specimens you can conclude that nitridation at 350ºC produces the amount the lowest nitrogen compared to nitriding temperatures of 400ºC, 450ºC, 500ºC, and 550ºC. The effect of temperature also determines the diffusion of nitrogen that occurs, resulting in hard nitride phases such as Fe-N, γ-Fe, α-Fe, δ-Fe. According to ASM volume 4 p 946 [19, 20] to obtain hard phases such as Fe4N, and Fe2–3N during the nitriding process, it is necessary to pay attention to the equilibrium between the gas mixture N2 and H2. The hard structure of the nitride Fe4N layer is obtained when nitrogen gas flowing in the nitriding chamber is 15-30% nitrogen, and Fe2–3N nitride can be obtained by flowing 60 - 70% nitrogen with a mixture of C2H4 (methane) 1 - 3% [11-20]. The recapitulation of the test results and observations is shown in Table 4 as follows:

| Item                        | Before Hardening | After Hardening | Temperature process DC plasma nitridation (ºC) |
|-----------------------------|------------------|-----------------|-----------------------------------------------|
| Hardness (HV)               | 460              | 531.32          | 350 601.1                                    |
| Depth (mm)                  | 2                | 2               | 488.7 619.6                                  |
| Microstructure              | Ferrite-pearlite | martensite      | FeN, FeN, FeN, CrN                           |
| Nitrogen Concentration (%)  | 0                | 0               | δFe, δFe, γFe, 2CrN, 2CrN                    |

4. Conclusion
1. The best formation of hard nitride layer is when plasma nitriding temperature of 550ºC for 10 hours with the nitrogen concentration obtained is 1.75% mass and the possibility of the phase formed is δFe, FeN, Cr2N, and VN.
2. Testing of Vickers nitriding plasma at a temperature of 500ºC for 10 hours has the highest hardness value compared to other nitriding specimens which is equal to VHN 955.29 kgf/mm² with a depth of 100 µm.
3. The optimum condition is when the hardness produced increases 1.79 times from the previous 531.32 kgf/mm² after hardening to 955.29 kgf /mm² after the nitriding process of plasma at a temperature of 500ºC.
4. Vickers hardness testing of SKD61 specimens after hardening increased 1.15 times from the previous 460 kgf/mm² to 531.32 kgf/mm².
5. The effect of Plasma Nitridation has been shown to improve the surface quality of SKD61 materials, especially surface hardness with optimum conditions achieved at plasma nitriding temperatures of 500 ºC for 10 hours.
6. Metallographic observation of SEM-EDS shows that the nitriding temperature affects the concentration and depth of diffusion of nitrogen in the SKD61 material.

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**Acknowledgement**

The authors contributed equally to this work. The authors also would like to thank Dr. Jan Setiawan for reviewing and proofreading the manuscript.