IOP Conference Series: Materials Science and Engineering

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Preface

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The iCOMERA conference showcases research result and industrial findings and provides a great opportunity for global industrial executives, practitioners, scientists, academicians, and scientists for sharing knowledge and ideas in all aspects of mechanical and industrial engineering. The iCOMERA also provides opportunity and room for focus group discussion among stakeholders to address the current and future global challenge, particularly in the industrial sector. Due to the COVID-19 pandemic situation all around the globe, we have organized this conference in virtual form for all Indonesian and international participants.

The Mechanical Engineering Department – Brawijaya University organizes the iCOMERA conference every other year, taking turn with the National Conference SAINTEK. Therefore, postponing iCOMERA to the next year would have destroyed the already fixed agenda of ME conferences. On the other hand, nobody really knows when this pandemic will finally be over and chances are that the pandemic will stay with us during 2021, too. We also had commitments already taken and signed, such us publication with IOP – Material and Science Engineering, UB Alumni and with some colleagues who wanted to joint this conference. For all these reasons the iCOMERA committee has decided to keep the original schedule, which was on 7-9 October and shifting to virtual format.

We have tried to keep the virtual conference as close as possible to the real one. Each speaker has 15 minutes to present his/her paper, with 10 minutes allocated for presentation and 5 minutes for Question and Answer. We have hired professionals to carry out the conference using Zoom Application to anticipate all unexpected problems which may occur and we were not aware of.

It was then quite a surprise that despite all those difficulties, 249 contributors submitted articles to the conference, which is even a higher number than what was registered for the previous iCOMERA edition in 2018. Out of the 249 papers, 32 were rejected, and 217 articles have been chosen for publication in the IoP Conference Series: Material Science Engineering. The high number of participants states to the fact that the iCOMERA conference is becoming popular in the engineering community. This is the result of the good service given in the previous iCOMERA 2018 by the organizing committee. All this hard work should be preserved to make the iCOMERA conference series sustainable and make it a fixed event in the engineers' international conference agenda.

Thank You,

Ir. Djarot B. Darmadi, MT., PhD.

Head of Mechanical Engineering Department.

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The role of nitrogen gas in fluidized bed reactors on the nodular iron nitridation processs

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The role of nitrogen gas in fluidized bed reactors on the nodular iron nitridation processs

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Abstract. Nitriding process using ammonia gas as a nitrogen source has been carried out with the aim of improving the surface quality of nodular cast iron until the highest surface hardness reaches 426.2 HV. In this study, the nitriding process was carried out with a variety of advanced processes using nitrogen gas in the fluidized bed reactor and without using nitrogen gas in the muffle furnace with a constant variable nitriding time of 4 hours. The continued process is 2 hours each, while the gas composition used is 80% ammonia and 20% nitrogen with a total gas flow of 0.7 m^2 $hour^{-1}$. From the result of SEM test analysis obtained in the advanced process variations with nitrogen gas in the fluidized bed reactor and without nitrogen gas in the muffle furnace, on the surface of nodular cast iron a nitride layer was formed in the form of Fe_3N and Fe_4N. The largest average thickness formed in advanced process specimens using nitrogen gas in the fluidized bed reactor was 24.64 µm and without using nitrogen gas was 6.06 µm. The EDX test analysis showed that the specimens that received treatment with nitrogen gas had a nitrogen gas content of 43.7% higher than the specimens that did not receive the treatment. The hardness test results showed the distribution of hardness values in specimens that received treatment with nitrogen and without nitrogen. The highest surface hardness is 560.6 HV on the surface of the specimen with nitrogen gas while the specimen without nitrogen gas has a hardness of 426.2 HV.

Keywords: nitriding, surface hardness, nitride layer, nitrogen gas role

1. Introduction

Surface hardening of materials is a process to improve the performance of components used in various fields of engineering and industry [1]. The surface of components and equipment used in the fields of petro-chemical, automotive and energy generation especially those operating at high temperatures need to be hardened to prevent changes in strength in the material. With this technique, the wear resistance of material is increased while the overall strength of the material remains high so that component life will increase.

Various studies on the diffusion of nitrogen atoms during the nitriding process have been carried out [2]. The previous study has conducted nitriding research on the hardness of nodular cast iron using urea nitriding media, with a temperature of 550 ^{0}C the thickness of the nitriding obtained from the results of the study showed a value of 2.1 μm with a resulting hardness value of 430 HV thin layer formed because nitrogen atoms were not can diffuse well on the surface [3]. Formation of the arising layer needs to be done by modifying the process in order to get the maximum thickness [4]. Therefore, the improvement of nitride layer formation in nodular cast iron needs to be reviewed. In this study, efforts to improve the formation of a nitride layer on nodular cast iron were carried out.

2. Methods and material

Chemical composition test is conducted to the specimen material in order to determine the content of its elements. The data on the chemical composition of the specimen material is matched with ASTM A536 standards to ensure that the material is nodular cast iron. Furthermore, the specimens received nitriding treatment using two types of furnaces, namely the fludized bed reactor and the muffle furnace. Nitriding was carried out at a temperature of 550 °C and a holding time of 4 hours. The treated material is cooled in the oil. Next, it is cut to obtain cross-sections (**Figure 1**) and then mounted with resin in preparation for the micro Vickers hardness test in order to obtain a distribution of hardness values in the surface layer of the specimen. Microstructure observations were also carried out through a scanning electron microscope (SEM) to determine the microstructure and thickness of the specimen layer. The sample used in this study is shown in Figure 1 with a diameter of 15 mm and a thickness of 4 mm.

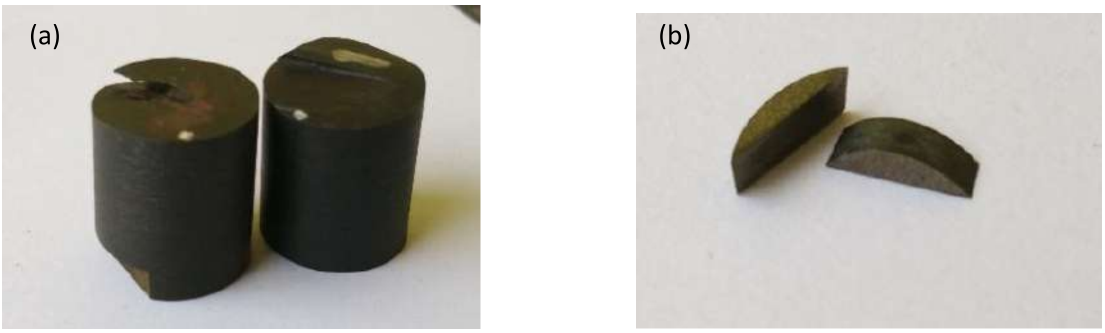

Figure 1. Sample condition: a) initial specimens b) specimens after cutting

Figure 2 shows that the heat treatment process carried out is gas nitriding with 2 different stages of the process on each sample, i.e.

- On sample 1; nitriding T = 550 ^{0}C; t1 = 4 hours; t2 = 2 hours with N_2 gas in the fludized bed reactor
- On sample 2; nitriding T = 550 ^{0}C; t1 = 4 hours; t3 = 2 hours without N_2 gas in the muffle furnace
- Oil cooling

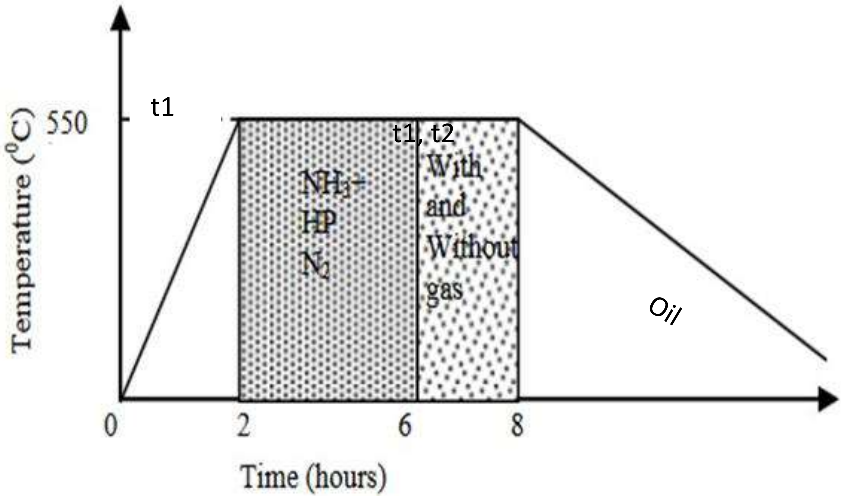

Figure 2. Experiment procedure

3. Results and discussion

3.1 Composition test

Table 1. Test results for nodular cast iron composition using OES Chemical

Element	Composition in Research	ASTM A536 Standard
Fe	89.60	
C	3.50	3.5 – 3.9
Si	2.55	2.25 – 3.00
Mn	0.409	0.15 – 0.35
P	0.0129	0.05 max
S	0.0165	0.025 max
Cr	0.0406	
Mo	< 0.0050	
Ni	0.0190	
Al	0.0080	
Co	< 0.0050	
Cu	0.0424	
Nb	< 0.0050	
Ti	0.0087	
V	< 0.0050	
W	< 0.0050	
Pb	0.0124	

The chemical composition of specimens is compared with the International standard (**Table 1**). It can be seen the similarity of the percentage values of elements C, Si, Mn, P, S so that the specimens are nodular type of ASTM A536. For the composition of the most dominant elements and allow the formation of nitride is the element Fe at 89.60%, which allows the formation of nitride compounds such as Fe_2N, Fe_3N, Fe_2N. From the content of the elements and the hard nature of the metal nitride formed it will determine the increase in hardness of the results of nitriding. To find out the level of hardness of nitriding results will be shown in the hardness analysis and SEM/EDX test below.

3.2 SEM analysis

Figure 3, Figure 4 and **Table 2** show different layer values indicate the influence of the two processes which impact on thickness. The thickness that occurs in process A1 tends to have a greater thickness value. The result of the average value of process A1 is four times greater compared with process B1. The further process in the presence of N_2 gas as an inert gas has a role to prevent oxidation on the surface of the specimen during the nitriding process. N_2 gas is not easy to react and is used as a protective gas due to the large N triple bond energy [5].

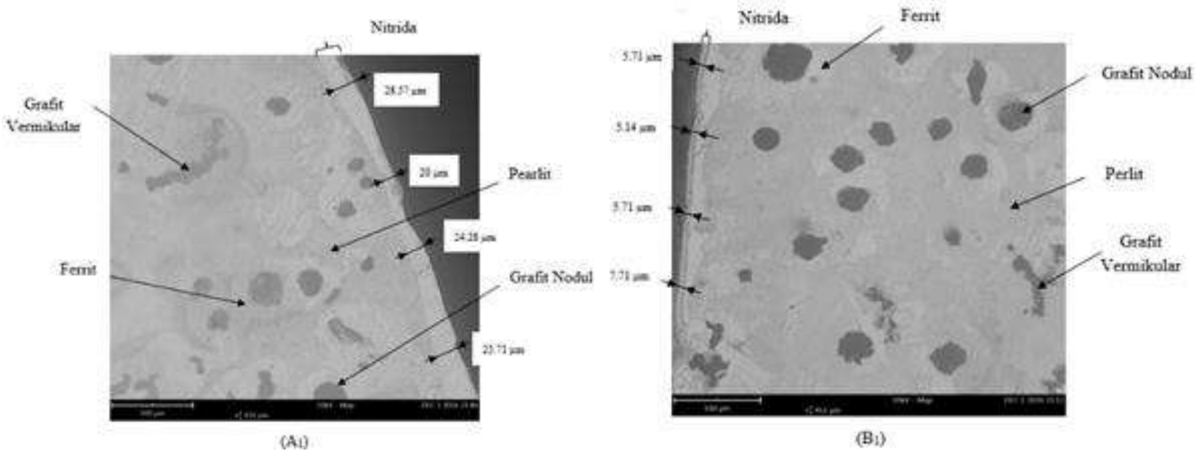

Figure 3. Photo of micro structure of advanced process specimens: (A1) Fluidized bed reactor with N_2 gas; (B1) Muffle furnace without N_2

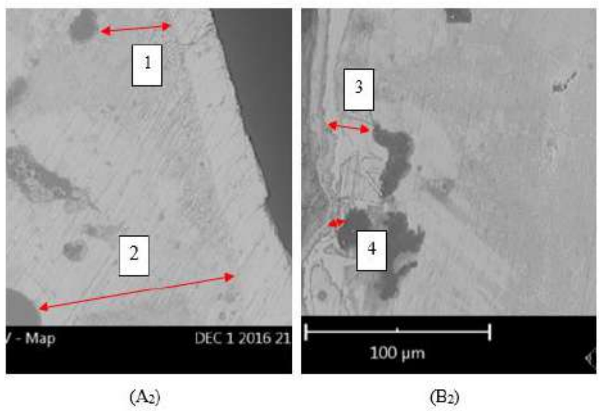

Figure 4. Micro photo specimen of advanced process: (A2) fluidized bed reactor with N_2, (B2) muffle furnace without N_2

At 550 ^oC the ammonia gas will dissociate so that it produces hydrogen and nitrogen atoms with the reaction of $3NH_3 \rightarrow 2H_2 + 3N$ [6]. From this dissociation the nitrogen atoms dissolve on the surface of the specimen. N atoms that have dissolved interstitially into the surface of the specimen will form nitrides as shown in the SEM photo above. A study of the surface of nodular cast iron with urea media with a temperature of 550 ^oC and time variations indicated that the the layer on the surface of nodular cast iron was only 2.1 μm. High effectiveness is demonstrated by gas nitriding for nodular cast iron.

Table 2. Nitride layer thickness

Process	Thickness of Layer (μm)				Average (μm)
Advanced process (A1) fludized bed reactor with N_2 gas	28.57	20	24.28	25.71	24.64
Advanced process (B1) muffle furnace without N_2 gas	5.71	5.14	5.71	7.71	6.06

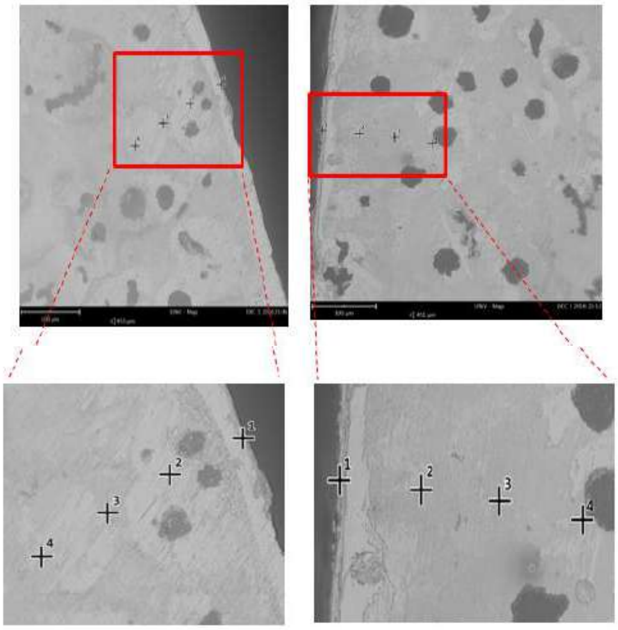

Figure 5. EDX result of advanced process specimens: (A1) Reactor fludized ded with N_2 Gas (B1) Furnace muffle without N_2

Table 3. Results of EDX test for advanced process specimens: (A1) Reactor fludized bed with N_2 gas; (B1) Furnace muffle without N_2

Process	Depth (μm)	Chemical Composition (%wt)				
		Fe	O	N	Si	Cr
Advanced process (A1) fludized bed reactor with N2 gas	0	72.12	12.54	10.50	4.30	0.11
	57	75.82	14.51	3.53	5.52	0.11
	114	80.08	10.83	3.99	4.41	0.21
	171	70.54	16.52	4.06	8.27	0.27
Advanced process (B1) muffle furnace without N2 gas	0	70.51	16.15	5.91	6.17	0.32
	57	70.39	18.87	3.04	7.07	0.12
	114	60.52	23.55	3.50	11.78	0.00
	171	62.83	27.82	3.50	8.85	0.59

The difference in thickness can be seen in **Figure 5**. The difference formed can be estimated due to the influence of the composition contained in the specimen that causes the homogeneity of the formed layers (**Table 3**). The previous study conducted experiments with diffusion media on AISI 4140 steel, P 20, 4340 homogeneity of the white layer formed was influenced by Cr content, AISI P20 steel had a high Cr composition and had better white coating uniformity [7].

Looking at the composition test data above the test specimens appear to have a relatively low Cr content of high C. This low Cr content causes the formation of white layers (nitride) which are not uniform. It can be seen the difference in thickness between the two elements. Possible factors causing the occurrence of this phenomenon can be due to element C; the layer adjacent to graphite tends to have a thinner layer (**Figure 6**). Nodular graphite is free carbon formed in nodular cast iron; the presence of high carbon elements in nodules causes the ability of N atoms to diffuse into Fe to be less because carbon binds iron and prevents diffusion of N atoms to the surface.

3.3 EDX Analysis

Figure 6 a to d which were the result of firing of both specimens shows the nitrogen diffusion layer compound which forms the nitride phase. (A1) The result of nitrogen diffusion forms a white layer with a thickness reaching 24.64 µm. This white colored film identified nitrogen levels twice as large as Figure (B2). (B2) White coating with a thickness of 6.06 µm, identified N levels of 5.91%. Referring to the Fe-N diagram the layer at point 1 (A1) of the sample treated with N_2 gas in the fluidized bed reactor shows the formation of the hard epsilon (ε) Fe_3N phase, at points 2, 3.4 with levels of 3.53%, 3.99, 4.06 tends to form α + γ (Fe + Fe_4N) phases. Whereas the sample without N_2 gas is in the muffle reactor, the layer at point 1 which contains 5.91% N forms the Fe_4N phase, and points 2,3 and 4 at 0.1-5.7% sensitivity tend to form α + γ (Fe + Fe_4N).

Figure 6 shows an EDX graph that produces several new elements due to the thermochemical treatment process. The formation of the new element is the result of the diffusion process of the element nitrogen which penetrates through the surface of the specimen. **Table 3** shows the composition of elements on the surface of the white layer identified to contain nitrogen (N) element N is relatively high reaching 10.5%. In Figure 6d, the comparison of depth and nitrogen content in both specimens shows that the nitrogen value in both samples shows a decreasing value, with the difference of nitrogen in both samples at point 1 reaches 43.7%. Nitrogen (N) found on the surface of specimens treated with N_2 gas tends to have a greater value of N atomic concentrations when compared to specimens without N_2 gas treatment.

This condition can occur because of the role of N_2 gas in the nitriding process. N_2 is an inert gas that serves as a protective gas to prevent the entry of gas from the atmosphere. But in certain conditions as in Widi *et al.* [7], it shows the effectiveness and efficiency of the gas nitriding diffusion process can be achieved without using the role of gas in the muffle reactor [8]. However, the specimen must have a hard chorm coating. His research also explained the phenomena that were dealt with in the process with the muffle reactor showing porosity which is useful for applications that require the ability of soaking and lubrication.

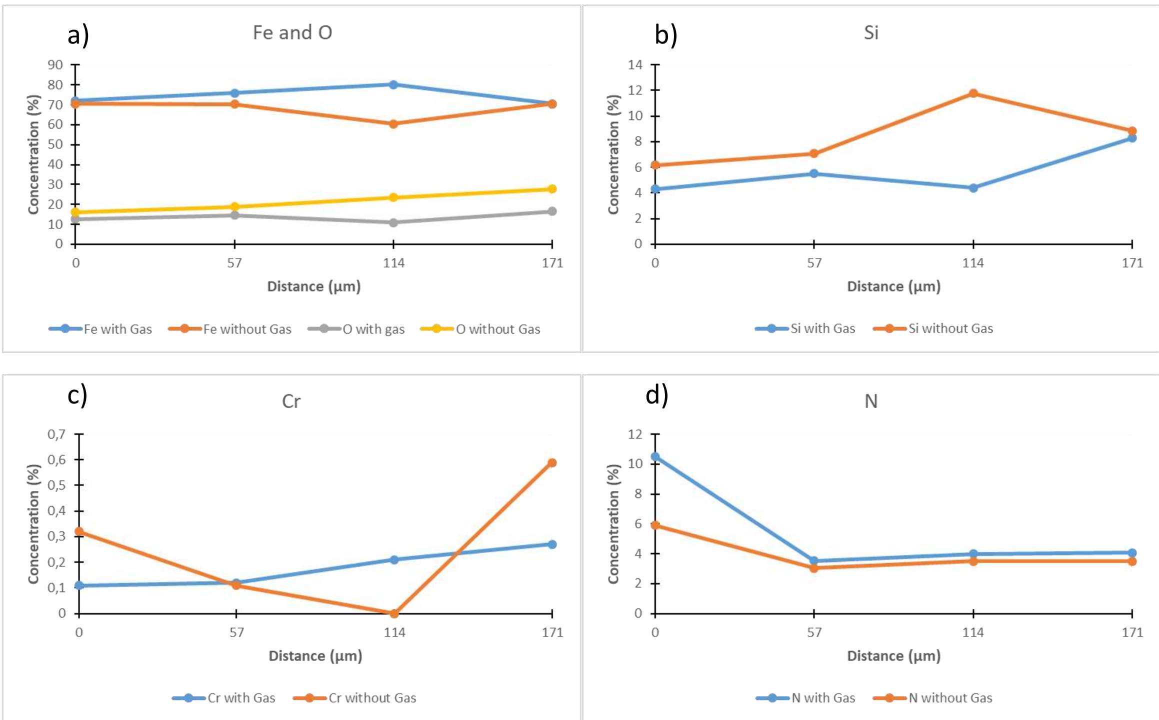

Figure 6. The concentration of elements formed based on the depth of hardening in the furnace muffle specimen without N2 a) concentration of Fe and O, b) concentration of Si, c) concentration of Cr, d) concentration of N

In the sample transferred to the muffle furnace the amount of diffused nitrogen tends to be lower with a percentage of 5.91% (**Table 3**). This can be caused by the presence of atmospheric gas that reacts with N atoms that have entered the specimen surface, the condition occurs because during the process in muffle furnace temperature 550 ^{0}C atomic vibrations still occur in the specimen, without the presence of protective gases, the atmospheric gas that is in the muffle furnace like oxygen will react with N atoms which can cause new gases and wasted into the atmosphere, causing the concentration of N atoms on the surface decreases. Comparison of the magnitude of N atoms diffused on a surface with a certain depth can be seen in Figure 6d.

3.4 Hardness test analysis

Table 4 presents the results of micro hardness case depth hardness testing using the Vickers method of the sample after nitriding with an advanced process using N_2 gas (fludized bed) and without using N_2 gas (muffle). From the data above, it can be seen that there are differences in surface hardness in the two samples. The graph shows that the level of surface hardness in the layer at a distance of 30 µm, samples treated further using N_2 gas on fludized beds have a hardness value of 560.6 HV, 391.3 HV, 375.9 HV higher than samples without treatment using N_2 gas in muffle furnaces which has a hardness value of 426.2 HV, 237.6 HV, 155.6 HV.

The hardness of the layers indicates that on the surface the outermost layer appears higher, the further from the surface the lower the thickness. This is possible because the surface layer is formed by the process of further nitriding thermochemical treatment with N2 gas in the fludized bed and muffle furnaces to produce different nitride phase compounds. Different nitride phase compounds will have a major effect on the contribution of hardness. In other words, that each nitride phase has a hardness nature [9].

Table 4. Hardening depth testing data

Depth (µm)	Process	X	Y	Hardness (HV)
30	Without	44.3	48.98	426.2
80	Nitogen	61.62	63.33	237.6
130	(N_2)	82.9	71.48	155.6
30	With	40.45	40.88	560.6
80	Nitogen	49.75	47.38	393.1
130	(N_2)	49.22	50.1	375.9

The phase compound ε (Fe_3N) formed in the outermost layer has relatively high hardness properties reaching 560.6 HV for samples with advanced processes N2 gas decreases the hardness up to 24% in samples without further processing of N2 gas. Hardness decreases with increasing distance from the surface as nitrogen levels decrease. The decrease in the nature of the turbidity and the decrease in nitrogen levels also show that the layers in the area undergo changes in phase compounds. The hardness nature of phase γ (Fe4N) with a level of 5.91% nitrogen is relatively lower than that of the phase ε. Likewise, the hardness of α + γ (Fe + Fe_4N) phase with a level of 0.1-5.7% nitrogen tends to approach the hardness of the basic material.

4. Conclusion

From the results of the research, it can be concluded that the use of nitrogen gas media in the advanced nitriding process can increase the thickness of the nitride layer formed on the surface of the specimen because N_2 gas acts as a protective gas when diffusion of N atoms to the specimen surface. the value of the thickness of the advanced nitriding with N2 gas reaches four times that of the advanced process without N_2. Nitride layer formed on specimens with advanced processes with N2 gas reaches a thickness of 24.64 µm, while the advanced process without N2 gas reaches 6.06 µm. The hardness formed on the surface of specimens in specimens with advanced processing with N2 gas of 560.6 HV is greater than that of advanced processing specimens without N2 which reaches 426.2 HV hardness

5. Future Research

Other nitriding researches need to be conducted for other types of cast iron. In addition, other researches on the influence of graphite types on cast iron on the formation of nitride layers on the surface of cast iron are needed.

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