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October 4th-6th, 2015

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"Beyond Materials Innovations and Tecnologies"

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Materials and Metallurgical Engineering

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"Beyond Materials Innovations and Tecnologies"

Materials and Metallurgical Engineering

Institut Teknologi Sepuluh Nopember (ITS) Surabaya Indonesia

October 5th, 2015

CONFERENCE PROGRAM ICOMMET 2015				
07.00 - 07.30	Registration			
07.30 - 08.30	Opening Ceremony			
	Foreword by the Chairman ICOMMET 2015			
	Sigit Tri Wicaksono, S.Si, M.Si, PhD			
	Foreword by the Rector of ITS			
	Prof. Jhony Hermana			
08.30 - 09.30	**KEYNOTE SPEAKER 1** Professor Shih-Hsuan Chiu Materials science and Engineering Department, National Taiwan University of Science and Technology (NTUST), Taiwan (Moderator: Dr. Agung Purniawan)			
09.30 - 10.00	COFFEE BREAK			
10.00 - 11.00	**KEYNOTE SPEAKER 2** Professor Paddy French EI-EWI, TU Delft, Mekelweg4, 2628CD Delft, The Netherlands			
11.00 - 12.00	**KEYNOTE SPEAKER 3** Prof. Bambang Kuswandi, P.hD Universitas Negeri Jember			
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CODE : MM03/06

Structure And Properties Of Stainless Steel Nitride Layers Produced By Fluidised Bed And Muffle Reactors by Diffuse Step Nitriding Processes

Komang Astana Widi[1], ING Wardana[2], Yudy Surya Irawan[2], Wahyono Suprapto[2]

[1] Department of Mechanical Engineering, National Institute of Technology of Malang , Malang 65145, Indonesia
[2] Department of Mechanical Engineering, Brawijaya University, Malang 65145, Indonesia

Abstract: In manufacturing industry, high purity nitrogen gas as diffuse media nitriding treatment is highly consumptive. In this paper, diffuse media technology by muffle and fluidized bed reviewed thoroughly. Austenitic stainless steel samples were submitted to nitridation in varying conditions of atmosphere diffuse reactors. The chemical composition after those treatments were investigated by EDAX analysis. The characteristic of microstructure were studied by optical microscope, SEM and XRD. The mechanical properties were investigated by microhardness test. The effects of both reactors diffuse treatment were had analyzed. As results, samples with nitride layer by fluidized bed are more dense than that of muffle samples, and had a high hardness. Muffle diffuse nitriding treatment outer layer samples showed lower hardness but the inner layer almost similar to those of fluidized bed diffuse nitriding treatment. It was concluded that the lower of hardness is ascribe to the porosity formation as affected by nitrogen, hydrogen and chrome oxide reactions.

Keywords : stainless steel; fluidized bed; muffle; microstructure; microhardness; diffuse

Corresponding author : Komang Astana Widi, E-mail : aswidi@yahoo.com, Telp. +62-341-417636 Ext.518, Fax +62-341-417634

1. Introduction

The advantage of nitriding surface treatment is a very low distortion level, even ofentimes distorseless. The component that is given nitriding treatment will have better wear resistance, corrosion resistance and fatique resistance if it is applied to alloy steel and iron using combination composition, especially low chrome element. This combination element will help the nitriding process efficiency and effectivity in improving hard case depth. However, nitriding process is less efficient and effective compared to other thermochemical treatments, in which, based on standard time, gas nitriding treatment needs until 72 hours [1].

Some component failure can caused by nitriding process still oftentimes happens that is caused by the limitation factor in case depth and the non-homogeneity of nitrogen atomic difusion layer into substrat. The problem in case depth and diffusion of nitrogen atomic in steel gives an effect in the productsusingrestrictiveness. This thin layer forming will decrease the toughness value because it will be easy to crack in case of impact loading, therefore, the materials that is processed by nitriding isgenerally only capable to adhesive loads, that is friction without pressing load.[2]

Investigated shows that the nitride layer thickness depends on nitriding treatmentcondition, material characteristic, reaction and diffusion that happen on the material surface, fusion element and physical defect solidity on the material surface [3].The utilization of chrome based material in this study is becauseit has a good forming of oxide layer and nitride. Besides, this high chrome based materials is rarely used as a material for engineering application, especially at construction and structure. It is because chrome's characteristic is brittle with less ductility, whether in a condition or high temperature [4,5,6] .

The presence of oxygen in high temperature will be more reactive with lower energy activity in producing oxide layer Cr_2O_3[7]. The passive layerforming can be used as protector from outer atmosphere, in that the material's activity and usage will be better, especially the steel material that uses high fusion element. This passive layer forming is very correlated to alloy element, atmosphere process, temperature, and process timing. Not only passive layer that has direct correlation in diffusion mechanism process, surface preparation also has a role in nitrogen atomic diffusion process into substrat. Therefore, it can be concluded that there are other factors that have role in producing homogenity and case depth of nitrogen atomicdiffusion in steel that still have no deep observation that will be conducted in this research.

The non-homogeneity difusion diversity phenomenon will lead to residual stress as a result of gradient forming in a form of thermal stress, thermal expansion coefficient, density, chemical composition, and surface tension [8]. Investigated that residualstress that is formed in the process of nitriding surface hardness is a result of interaction between compound layer and substrat, that is in a form of nitrogen composition gradient, phase changing, and volume in Yand ε phases [9].

Some mechanism understandings of nitrogen atomic diffusion that have been observed are supersaturated reaction and the forming of grain growth and physical defect mechanisms as a result of N atomic diffusion [10,11]. In addition, the previous research the hardening mechanism conductedby nitrogen atomicinto steel is an impact of nitrogen excess forming at grain boundaries. The whole control of diffusion mechanism theories is influenced by some process variables, such as alloy, atmosphere media gas, temperature and time nitriding process [12].This research will study about the effect of media during nitriding diffusion process, in that the phenomenon occurred during this diffusion step can be understood more clearly. The using of muffle reactor using atmosphere atmosphere media in nitriding diffusion process is never observerd by previous researchers, and to study about nitriding diffusion phenomenon, it is very important to understand the passive layer role as a product of nitriding atmosphere role. This research also compares the using of diffusion process using fluidized bed reactor with pure nitrogen gas media.

2. Experimental

Austenitic stainless steel is used as samples then investigated at cross section after diffusion nitriding process to find out the structure, case depth and chemical composition changes each layers form. Nitriding process consists of two steps processing, those are: boost process,which is obtained in fluidized bed gas reactor (the process of inserting atomic N from gas NH_3 dissociation), and the process that will vary atmosphere of diffusion process (the process of atomic N spreading in substrate). Diffusion process will be the focus on this research in producing optimum, efficient, and effective nitride layer spreading. Boost process uses temperature550^{0}C during 4 hours process in fluidized bed reactor using ammonia gas and pure nitrogen (high purity with the level of 99.98%) with the comparation of gas composition flowed into fluidized bed 80 NH_3: 20 N_2 and the flow of total gas is 0.7 m^3/hour. Whereas, the next process is observation at diffusion process that needs processing time (2 hours), temperature process550^0 C, gas media with pure nitrogen (in fluidized bed) and atmosphere atmosphere (in muffle). That flow process diagram can be shown at Fig. 1.

The observation of microstructure and phase formed on the surface layer and the area under surface layer are conducted at cross section samples. The test will give information about the thickness of oxide layer and nitride that is formed at samples that has already gotten nitriding treatment. The way another observer did to support the result of micro structure and phase is by using case depth distribution test as a purpose to get information about homogeneity of nitrogen atomic diffusion spreading into substrat.Other expected information is about porosity (gas that is caught). Another characteristic that will be used, such as hard case depth (microhardness vickers), and metallography (electron microscope, XRD, and SEM/ EDAX).

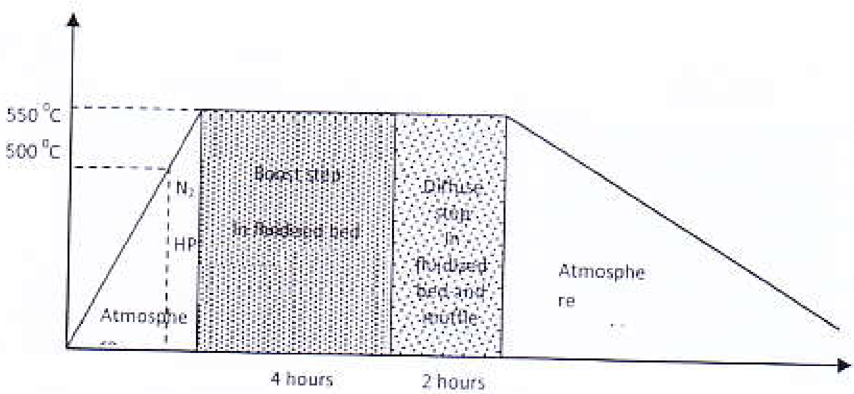


Fig. 1. Boost and diffuse step of nitriding process diagram

3. Results and Discussions

According to the result of microstructure observation (scanning electron microscope, microscope optic, and EDAX), Figs2 and 3 show the different characteristic on nitride compound layer that is formed at specimen surface where atmosphere diffusion media (muffle reactor) shows the layer that is less solid with more porous surface morphology (showed by thicker nitride layer) compared to the treatment using high purity nitrogen diffusion media (fluidized bed reactor). It causes the decreasing of hardness value, especially at the top layer as showed at the result of hardness case depth test (table 1 and figure 4a). Porosity phenomenon at the top surface is an effect from too much atomic N concentration where it is exceeded Fe and Cr substances alloy ability in binding atomic N in that there is a reaction of supersaturated. It is showed by the decreasing of hardness value compared to the using of nitrogen diffusion media. Nevertheless, Fig. 4d showed that nitrogen atomic concentration at the top surface layer is almost the same for both diffusion media variables. It shows that not only supersaturated reaction that causes porosity ($N+N \rightarrow N_2$), atomic N bond with Fe substance gives lower hardness value effect compared to atomicN bond with chrome substance. This mechanism is showed by EDAX analysis where atmosphere diffusion media variable has higher Fe concentrationand lower Cr concentration compared to variable of nitrogen diffusion media (Fig.4b and 4c). It shows that there is correlation between hardness value to the chrome concentration where the ability of Cr bond with N in making better hard nitride layer compared to Fe with N bonding.

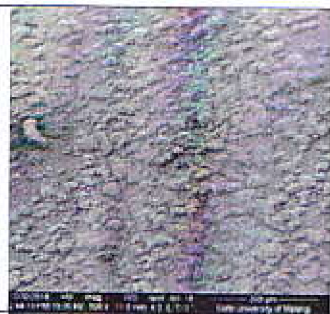
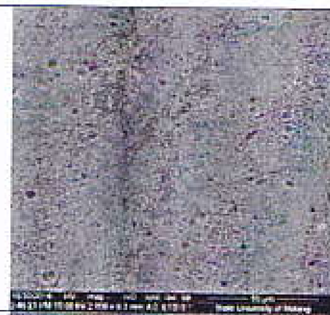
Reactor Difuse	Fluidised Bed	Muffle
Surface structure		
Cross section structure		

Fig. 2. SEM structure nitride layer on stainless steel (20.000x).

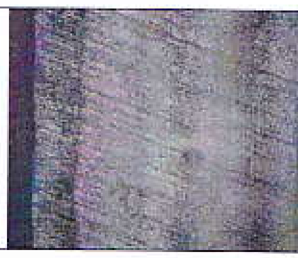
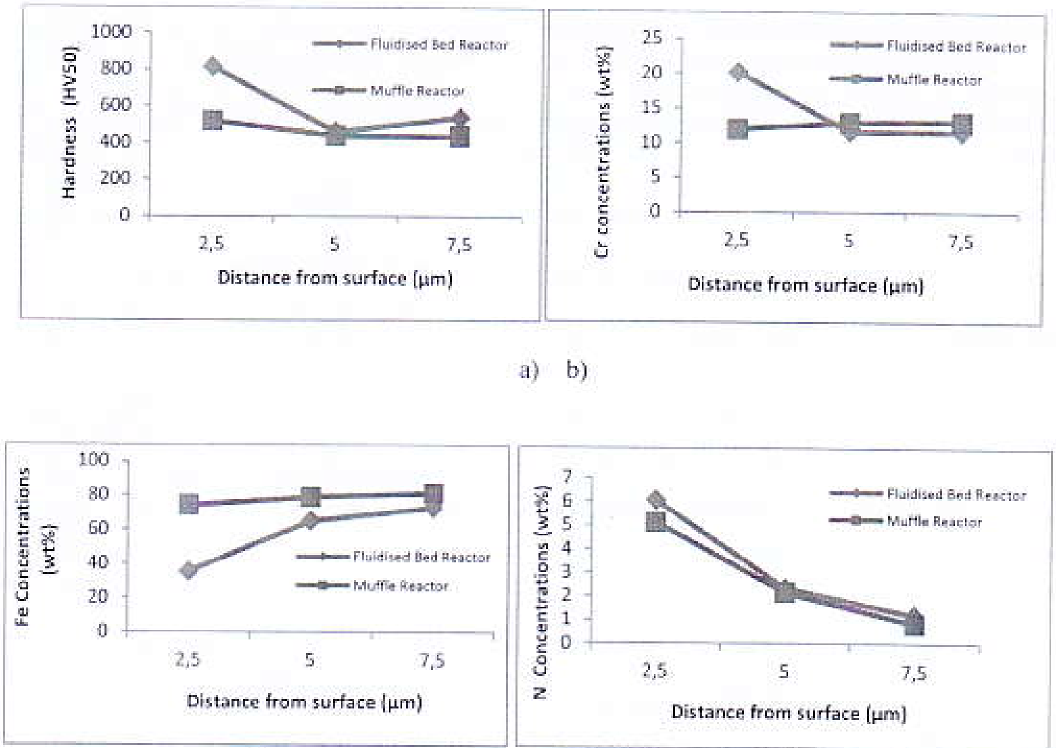
Diffuse Reactor	Fluidised Bed	Muffle
Cross section microstructure		

Fig. 3. Microscope optic microstructure on cross section stainless steel (250x).

Table 1. Case depth hardness average of nitride layers

Reactor Media Diffusion	Case Depth				Average
Fluidised bed Reactor	2.5	832,5	750	841	841
	5	461	470	440	458
	7.5	531	565	525	538
Muffle Reactor	2.5	517	525	505	517
	5	455	425	434	437
	7.5	425	445	439	437



a) b)

c) d)

Fig. 4. Characteristic nitriding from surface to the depth a) case depth b) Cr concentrations, b) Fe concentrations c) Nitrogen atomicic concentrations

The number of atomic N concentration that is compounded with Fe and porosity forming on compound layer show the solubility of atomic N into lower Fe compared to chrome.The beneath layers of white layer until diffusion layer of all specimen variables shows relevancy. Therefore, from all testing data, it can be concluded that the low hardness value at the surface is not only influenced by nitriding diffusion media where the role of nitrogen diffusion media will be more effective in binding nitriding nitrogen atomic in order to improve the characteristic of specimen surface hardness. On the contrary, the using of atmosphere diffusion media in muffle reactor is less effective in binding nitrogen atomic as a result of the decreasing of chrome concentration value at the top layer.

The result of microstructure analysis at cross section, especially at atmosphere diffusion media (figure 4) shows that there are three forms layers on the surface, those are: white layer, nitride layer, and diffusion layer.The analysis of case depth in this research will be connected with the influence of concentration of alloy, nitrogen and oxygen diffusions. Looking at the correlation of that result, the forming mechanism of case depth can be known exactly.

So, it can be explained that nitrogen atomic diffusion mechanism into specimen during the diffusion process shows oxygen role in open the oxide layer grain limit to ease the entry of nitrogen atomic diffusion into substrat. It is showed by a highly concentration of nitrogen atomicat the nitrogen diffusion media. The lack of formed oxide layer resulted in oxygen role does not produce oxide layer evaporation and even increase the forming of oxide layer because the limitation of oxide layer thickness has not reached yet. Based on the result of EDAX test, it shows that maximum atomicnitrogen at this research is 6.04 wt%. It means that this sample will have gamma (Υ)or FeN (less than 6 wt% based on Fe-N phase diagram). It is in line with the XRD analysis (Table 2 and figure 6) and white layer formed is still very thin, about 0.5 to 1 µm of thickness. It shows that white layer forming has already occured at the concentration of 6 wt% at atmosphere diffusion media. The using of atmosphere as diffuson media will accelerate the forming of white layer at stainless steel sample. Υdan εprocentages are also influenced by carbon content where the higher carbon content will be able to promote ε phase layer, and the lower carbon content will promote the ε phase layer producing of Υ.

Based on the alloys, oxide layer and phase that exist at samples show that nitriding diffusion reaction has already formed. The forming reaction of nitride and oxide is showed by the presence ofCr_2O_3, Fe_2O_3, and $FeN_{0.0897}$ at the EDAX and XRD analysis. Whereas, chrome nitride can not detected on this test. The observation shows that oxide has already occured at the temperature of 500^0 C at the nitrogen diffusion media specimen that will give an effect to the number of chrome ion extrication to the atmosphere that is showed by the high element of Cr and chrome oxide (Cr_2O_3) at the material surface compared to the the layer beneath.In the contrary, at specimen that uses atmosphere diffusion media where chrome and oxide chrome elements are lower on the surface part compared to the composition in the inside. It shows that chrome ion experiences extrication phenomenon to the atmosphere in time with the lower chrome oxide layer. This mechanism occurs as a result of oxygen role that helps chrom ion extrication.

The mechanism of chrome extrication reaction from chrome oxide that is caused by the presence of hydrogen element from atmosphere diffusion media and the presence of iron and steel elements can be showed with the chemical reaction as follows:

$Cr_2O_7^{2-} + 14H^+ + 6e^- \rightarrow 2Cr^{3+} + 7H_2O$ .... (Eq. 1)

$Fe^{2+} \rightarrow Fe^{3+} + e$ .... (Eq. 2)

$Cr_2O_7^{2-} + 14H^+ + 6Fe^{2+} \rightarrow 2Cr^{3+} + 7H_2O + 6Fe^{3+}$ .... (Eq. 3)

The surface chrome oxide at the nitrogen diffusion media is higher compared to the using of atmosphere diffusion media.It is in the contrary of nitrogen atomic concentration, and oxygen at the surface is more excessively at atmosphere diffusion media. This nitrogen atomic concentration increasing is together with the iron nitride concentration increasing (Fig.5).

Table 2. XRD test of compound layers phase formedafter diffuse nitriding process.

	Fluidized bed Reactor		Muffle Reactor	
Compound	Fe-Cr	$FeN_{0,056}$	Fe-Cr	$FeN_{0,088}$
Cons. (%)	98	2	96	4

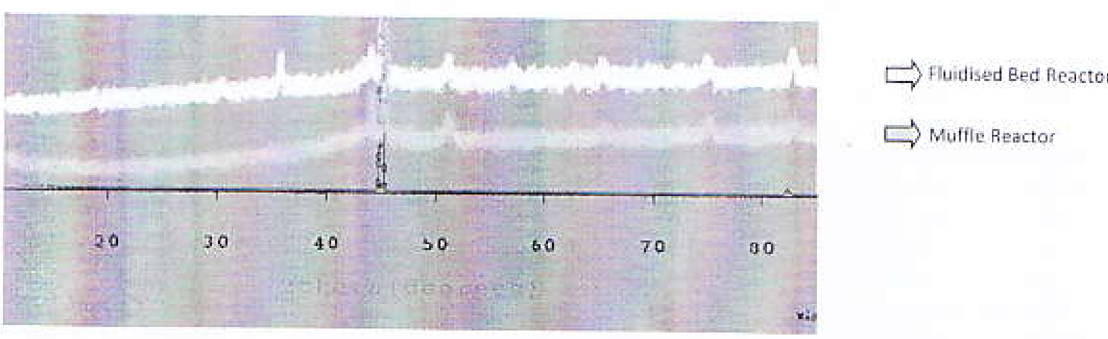


Fig. 5. XRD peaks test on stainless steel after diffuse nitriding process.

There are two step mechanisms in inserting nitrogen atomic into specimen surface in this research, those are: boost step (inserting nitrogen atomic), and diffusion step (nitrogen atomic spreading). At diffusion step, the using of pure nitrogen gas will decrease surface passive layer because there is evaporation reaction of chrome oxide layer, but the evaporation influence of $Cr_2O_3$is not significant at the thick layer (the using of atmospheremedia), evaporation rate is comparable with the diffusion growth rate.

4. Conclusions

Nitrogen diffusion media (fluidized bed reactor) variable process that shows surface chrome oxide concentration at 20.17 wt% will increase nitrogen atomic diffusion concentration from 6.04 wt% at the surface to 2.34 wt% at the under the surface (at nitrogen media), whereas and at surface chrome oxide atmospheremedia at 11.91% will increase Nitrogen atomic diffusion concentration from 5.11 wt% at the surface to 2.15 wt% at the under the surface.Atmosphere diffusion media (muffle reactor) shows that nitrogen atomicic concentration at the surface is less than 1.2 times compared to nitrogen diffusion media, but nitride phase formed is 2 times in a form of iron nitrided, therefore it can be concluded that the rest of N atomicic at the nitrogen diffusion atmosphere does not form nitride phase, but it forms nitrogen excess. It is influenced by the presence of Fe concentration and chrome.

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