CERTIFICATION OF APPROVAL

Empirical Modeling for Diffusion of Nitrogen into AISI 430 Ferritic Stainless Steel

by

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CERTIFICATION OF ORIGINALITY

This is to certify that I am responsible for the work submitted in this project, that the original work is my own except as specified in the references and acknowledgements, and that the original work contained herein have not been undertaken or done by unspecified sources or persons.

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ABSTRACT

This study is based on the solution nitriding of ANSI 430 ferritic stainless steel. Solution nitriding play a significant role in enhancing the surface hardness of AISI 430 ferritic stainless steels. However, this particular area need further research in term of empirical modeling for future engineering guidance and references.

The study of this project focused on the identification of the appropriate empirical model for diffusion of nitrogen into AISI 430 ferritic stainless steel. And then, the results were compared with the existing experimental results which were conducted on the same temperature. This research starts by searching for any other relevant journal or article published by other researchers and from there, the results were reviewed and useful information were summarized and taken as information to this research. This study involved few analytical calculations, more to theories and literature reviews, research of pre-existing experimental works and results and finally comparison between theoretical results and existing experimental results. As for software, the familiarization of Microsoft Excel is more than enough to solve the developed formulations.

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CHAPTER 1: INTRODUCTION

1.1 Background of study

Nitriding is a highly specialized surface hardening treatment that produces a thin but high hardness case on a wide variety of steels. Nitriding of iron and steel is known to improve hardness, wear and corrosion resistance of surfaces [1]. An additional advantage of nitriding is that the surface hardness is resistant to softening by temperatures up to the process temperature. The significant advantage of nitriding over other surface hardening processes is that the case hardness is developed without quenching and the attendant distortion problems. The nitriding process involves the diffusion of nitrogen into the base steel. This diffusion takes place at relatively low temperatures (typical process temperature is 975 F) and the hardening occurs without quenching. Core properties are not affected by the nitriding process provided the final tempering temperature for the product was higher than the nitriding process temperature. Regardless of the steel used for nitriding, one of the two methods is recommended for heat treating:

- Method 1. For minimal distortion.
  - Quench and temper stock to specified core hardness
  - Rough machine
  - Stress relieve
  - Finish machine
  - Nitride
  - Lap or lightly grind as necessary

- Method 2. For maximum machinability.
  - Rough machine
  - Quench and temper to specified core hardness
  - Finish machine
  - Nitride
  - Lap or lightly grind as necessary

Ferritic stainless steels are plain chromium steels with no significant nickel content; the lack of nickel results in lower corrosion resistance than the austenitic (chromium-nickel stainless steels). Ferritic are best suited for general and high-temperature corrosion applications rather than services requiring high strength. They are used in automotive trim and exhaust systems, interior architectural trim, and hot water tanks. Two of the most common grades are type 430 (general-purpose grade for many applications, including decorative ones) and type 409 (low-cost grade well suited to withstanding high temperatures). In this project, nitriding will take place into ANSI 430 ferritic stainless steel. 430 ferritic stainless steel is the most widely used ferritic (plain chromium stainless category) stainless steel, offering general-purpose corrosion resistance, often in decorative applications.

However, diffusion nitrogen into steel at liquid state is very limited, even at high pressure. Due to this difficulty, solid diffusion of nitrogen into steel by using high-temperature gas nitriding was frequently employed. Traditional methods of nitriding, such as pack nitriding, gas nitriding, and salt-bath nitriding have been well documented.

Traditional gas nitriding, which has been favored by metallurgists and engineers for many years, now is being challenged by more recent techniques such as controlled nitriding, ion nitriding, enhanced ion nitriding and RF nitriding. Controlled nitriding and ion nitriding have gained acceptance and now are commercial techniques. Another development for use with the pulsed plasma nitriding system is surface deposition after the nitriding treatment. This allows nitriding to be carried out in the ion nitriding chamber followed by a surface deposition treatment, such as depositing titanium nitride.

The behavior and properties of nitrogen on steels has been studied extensively for many years, owing to its relevance in catalysis processes, corrosion, etc. Stainless steels have an excellent corrosion resistance because of their native passive layer. However, their load-bearing capacity is not very high. To increase the surface hardness, conventional nitriding in gas and plasma [2], was initially carried out leading to a drop in corrosion resistance due to the formation of CrN precipitates at temperatures above 400°C [3].

Solid solution nitrogen enhances the mechanical and corrosion properties [4]. Solution nitriding is a relatively new technique to strengthen stainless steel. In this treatment, the material is heated at high temperature (1100 – 1200ºC) under nitrogenous atmosphere. Diffusion of nitrogen into stainless steel changes the microstructure into austenite or martensite. Consequently this increases hardness of the surface layer [4]. This technique is widely applied to harden austenitic [5], ferritic [4], duplex [6] and martensitic [7] stainless steels.

The dissolution of nitrogen in ferrite as well as the formation of compact nitride layers on the surface of steel by means of thermo-chemical nitriding treatments produces an important enhancement of the mechanical and tribological properties of the treated samples. Measurement of the nitrogen potential as well as a mathematical simulation of the growth of the compact nitride layers allows us to optimize and automate the nitriding processes. It has been observed that high nitrogen content in stainless steel improves its mechanical properties. Nitrogen content increases the hardness, impact strength and scratch resistance of stainless steel. It is also very effective for improving yield strength and corrosion resistance without reducing ductility and toughness as well as compression strength.

In this project nitriding process will take place into AISI 430 ferritic Stainless Steel. This is the basic and most generally used straight chromium ferritic stainless steel. Because of its high chromium content, it is highly corrosion and heat resistant but somewhat less than Types 301, 302 and 304.

1.2 Problem statement

Solution nitriding play a significant role in enhancing the surface hardness of AISI 430 ferritic stainless steels. This particular area need further research in term of empirical modeling for future engineering guidance and references.

1.2.1 Problem Identification

The works on solution nitriding of AISI 430 ferritic stainless steel have identified the phase change, the hardness of the solution nitrided ferritic stainless steel as well as the hardness profile of the steel due to the nitrogen diffusion. However, not many works investigated the empirical modeling for that particular area yet.

1.2.2 Significant of project

The aim of this research is to study and develop empirical modeling for diffusion of nitrogen into AISI 430 ferritic stainless steel.

1.3 Objective

The main objective or outcome from this project is to develop empirical modeling for diffusion of nitrogen into AISI 430 ferritic stainless steel. To fully achieved that, there are several tasks needed to be done when completing this project

1. Identify and study on the hardness profile of the material
2. Study the existing formulation of Case Hardened Depth, diffusion coefficient of nitrogen in ferrite and surface nitrogen concentration
3. Select and develop the appropriate formulation to get better results by varying nitriding time
4. Compare the theoretical results with the existing experimental results for same temperature.

1.4 Scope of Study

The study of this project will be focused on the identification of the appropriate empirical modeling for diffusion of nitrogen into AISI 430 ferritic stainless steel. This study will involve few analytical calculations, more to theories and literature reviews, research of pre-existing experimental works and results and finally comparison between theoretical results and existing experimental results. At the end of these studies

- Reading papers and journals that assist the study.
- Familiarization of the experiment methods and results
- Familiarization of terms and theory related to the study.

1.4.1 Relevancy of the Study

This project will focus on the topic of nitriding, composition and phase changes in materials. These topics are related to the courses of Introduction to Materials Science and Engineering, and Engineering Materials that I took earlier study of my mechanical engineering program. So, the knowledge of those particular courses is needed to perform research for this project. From general perspective, this topic is fully related to the mechanical engineering student which covers critical area in developing of stainless steel in industries. In addition, this project is really a challenge since have to discover about the empirical modeling to be used in order to reduce the costs and time. And also, by having done that, it will help and serve as guidance or references for future engineers.

1.4.2 Feasibility of the project within the scope and time frame

The first step in this project will be getting an introduction to the related topics by reading books, journals and research papers. Research will be done in order to understand better on the nitriding into AISI 430 ferritic stainless steel. The project is feasible to be completed within the scope of study and time frame.

CHAPTER 2: LITERATURE REVIEW

Nitriding divided into two categories, which are at lower temperature (500^oC – 700^oC) and high temperature (above 1000^oC). At this high temperature it is called high-temperature gas nitriding [7, 8] or solution nitriding treatment [8]. Recently, high-temperature gas nitriding (HTGN) or solution nitriding was introduced as a method for adding nitrogen to stainless steels. This new nitrogen addition method involves a diffusion process for nitrogen to permeate the surface of stainless steel through heat treatment in N_2 atmosphere at high temperatures [9]. When nitrogen (a strong austenite forming element) permeates from the surface into the interior of stainless steel, the surface microstructure changes into austenite or martensite depending on the amount of nitrogen permeated and the process temperature. As a result, the corrosion resistance and hardness of the surface layers increase. In general, HTGN has usually been applied to Cr based austenitic, martensitic and duplex stainless steels, which have high nitrogen solubility in the austenite phase. However, the low nitrogen solubility of a ferrite single phase prevents HTGN of ferritic stainless steel. Ferritic stainless steels were adopted to use in the manufacture of engine mufflers, nuts, bolts and heat resistant tools. In some cases, such steels require high surface hardness. The ferritic stainless steel is an attractive alternative to the most often adopted austenitic stainless steel due to its higher strength, better ductility and superior corrosion resistance in caustic and chloride environments and is suitable for application in process /petrochemical, oil & gas, nuclear and power industries [10].

A typical by-product of nitriding is the white layer, a thin layer of extremely hard iron nitride. This layer may or may not be objectionable, but in most cases it must be kept thin. The “white layer” or compound zone is a very hard, brittle layer that does not diffuse into the steel but remains on the immediate surface [11]. The process variables that control the depth (and make up) of the white layer are time, temperature and gas composition. It typically comprises two intermixed phases, gamma prime (γ’) and epsilon (ε). The Iron-Nitrogen phase diagram (Fig. 1) provides a “road map” to help determine the type of structures that will be produced in the nitriding process [11]. This figure tells us what happens when nitrogen diffuses into the surface of pure iron.

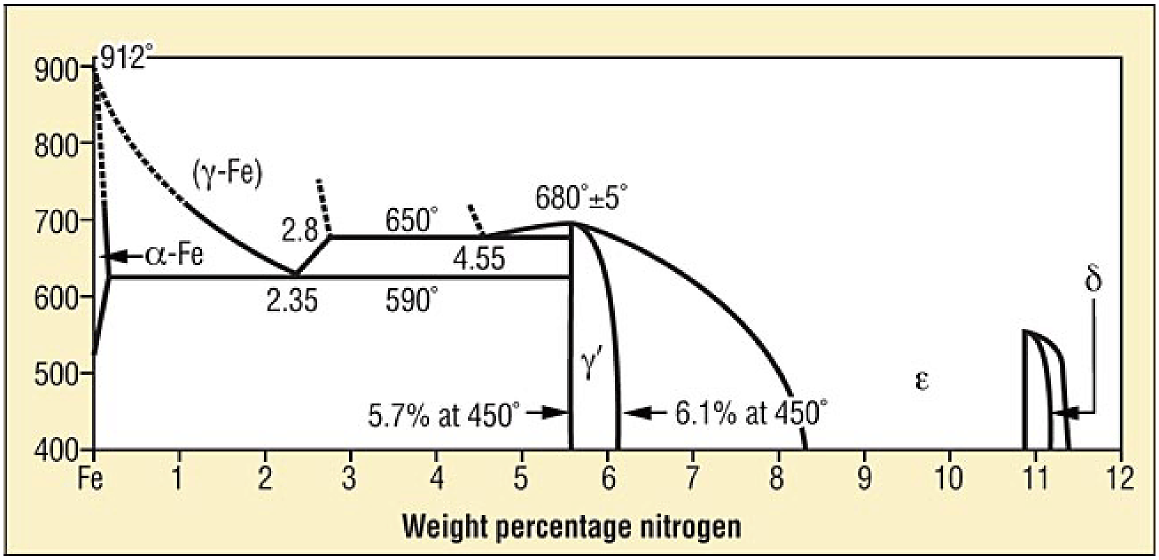


Fig. 1: Iron- nitrogen equilibrium diagram [11]

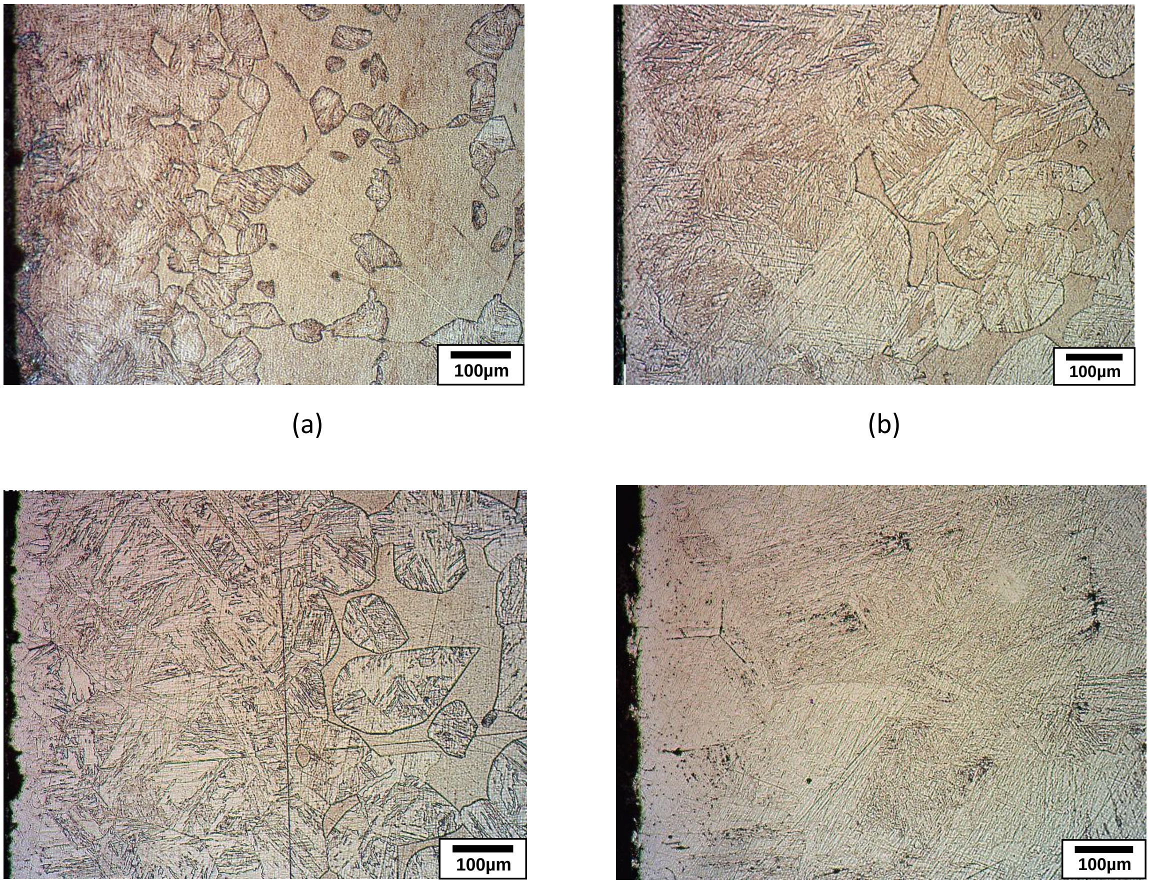
At nitriding temperatures above 450°C, nitrogen will dissolve (interstitially) in ferrite (alpha-iron) but only up to a concentration of 0.1% at 590°C [11]. When nitrogen content exceeds this value, Fe_4N or gamma prime (γ') forms up to a nitrogen content of 5.7-6.1% [11]. This nitride will precipitate at the grain boundaries and preferentially along certain crystallographic planes. As nitriding continues, these nitrides increase in size (as well as quantity) until the entire microstructure has been transformed into a layer of γ’. This is the so-called “compound or white layer.” As the concentration of nitrogen continues to increase, the nitrogen content in the layer also increases. When the nitrogen concentration exceeds 6.1%, γ’ nitride starts to change to Fe_3N and Fe_2N, both referred to as epsilon (ε) nitride [11]. This transformation starts at the surface (where the nitrogen concentration is greatest), and the γ’ layer gradually transforms into epsilon nitride [11].

The definition of microstructure is the structure of material at the microscopic scale [12]. The changes in the microstructure of any material will affect the changes of the material properties. The mechanical strength of many metals and alloys depends very strongly on the grain size. In general, at room temperature a finer grain size leads to higher strength. Many important properties of material are sensitive to the microstructure [12].

The microstructure of the nitrided samples contains martensite and coarse ferrite. The martensite development is begun at the outer layer and grows further into the core when nitriding time is increased. A partial martensite formation was also taken place at the core which was initiated at the ferrite's grain boundary. The existence of the martensite structure contributes to the hardness improvement of the steel. However, nitrogen diffusion is also believed to increase the hardness of the martensite. Prolonging the nitriding time diffuses more nitrogen into the material. It stimulates more martensite as well as the hardness improvement.

Nitrogen diffusion does not change the austenitic phase in austenitic stainless steel due to its higher solubility [5]. However, the solution nitriding of ferritic stainless steel has produced martensite phase [4] and increased the hardness of the steel. Thus, it indicates that the diffusion of nitrogen has a role in the transformation of martensite. Martensite formation due to solution nitriding in 9% Cr ferritic stainless steel was identified [4]. Longer nitriding time increases the case depth. Similar results were obtained when the treatment was applied to AISI 430 ferritic stainless steel. Martensite phase was discovered in nitriding the material at the atmospheric pressure [4] or at higher pressure [8]. Incomplete transformation of austenite and martensite during cooling after solution nitriding has been observed [8]; it leaves retained austenite in the steel. Cr_2N was also found.

The solution nitriding treatments on AISI430 ferrric stainless steel were conducted for 1 hour, 3 hours, 5 hours and 7 hours at 1200^oC [13].



(a) (b)

(c) (d)

Fig. 2: Optical micrographs of AISI 430 ferritic stainless steel after solution nitriding at various temperature and time. (a)1200°C, 1 hour (b)1200°C, 3 hours (c)1200°C, 5 hours (d)1200°C, 7 hours [13]

Fig. 2 shows that at 7hr the martensite structure is fully formed throughout the steel sheet cross section. There are no other structure can be observed except martensite structure.

The solution nitriding has successfully enhanced the surface hardness of AISI 430 ferritic stainless steels [8]. Analysis of the phase transformation and metallographic examination reveals that the nitrogen diffusion yields the martensite formation [14]. Therefore the existence of martensite indicates the nitrogen diffusion into the steel. This fact convinces that longer nitriding time diffuses more nitrogen into the steel. The work confirms that nitrogen has a significant role in the transformation of ferrite to martensite during the solution nitriding of ferritic stainless steel.

CHAPTER 3: METHODOLOGY

3.1 Research Methodology

This section consists of project analysis where it involves data and information gathering, theoretical analysis. Firstly research, collect and summarized data and experimental studies related to nitriding into AISI 430 Ferritic Stainless Steels. Secondly, identify and study on the hardness profile of the material. Thirdly, study the existing formulation of Case Hardened Depth, diffusion coefficient of nitrogen in ferrite and surface nitrogen concentration. After that, the appropriate formulation will be selected and developed to get better results by varying nitriding time. Finally, prior research and experimental results will be compared with the theoretical results from the developed formulations.

3.2 Project Work

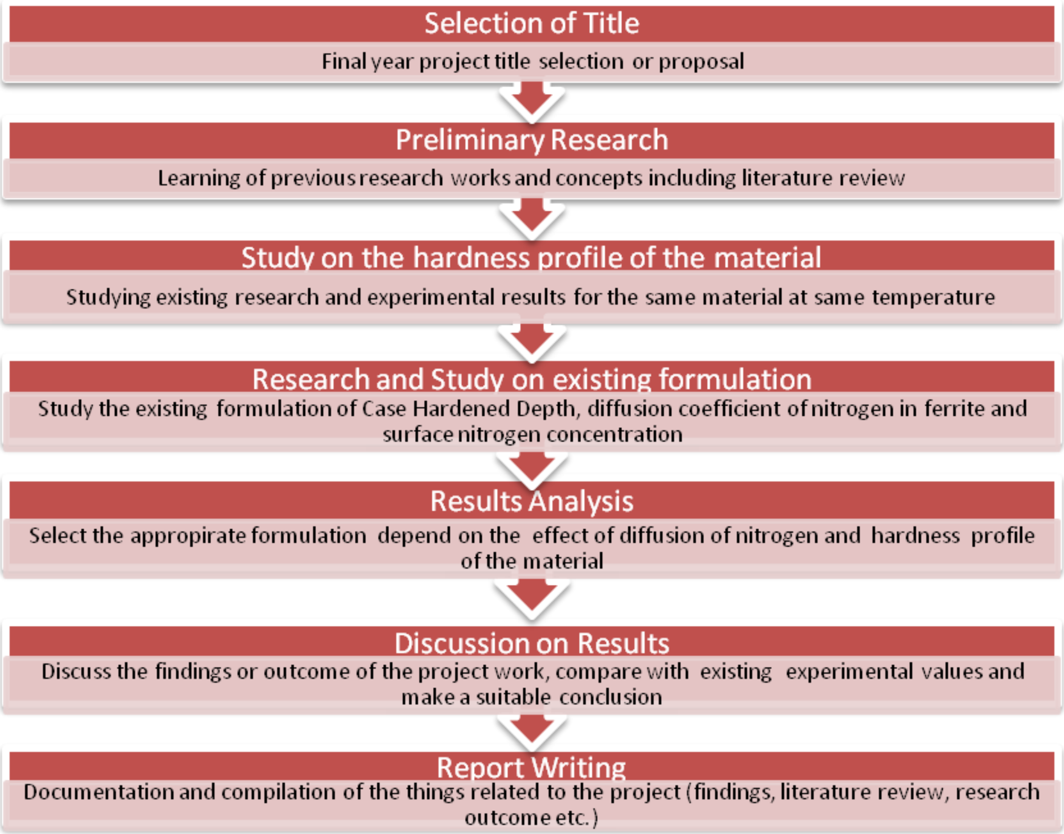


Fig. 3: Project Activities Flow Chart

3.3 Gantt Chart and Key Milestone

No	**Detail / Week**	**1**	**2**	**3**	**4**	**5**	**6**	**7**	**Mid Semester Break**	**8**	**9**	**10**	**11**	**12**	**13**	**14**	**15**
1	Study on existing formulations of case hardened depth	🟦	🟦	🟦													
2	Study on existing formulations of nitrogen diffusion in ferrite and surface nitrogen concentration				🟦	🟦	🟦	🟦									
3	Submission of progress report									★							
4	Develop appropriate formulation by varying nitrding time									🟦	🟦	🟦	🟦	🟦			
5	Pre-EDX												★				
6	Draft Report Submission													★			
7	Dissertation Submission (Soft bound)														★		
8	Technical Paper Submission														★		
9	Oral Presentation															★	
10	Project Dissertation Submission																★

Fig. 4: Gantt chart for the FYP II project implementation

🟦	Activities
★	Milestones

3.4 Tools required

The laboratory works and results will be taken from existing research since the project is only based on empirical modeling. So, familiarizations of below equipments are only needed just to understand the process flow of the equipment.

Scanning Electron Microscope X-Ray Fluorescence (XRF) X-Ray Diffraction (XRD)

Fig. 5: Familiarization of the equipments

Scanning Electron Microscope (SEM), Optical Microscopy (OM), X-Ray Fluorescence (XRF) will be used to characterize the morphology and surface composition. And also XRD tool will be used to characterize the structure, crystallite size (grain size) and grain orientation. Moreover, Microsoft Excel will be used in this project to develop the formulation.

CHAPTER 4: RESULTS AND DISSCUION

4.1 Data Gathering and Analysis

4.1.1 Chemical Composition

Table 4.1: Chemical composition of AISI 430 ferritic stainless steel (wt. %) used in this study [13]

Composition	**Wt. %**
C	0.031
Si	0.240
Cr	16.080
Mn	0.410
Ni	0.160
S	0.003
P	0.031

4.1.2 Pre-existing experimental data

Table 4.2: Case Hardened depth at 1200˚C by varying nitrided time [13]

Temperature(˚C)	Nitrided Time (hr)	Case hardened Depth (mm)
1200	1	0.29
1200	3	0.44
1200	5	0.53

Nitriding involves the diffusion of nitrogen in ferrite and precipitation of nitrides in the diffusion zone. Diffusion coefficients in each phase are estimated by setting the equation associated with growth of compacts nitrided layer in ferrite. **Case hardening** improves both the wear resistance and the fatigue strength of parts under dynamic and/or thermal stresses. The characteristics of case hardening are primarily determined by surface hardness, the effective hardness depth, and the depth profile of the residual stress. Case hardness depth - or the thickness of the hardened layer - is an essential quality attribute of the case hardening process.

In this project, the main equation that will be used to find the case hardened depth [15, 16] is given as below

$$\mathbf{x^2 = 2[N]Dt/r[M]} \qquad (4.1)$$

where

x = hardened case depth (mm)
N = surface nitrogen concentration (at%)
D = diffusion coefficient of nitrogen in ferrite (mm^2/s)
t = time (s)
r = ration of nitrogen to metal in nitride precipitate
M = originally alloy element concentration (at%)

First step is to find the surface nitrogen concentration, below equation can be used [16],

$$\mathbf{Ns = 12.3 \exp(-34700/RT)} \text{ wt\%} \qquad (4.2)$$

[R = 8.314 $Jmol^{-1}K^{-1}$, T = 1200˚C (1473K) (fixed)]
Ns = 12.3 exp (-34700/8.314 x 1473) = 0.723 wt%

However, the calculated surface nitrogen concentration is in term of wt%.

So, after wt% value of surface nitrogen concentration is known, put that value into original composition data and recalculate the total mole of the composition and mole of the nitrogen by using mole = wt%/Atomic weight. So the at% of surface nitrogen concentration is mole of nitrogen/total mol of composition.

Table 4.3: Mole calculation for composition of AISI 430 ferritic stainless steel

Composition	**Wt%**	**At. Weight**	**Mol**
C	0.031	12.011	0.003
Si	0.24	28.086	0.009
Cr	16.08	51.996	0.309
Mn	0.41	54.938	0.007
Ni	0.16	58.693	0.003
S	0.003	32.065	0.000
P	0.031	30.974	0.001
N	0.723	14.007	0.052
Fe	82.322	55.845	1.474
		Total mol=	1.857

Ns (at%) = 0.052/1.857 = 0.028 (x 100) (at%)

Secondly, from the above table, we can also calculate originally alloy element concentration. M value can be calculated by finding mole of Cr and total mole of the composition since original alloy element for this material is Cr.

M = mol of Cr/total mole of the composition

M (at %) = 0.309/1.857 = 0.17 (x 100) (at%)

Thirdly, diffusion coefficient of nitrogen in ferrite can be calculated by the below equation [16]:

$$\mathbf{D}_{N} = \mathbf{6.6 \times 10^{-7} \, exp} \left(-\frac{77900}{RT} \right) \mathbf{cm^2/s} \qquad (4.3)$$

[R = 8.314 $\text{Jmol}^{-1}\text{K}^{-1}$, T = 1200˚C (1473K) (fixed)]

$\mathbf{D}_{N}$ **= 6.6 x** $\mathbf{10^{-7}}$ **exp (- 77900/ (8.314 x 1473)) = 1.14 x** $\mathbf{10^{-9}}$ $\mathbf{cm^2/s}$ **= 1.14 x** $\mathbf{10^{-5}}$ $\mathbf{mm^2/s}$

And then, r value will be put as 0.5 since the pre-existing experimental works show that Cr_2N precipitation was found [14].

Finally, nitrding time will be used as 1hr, 3hr, 5hr so that we can easily compare the final theoretical results with existing experimental results.

So, by using the main equation, the case hardened depth for each nitriding time can be calculated as per below table,

Table 4.4: Case Hardened depth calculation by varying nitriding time

Nitriding Time (hr)	Nitriding Time (s)	Case hardened depth	
		x^2 (mm)	x (mm)
1	3600	0.027	0.16
3	10800	0.08	0.28
5	18000	0.14	0.37

So, comparison can be made between theoretical results done by the equation and experimental results done by previous research.

Table 4.5: Comparison of Case hardened depth (Experimental vs Theoretical)

Temperature (˚C)	**Nitrided Time (hr)**	**Case Hardened Depth (mm) Experimental Results [13]**	**Case Hardened Depth (mm) Theoretical Results**
1200	1	0.29	0.16
1200	3	0.44	0.28
1200	5	0.53	0.37

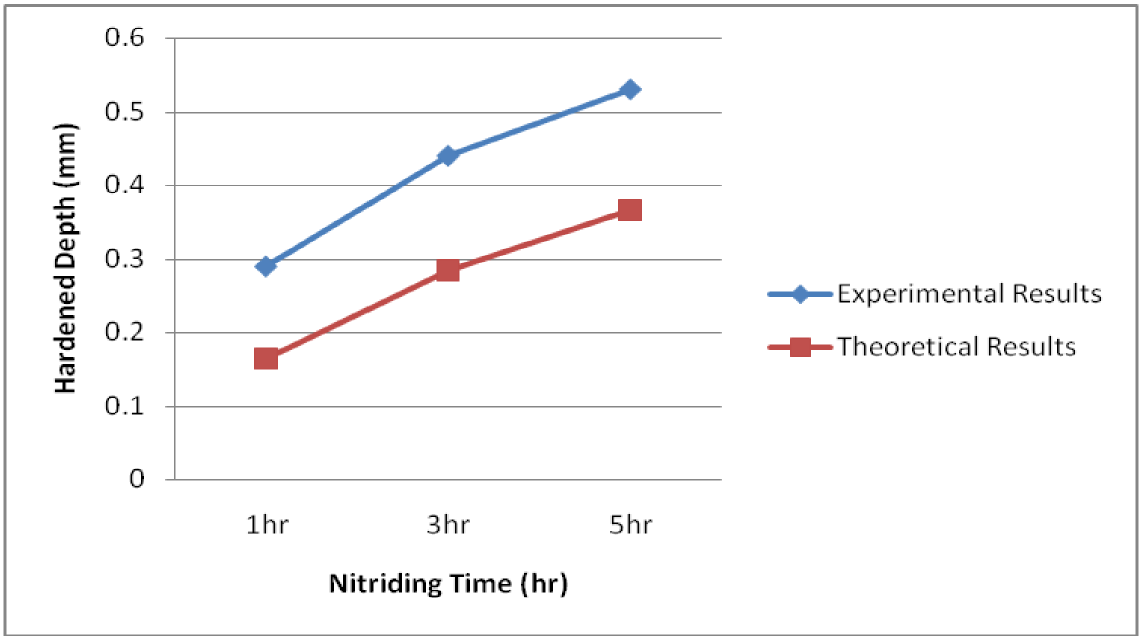


Fig. 6: Comparison of Case hardened depth (Experimental vs Theoretical)

By looking at the above graph, it can conclude that case hardened depth can be calculated without doing experiments. But, there is still a gap between experimental results and theoretical results since it was assumed that Cr_2N precipitation was formed during the process. However, by looking at the result surface nitrogen concentration (Ns=2.8 at %) and temperature (1200˚C), it can conclude that the process was undergoing a phase transition (Incomplete transformation of austenite and martensite during cooling) at that temperature, it leaves retained austenite in the steel and Cr_2N precipitation was actually found according to the literature review.

CHAPTER 5: CONCLUSION AND RECOMENDATION

In this project, the empirical modeling for diffusion of nitrogen into AISI 430 ferritic stainless steel was formulated and developed. The objectives of this study were achieved. However, because of the time constraint, laboratory works and results were referred from previous research to develop the formulation since those were already discovered or conducted before. The diffusion of nitrogen in ferrite, surface nitrogen concentration and precipitation of nitrides in the diffusion zone play significant roles in developing the empirical model. By knowing those values, we can formulate and predict the case hardened depth at specific temperature by varying time. Case hardened depth is an essential quality attribute of the case hardening process. So, this empirical modeling can be applied in the industry for wide application in order to predict case hardened depth so that it will help and serve the future engineers as the guidance or references before doing the actual experiments.

As for the recommendation, this study can be improved by taking consideration of the microstructure and phase occurred in the process at the specific temperature. So that, according to the phase occurred, surface nitrogen concentration can be calculated. For example if Fe_4N phase was occurred in the process, find the nitrogen at% in Fe_4N and recalculate the case hardened depth. The theoretical results will be better and closer to the experimental results than before. And also, this study can be continued by using the various temperatures in order to predict the case hardened depth. To do that, the most important thing to take into consideration are the microstructure of the material, phase occurred during the process and nitriding time. By knowing those, one can predict better theoretical results without doing actual experimental works.

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