



**ITS**  
Institut  
Teknologi  
Sepuluh Nopember

FINAL PROJECT - TM 184835

# FAILURE ANALYSIS OF NBVC COATING FORMATION ONTO AISI D2 TOOL STEEL DURING THERMO REACTIVE DIFFUSION BY PACK METHOD

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DEPARTMENT OF MECHANICAL ENGINEERING  
Faculty of Industrial Technology  
Sepuluh Nopember Institute of Technology  
Surabaya 2019





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**TUGAS AKHIR - TM184835**

**ANALISA KEGAGALAN PEMBENTUKAN  
LAPISAN NBVC PADA BAJA PERKAKAS AISI  
D2 SELAMA *THERMO REACTIVE DIFFUSION*  
MENGUNAKAN METODE *PACK***

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Surabaya 2019

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## **FINAL PROJECT**

Is Proposed to Fulfill Engineering Bachelor Degree Requirement  
In

Mechanical Engineering Department Undergraduate Program  
Faculty of Industrial Technology  
Sepuluh Nopember Institute of Technology

by:

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**SURABAYA  
JANUARY, 2019**

# **FAILURE ANALYSIS OF NBVC COATING FORMATION ONTO AISI D2 TOOL STEEL DURING THERMO REACTIVE DIFFUSION BY PACK METHOD**

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## **ABSTRACT**

*Threads of metal fasteners can be created by thread rolling process using specific thread dies. Thread rolling is a cold forging process that can be carried out on ductile metal. The quality of the thread rolling die is determined by their wear-resistant properties and hardness number of the material. In order to increase the hardness and the wear resistance of the dies, surface treatment can be applied. One of the process that can be applied is thermo-reactive diffusion (TRD) technique due to its simplicity, low in cost, and environment friendly. TRD technique can produce super hard layers of carbide, nitride or carbonitride with the main elements vanadium, niobium, tantalum, chromium, molybdenum or tungsten. The carbon or nitrogen in the steel will diffuse by carbide-forming or nitride-forming elements. The diffused carbon-forming element (CFE) or nitride-forming element (NFE) in the deposited coating to form a dense and bonded carbide or nitride coating at the substrate surface.*

*This research will explore the effect of temperature variation of TRD process onto vanadium-niobium coating formation on the surface of AISI D2. The TRD process is carried out by powder-pack method in a sealed steel container containing powder mixture of niobium and vanadium as master alloy, alumina ( $Al_2O_3$ ) as inert filler and ammonium chloride ( $NH_4Cl$ ) as activator. The samples were covered with powder mixture and then heated in the electric*

*resistance furnace at temperature of 950, 1000 and 1050 °C for 6 hours. Upon finishing the process, the sample is allowed to cool in the open air. The coating cross section was analyzed using optical microscope. The hardness measurement also performed on the same sample. XRD was utilized to investigate the coating chemical composition. The wear of the coatings are assessed using revolving tribological test under dry sliding condition.*

*The microstructure, hardness, chemical composition and wear properties of treated and untreated AISI D2 substrate were studied. The XRD and microstructure test results did not find any niobium-vanadium carbide coating on AISI D2 substrate. In untreated specimens found chromium carbide ( $\text{Cr}_7\text{C}_3$ ), so there is no sufficient supply of carbon to form niobium-vanadium carbide when the thermo reactive diffusion process takes place. In addition, the chromium carbide ( $\text{Cr}_7\text{C}_3$ ) has the lowest free energy of formation compared to NbC and VC. So that the carbide formed only on the substrate does not form a coating. Untreated specimen has hardness number of 277.8 HV. After TRD treatment, the hardness number is increase. For heating temperature variation of 950°C, 1000°C, and 1050°C have hardness number of 798.21HV, 787.3HV, 774.2HV respectively. The value of hardness rises due to the hardening of the AISI D2 specimen. The highest wear resistance owned by temperature variation of 950°C and the lowest wear resistance owned by the untreated specimen.*

**Keyword : thermo-reactive diffusion, niobium-vanadium carbide, AISI D2, hardness, wear.**

# **ANALISA KEGAGALAN PEMBENTUKAN LAPISAN NBVC PADA BAJA PERKAKAS AISI D2 SELAMA PROSES *THERMO REACTIVE DIFFUSION* MENGUNAKAN METODE *PACK***

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## **ABSTRAK**

*Thread dari fastener logam dapat dibuat dengan proses thread rolling menggunakan thread dies yang spesifik. Threading adalah proses penempaan dingin yang dapat dilakukan pada logam ulet. Kualitas cetakan ulir ditentukan oleh sifat tahan aus dan nilai kekerasan material. Untuk meningkatkan kekerasan dan ketahanan aus pada cetakan, perawatan permukaan dapat diterapkan. Salah satu proses yang dapat diterapkan adalah teknik thermo reactive diffusion (TRD) karena kesederhanaannya, murah, dan ramah lingkungan. Teknik TRD dapat menghasilkan lapisan super keras karbida, nitrida atau karbonitrida dengan elemen utama vanadium, niobium, tantalum, kromium, molibdenum atau tungsten. Karbon atau nitrogen dalam baja akan berdifusi oleh unsur pembentuk karbida atau pembentuk nitrida. Elemen pembentuk karbon terdifusi (CFE) atau elemen pembentuk nitrida (NFE) dalam lapisan yang diendapkan untuk membentuk lapisan karbida atau nitrida yang padat dan terikat pada permukaan substrat.*

*Penelitian ini akan mengeksplorasi pengaruh variasi suhu proses TRD terhadap pembentukan lapisan vanadium-niobium pada permukaan AISI D2. Proses TRD dilakukan dengan metode powder pack dalam wadah baja tertutup yang mengandung campuran bubuk niobium dan vanadium sebagai paduan utama, alumina ( $Al_2O_3$ ) sebagai pengisi dan amonium klorida ( $NH_4Cl$ )*



sebagai aktivator. Sampel ditutup dengan campuran bubuk dan kemudian dipanaskan dalam tungku tahanan listrik pada suhu 950, 1000 dan 1050 °C selama 6 jam. Setelah menyelesaikan proses TRD, sampel dibiarkan dingin di udara terbuka. Penampang melintang dianalisis dengan menggunakan mikroskopi optik. Pengukuran kekerasan juga dilakukan pada sampel yang sama. XRD digunakan untuk mengetahui komposisi kimia dari spesimen. Keausan specimen diuji dengan menggunakan alat uji tribologi di bawah kondisi kering.

Struktur mikro, kekerasan, komposisi kimia dan sifat keausan substrat AISI dianalisa sebelum dan sesudah diberi perlakuan. Hasil uji XRD dan mikro tidak menemukan lapisan karbida niobium-vanadium pada substrat AISI D2. Dalam spesimen yang tidak diberi perlakuan ditemukan kromium karbida ( $\text{Cr}_7\text{C}_3$ ), sehingga tidak ada pasokan karbon yang cukup untuk membentuk niobium-vanadium karbida ketika proses teknik thermo reactive diffusion berlangsung. Selain itu, kromium karbida ( $\text{Cr}_7\text{C}_3$ ) memiliki energi bebas pembentukan terendah dibandingkan dengan NbC dan VC. Sehingga karbida yang terbentuk hanya pada substrat dan tidak membentuk lapisan. Spesimen yang tidak diberi perlakuan memiliki nilai kekerasan 277,8 HV. Setelah diberi perlakuan TRD, jumlah kekerasan meningkat. Untuk variasi suhu pemanasan 950°C, 1000°C, dan 1050°C masing-masing memiliki angka kekerasan 798.21HV, 787.3HV, 774.2HV. Nilai kekerasan meningkat karena pengerasan spesimen AISI D2. Nilai ketahanan aus tertinggi dimiliki oleh variasi suhu 950°C dan nilai ketahanan aus terendah dimiliki oleh spesimen yang tidak diberi perlakuan.

**Kata kunci :** *thermo-reactive diffusion*, karbida niobium-vanadium, AISI D2, kekerasan, keausan.

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## **CHAPTER I**

### **INTRODUCTION**

#### **1.1 Background**

Threading is the process of creating a screw thread. There are various methods for generating screw threads. The method chosen for any one application is based on time, money, degree of precision needed (or not needed), what equipment is already available, etc. One method for making threads is thread rolling process. Thread rolling process is the most common method for producing strong, smooth, precise and uniform external threads. Thread rolling is a cold forging process that can be carried out on any ductile metal. This process is widely used to produce special forms, such as knurls. The quality of the thread rolling die is determined by their wear-resistant properties and hardness number of the material. One of the suitable materials for thread rolling die is tool steel AISI D2 which has range of hardness number from 55 to 62 HRC.

AISI D2 tool steel is often referred as semi-stainless because it has a high chromium content of 11 to 13 wt. %. This is not high enough to be classified as stainless steel. AISI D2 steel has high wear resistance and high hardness number properties. This is because AISI D2 steel has a high carbon content. In order to improve the properties of wear resistance and achieve higher hardness number, a surface treatment is recommended for AISI D2 steel. Element such as niobium and vanadium are proposed since niobium metal has soft and ductile properties, while vanadium has a higher hardness number when compared to most of the steels and metals. These two elements are widely used as alloying element for cutting tools. [19]

In industry, the use of surface treatments can improve the mechanical properties of cutting tools, for example can increase hardness and wear resistance. At present, there are efficient surface engineering process to improve surface properties such as physical vapor deposition (PVD), chemical vapor deposition (CVD) and

thermo-reactive diffusion (TRD) techniques. These processes may obtain good result, but each of them has its own weakness. For example, CVD and PVD suffer high cost and operational difficulties. The TRD technique is relatively simple, low in cost, and environment friendly. TRD technique can produce super hard layers of carbide, nitride or carbonitride with the main elements vanadium, niobium, tantalum, chromium, molybdenum or tungsten. The carbon or nitrogen in the steel will diffuse by carbide-forming or nitride-forming elements. The diffused carbon-forming element (CFE) or nitride-forming element (NFE) in the deposited coating to form a dense and bonded carbide or nitride coating at the substrate surface. The TRD process is carried out by powder-pack method in a steel container sealed with alumina cement containing a powder mixture consisting of CFE or NFE as master alloy, alumina ( $\text{Al}_2\text{O}_3$ ) as inert filler and ammonium chloride ( $\text{NH}_4\text{Cl}$ ) as activator. Based on the background to increase the hardness and wear resistance of AISI D2 steel, a research was conducted on coating AISI D2 steel with niobium-vanadium carbide through thermo-reactive diffusion process.

## **1.2 Problem Statement**

Thermo-reactive diffusion technique is influenced by several factors, namely holding temperature, holding time, and material composition. Based on these factors, the formulation of the problem to be examined is as follows:

1. How is the effect of the holding temperature on the substrate resulting from the thermo-reactive diffusion process?
2. How is the mechanical properties of the substrate after thermo-reactive diffusion process?

## **1.3 Objective of the Study**

The objectives of this study are as follows:

1. Investigating the effect of the holding temperature on the substrate resulting from the thermo reactive diffusion process.
2. Knowing the hardness of the substrate after thermo reactive diffusion process.
3. Knowing the wear resistance of the substrate after thermo reactive diffusion process.

#### **1.4 Limitation of the Study**

In order to achieve the objective, the scope are prepared as shown below:

1. The holding time for each temperature is 6 hours.
2. The TRD process is homogeneous on all sides of pack.
3. Composition comparison of niobium and vanadium is 1:1
4. The effect of  $\text{NH}_4\text{Cl}$  as an activator is constant.

#### **1.5 Benefits of Research**

From this research it is expected to provide the following benefits:

1. As a reference for similar research developing.
2. Adding knowledge about the effect of thermo-reactive diffusion techniques, especially using niobium-vanadium as CFE.
3. As a consideration of choosing the right material in the industry, especially material coating industry.

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## CHAPTER II

### LITERATURE REVIEW

#### 2.1 Thread Rolling Dies

Thread rolling is a simple cold forging process that is often used for creating external threads. This is referred as cold forging process because most of the rolling is done in cold blanks. Even so, thread rolling on heated blanks has advantages in some applications. At present, thread rolling is accepted in many industries as a reference method for producing uniform, smooth, and precise threads.

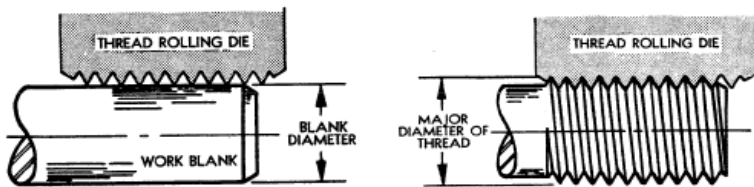


Figure 2.1 Thread Rolling Process [17]

Hardened steel dies are used to roll the thread. The performance of the rolling dies thread depends on 5 factors, namely [8] :

1. Proper design of the die
2. Selection of an proper tool steel
3. Accuracy of the die is made
4. Heat treatment to achieve the optimum hardness and toughness
5. Surface engineering, treatment of the die surface.

A thread rolling die will only work well when the first four factors are met. But over time because of the need for increased die life, factor one and factor three is not a factor that can be improved again. That way, factors one, two, and four are factors that can increase the working lifetime or thread rolling dies.

The rolling dies thread can be manufactured using either machining or grinding. For grinding, the process can be before hardening or after hardening. In general, what is often used is

grinding before hardening because it is very suitable for all types of steel tools, especially AISI D2. However, there is no clear advantage of dies life regarding the process sequence, whether grinding before hardening or grinding after hardening. [8]

The selection of materials for thread rolling dies often requires research to obtain optimal material combinations. Material properties that are very important for thread rolling dies are hardness, toughness, and wear resistance. Hardness and toughness must be high because to hold the force given to the die while working. But, when the hardness number is too high will make the die becomes brittle and cannot hold the given load. Excessive hardness number can cause the dies to crack during the rolling process. The optimal combination of toughness and hardness of die material is a major consideration in most applications. Wear resistance in steel can be enhanced by alloys such as chromium, niobium, vanadium which form hard carbides in microstructure. In addition, the surface modification process can be recommended. The more carbide, the greater the hardness and wear resistance.

## **2.2 AISI D2 Steel**

AISI D2 steel is tool steel for cold working which is often used in mechanical industries, usually as cutting and forming tools. AISI D2 steel is categorized as semi-stainless steel because the chromium content is almost the same as stainless steel. The carbon content in this type of steel is quite high at around 1.4% -1.6% with 11% -13% chromium content. In general, an increase in the level of hardness number of material is followed by an increase in wear resistance properties. Many studies have been carried out to increase the hardness of AISI D2 steel, for example by using conventional heat treatment and surface treatment. This steel is very useful for applications that require high wear resistance properties on dies. AISI D2 steel is usually used as drawing and forming dies, cold drawing punches, blanking or stamping dies, and extrusion dies. [14]

Table 2.1 Composition of AISI D2 steel based on ASTM [4]

%C		%Mn		%Si		%Cr		%V		%Mo	
M in	M ax	Mi n	M ax	Mi n	M ax	Mi n	M ax	Mi n	M ax	Mi n	M ax
1. 40	1. 60	0.1 0	0. 60	0.1	0. 6	11	13	0.5	1. 10	0.7	1. 20

## 2.3 Diffusion

The atoms of metal in a state will arrange themselves to make the atom in a balanced or stable state. Movement or displacement of these atoms can be diffused or by other means. To diffuse, atoms must have enough energy. The higher the energy that is owned, the greater the possibility of diffusion. The energy meant here is thermal energy. The terms of the movement are motivated by two things, namely there must be an empty adjacent side and there is sufficient energy for migrating atoms. The mechanism of diffusion is divided into two types, vacancy diffusion and interstitial diffusion.

### 2.3.1 Vacancy Diffusion

In vacancy diffusion requires a vacuum of atomic structure in the process so that the nearest atom can move to the empty area. Emptiness in atomic structure can occur in heated metal materials. Diffusion of this type, atoms and empty regions move position so that it can be said that there is a movement or displacement of atoms.

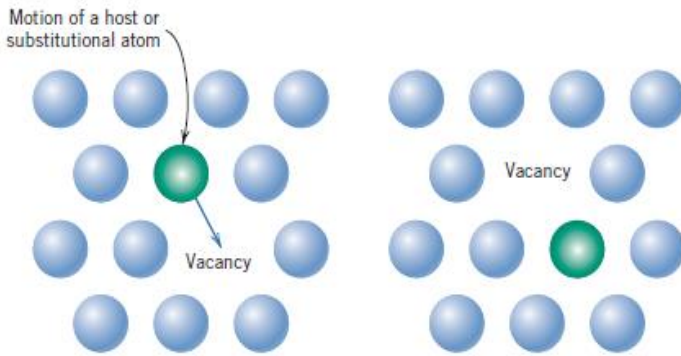


Figure 2.2 Vacancy Diffusion [5]

### 2.3.2 Interstitial Diffusion

Interstitial diffusion is the transfer of atoms from an atomic interstitial position to another empty position. This mechanism can occur in an impure compound, for example compounds containing hydrogen, carbon, nitrogen, and oxygen. Where these atoms have a size that is not the same as other atoms in the bond, so that the atom can be tucked into the interstitial position.

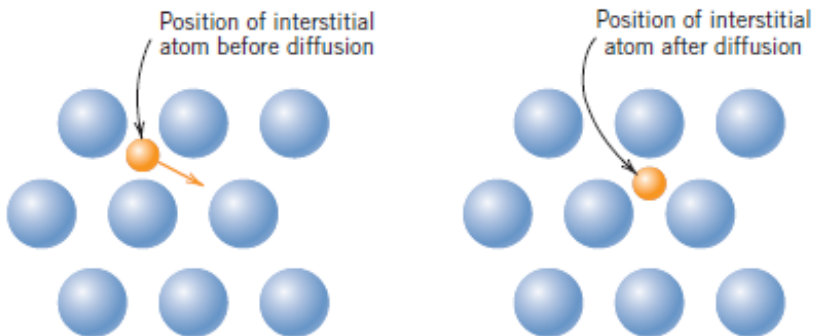


Figure 2.3 Interstitial Diffusion [5]

Most metal alloys have more frequent interstitial diffusion than vacancy diffusion. It is because interstitial atoms are smaller



$$V = B \left[ r^2 \sin^{-1} \left( \frac{b}{2r} \right) - \frac{b}{2} \sqrt{r^2 - \frac{b^2}{4}} \right] \quad (eq. 1)$$

where:

V = volume loss (mm<sup>3</sup>)

B = disk thickness (mm)

r = disk radius (mm)

b = wear length (mm)

## 2.4 Surface Hardening

In the use of a machine or tool section, it is often necessary to have a hard and wear-resistant surface with a relatively soft, resilient, and tough core part. Conventionally hardened steel can indeed produce a hard and resistant surface, but overall the hardened object becomes brittle. Surface hardening is intended to increase surface hardness without affecting the interior which is relatively soft and ductile. This combination of hard surface and soft interior is needed for several applications. One example is the gear that must withstand high pressure and fatigue. In addition, steel whose surface is hardened is more popular because the price is cheap but the quality is superior. Conventional surface hardening techniques are carburizing, where steel is placed in a carbon environment with a high temperature. The carbon will diffuse into steel. At present, surface hardening development is increasingly diverse. One is to use a thermo-reactive diffusion technique. [9]

### 2.4.1 Thermo-Reactive Diffusion (TRD)

Thermo-reactive diffusion is a steel coating method to improve the hardness and wear resistance by forming a layer of carbides, nitrides or carbonitides. In the TRD process, the carbon or nitrogen in the steel will diffuse by carbide-forming or nitride-forming elements such as vanadium, niobium, tantalum, chromium, molybdenum, or tungsten. The diffused carbon-forming element (CFE) or nitride-forming element (NFE) in the deposited coating form a dense and metallurgically bonded carbide or nitride coating at the substrate surface. The TRD coatings are

formed due to the strong affinity of CFE and NFE in coating reagents to the carbon/nitrogen atoms in substrates of the parts to be coated. [21]

The TRD process is not like a conventional hardening process, where specific elements (carbon and nitrogen) diffuse for hardening. The TRD method produces intentional buildup on the surface. Hard layer thickness can be produced from the TRD process around 5 to 15  $\mu\text{m}$ . TRD can be done using salt bath furnace or electric resistance furnace. [2] At the electric resistance furnace reaction pack is needed (Figure 2.5) where the contents are CFE or NFE as master alloy,  $\text{NH}_4\text{Cl}$  as activator,  $\text{Al}_2\text{O}_3$  as inert filler, and substrate.

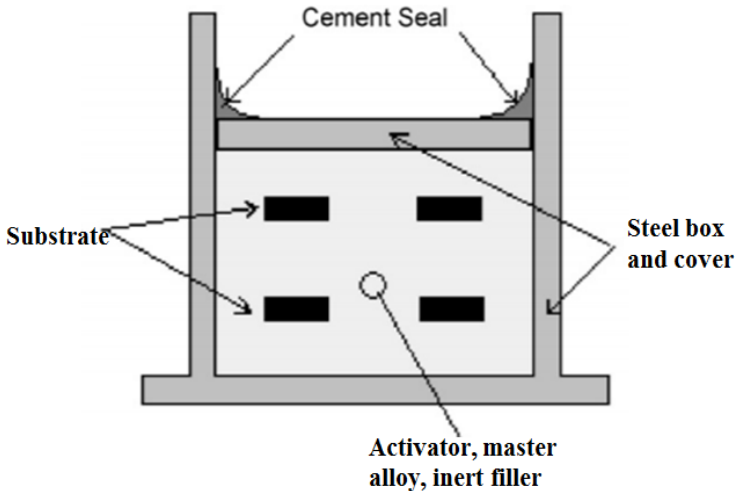


Figure 2.5 Reaction Pack Scheme [1]

The coating growth rate is determined by the rate diffusion of carbon into the surface [2], and if the layer growth is perpendicular to the substrate surface, it can be assumed that the coating growth rate obeys an Arrhenius function (eq. 2) [22].

$$x^2 = Dt \quad (\text{eq. 2})$$

Where  $x$  is the thickness (cm),  $D$  is the diffusion coefficient ( $\text{cm}^2/\text{s}$ ) and  $t$  is the processing time (s).

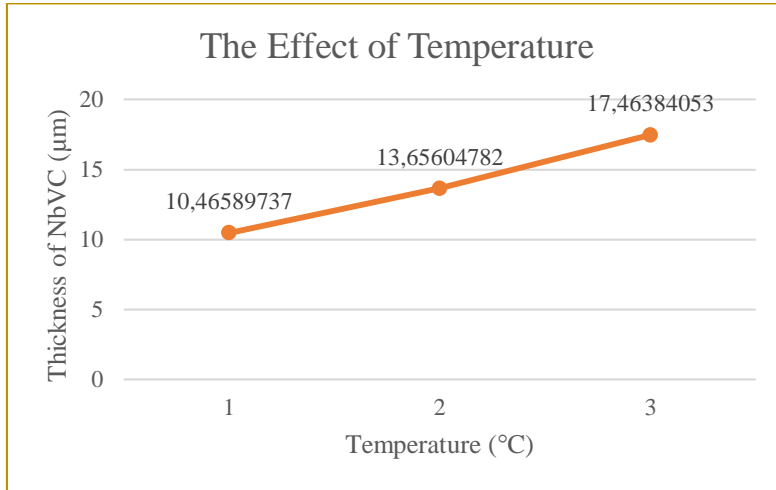


Figure 2.6 The Effect of Temperature on The Thickness of NbVC based on equation 2

By using equation 2, the thickness value is obtained through the diffusion coefficient and processing time. Figure 2.6 shows that the thickness will increase with increasing temperature. So that the graph above will be used as the basis for comparing the results of testing of experiments conducted.

## 2.5 Gibbs Free Energy of Formation

The Gibbs free energy of a system at any moment in time is defined as the enthalpy of the system minus the product of the temperature times the entropy of the system.

$$G = H - TS \quad (eq. 3)$$

The Gibbs free energy of the system is a state function because it is defined in terms of thermodynamic properties that are



state functions. The change in the Gibbs free energy of the system that occurs during a reaction is therefore equal to the change in the enthalpy of the system minus the change in the product of the temperature times the entropy of the system.

$$\Delta G = \Delta H - \Delta(TS) \quad (\text{eq. 4})$$

If the reaction is run at constant temperature, this equation can be written as follows.

$$\Delta G = \Delta H - \Delta TS \quad (\text{eq. 5})$$

The change in the free energy of a system that occurs during a reaction can be measured under any set of conditions. If the data are collected under standard-state conditions, the result is the standard-state free energy of reaction ( $\Delta G^\circ$ ).

$$\Delta G^\circ = \Delta H^\circ - T\Delta S^\circ \quad (\text{eq. 5})$$

The free energy of a system is ability to determine the relative importance of the enthalpy and entropy terms as driving forces behind a particular reaction. The change in the free energy of the system that occurs during a reaction measures the balance between the two driving forces that determine whether a reaction is spontaneous. As we have seen, the enthalpy and entropy terms have different sign conventions.

Favorable	Unfavorable
$\Delta H^\circ < 0$	$\Delta H^\circ > 0$
$\Delta S^\circ > 0$	$\Delta S^\circ < 0$

The entropy term is therefore subtracted from the enthalpy term when calculating  $\Delta G^\circ$  for a reaction. Because of the way the free energy of the system is defined,  $\Delta G^\circ$  is negative for any reaction for which  $\Delta H^\circ$  is negative and  $\Delta S^\circ$  is positive.  $\Delta G^\circ$  is therefore negative for any reaction that is favored by both the enthalpy and entropy terms. We can therefore conclude that any

reaction for which  $\Delta G^\circ$  is negative should be favorable, or spontaneous. Favorable, or spontaneous reactions  $\Delta G^\circ < 0$ . Conversely, C is positive for any reaction for which  $\Delta H^\circ$  is positive and  $\Delta S^\circ$  is negative. Any reaction for which  $\Delta G^\circ$  is positive is therefore unfavorable. Unfavorable, or non-spontaneous reactions  $\Delta G^\circ > 0$ . [28]

For this experiment, the free energy of formation ( $\Delta G^\circ$ ) of vanadium carbide, niobium carbide, and chromium carbide must be known to find out the tendency to form. The following is the free energy of formation of VC, NbC and  $\text{Cr}_7\text{C}_3$ .

Table 2.2 Free Energy of Formation [27]

Compound	Formulas (cal)	$\Delta G^\circ$ (cal)		
		T = 950°C	T = 1000°C	T = 1050°C
VC	$-24100 + 1.5T$	-22266	-22191	-22116
NbC	$-31100 + 0.4T$	-30611	-30591	-30571
$\text{Cr}_7\text{C}_3$	$-29985 - 7.41T$	-38717	-39074	-39431

## **CHAPTER III METHODOLOGY**

### **3.1 Experimental Flowchart**

Flowchart is a diagrammatic illustration shows in boxes represent the process overview takes place in this project. In the Figure 3.1 below shows a flow chart for experimental TRD process.

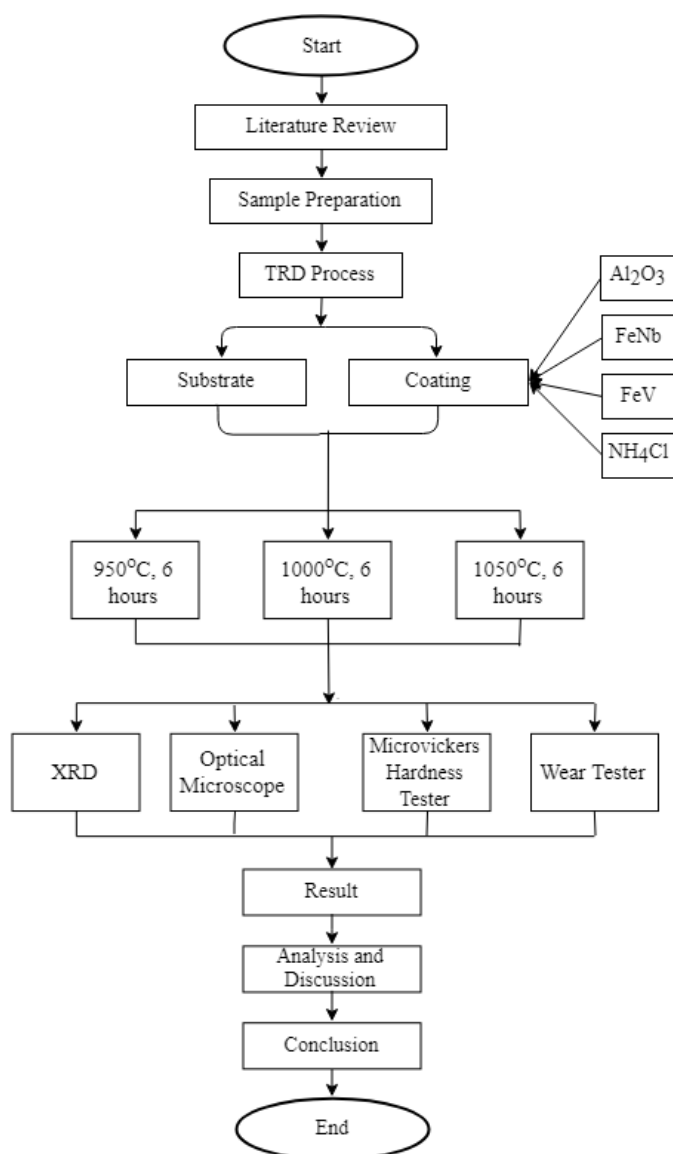


Figure 3.1 Experimental Flowchart

### 3.2 Experimental Overview

In this study, experiments are being carried out in determining the effect of the carbide layer produced on AISI D2 steel from thermo-reactive diffusion. The TRD process is carried out by powder-pack method in a steel container sealed with alumina cement containing a powder mixture consisting of niobium and vanadium as master alloy, alumina ( $\text{Al}_2\text{O}_3$ ) as inert filler and ammonium chloride ( $\text{NH}_4\text{Cl}$ ) as activator. The samples were placed in a box with a powder mixture and then put into the furnace at 950, 1000 and 1050 °C for 6 hours. After the TRD process, the sample is allowed to cool in the open air. Coating thickness measurement is carried out by an optic digital microscope. Hardness measurement using Vickers hardness tester. To investigate the wear resistant of uncoated and coated test pieces, a dry sliding wear test is performed using pin on disc set up.

### 3.3 Materials and Tools of Experiment

#### 3.3.1 Materials

##### 3.3.1.1 AISI D2 Tool Steel as Substrate

AISI D2 tool steel is one of the materials used to make thread rolling dies. This type of steel is often calls as semi-stainless steel because the carbon content almost the same as stainless steel. AISI D2 steel has high wear resistance and high hardness number properties. This is because AISI D2 steel has high carbon content. Table 3.1 shows the composition of AISI D2 obtained from the test results at PT. Barata Indonesia. The results indicate different results from the ASTM specifications.

Table 3.1 Chemical Composition of AISI D2

%C	%Si	%Ni	%Cr	%Mo	%V	%N	%Fe
1.064	3.289	1.288	11.923	0.889	0.939	1.020	5.321

### 3.3.1.2 FeNb and FeV as Master Alloy

Ferroniobium and ferrovanadium are chosen as master alloys because they can form coating of NbVC on the surface of AISI D2. Table 3.2 and Table 3.3 shows the composition of FeNb and FeV.

Table 3.2 Chemical Composition of FeNb

P	0.8%
Al	2.0%
C	0.3%
Ta	0.2%
Nb	65%
Si	3.0%
S	0.1 %

Table 3.3 Chemical Composition of FeV

S	0.1%
V	75%-82%
Si	2.0%
P	0.1%
C	0.25%
Al	4.0%

### 3.3.1.3 Al<sub>2</sub>O<sub>3</sub> as Inert Filler

The reaction pack filler used in this experiment is Al<sub>2</sub>O<sub>3</sub> because it has high temperature resistance with a melting point of 2072°C and a boiling point of 2977°C. Al<sub>2</sub>O<sub>3</sub> also does not react with ionized FeNb or FeV so it does not interfere with the TRD process that takes place.

### 3.3.1.4 NH<sub>4</sub>Cl as Activator

The activator used in this study is NH<sub>4</sub>Cl. The function of the activator itself is to break down FeNb and FeV to bind to the carbon in AISI D2 steel. The effect of activator is considered constant from the start of the treatment to completion.

### 3.3.1.5 Reaction Pack

The reaction pack used in this study has a function to limit the diffusion reaction, so that it is not disturbed by the external system. The material used for this reaction pack must also have high temperature resistance properties so that it does not interfere with the diffusion process. Figure 3.2 shows the reaction pack. The following are the specifications of the reaction pack.

Material	: ASTM A36
Thickness	: 10mm
Dimension (outside pack)	: 18.4mm x 9.93mm x 6mm
Dimension (inside pack)	: 20.69mm x 12.14mm x 7mm



Figure 3.2 Reaction Pack

### 3.3.2 Tools

#### 3.3.2.1 Wire Cut

To make the specimen into a smaller part that is 5 mm thick with a diameter of 35 mm, a cutting tool is needed. In this case, the cutting tool used is a wire cut. Wire cut includes cutting tools that have a good level of precision.

### 3.3.2.2 Electric Resistance Furnace

Electric resistance furnace is a tool used to increase temperature test pieces by inserting them into the furnace. This tool has automatic control to adjust the expected temperature, regulate holding time, and adjust the time needed to make the expected temperature. Figure 3.3 shows the electric resistance furnace in heat treatment workshop of Mechanical Engineering Department ITS.



Figure 3.3 Electric Resistance Furnace

### 3.3.2.3 Grinder – Polisher

Grinder is used to smooth the surface of specimens that will be used for research observations. The grinding process is carried out gradually using scouring paper at the level of the roughness grid, starting from the smallest grid to the large one while flowing water until the surface of the specimen becomes smooth.

Polisher is used to smooth and gloss the specimen, where the process uses a velvet cloth sprinkled with alumina powder or metal polishing while watering until the specimen surface becomes smooth and shiny. Figure 3.4 shows the grinder-polisher engine used in this experiment.





Figure 3.4 Grinder-Polisher

#### **3.3.2.4 Optical Microscope**

Observation of the thickness of the coating is done using an optical microscope with a digital camera as shown in Figure 3.5 connected to a computer unit. Images obtained from a microscope are sent to digital still recorders through a digital camera and then stored by a computer in the form of documents. This equipment can be used to observe with a magnification of 100x to 1000x according to the needs of the observer.



Figure 3.5 Optical Microscope

#### **3.3.2.5 X-Ray Diffraction (XRD)**

XRD testing is carried out in the ITS Material and Metallurgical Engineering Department with the aim to identify the substrate formed in the coating of NbVC.

#### **3.3.2.6 Micro Vickers Hardness Tester**

Micro Vickers hardness tester aims to determine the mechanical properties of hardness from the coating of NbVC formed from the TRD process. This test will show whether the coated area is harder than the uncoated area. The test was carried out on the coating area of NbVC formed, and the substrate with each data averaged for the hardness number of each region. Figure 3.6 shows the micro Vickers hardness tester in the Mechanical Engineering Department ITS.



Figure 3.6 Micro Vickers Hardness Tester

### 3.3.2.7 Wear Tester

To investigate the wear resistant of uncoated and coated test pieces using revolving disc under dry condition. Wear test were done in Bahan Teknik Laboratory of Mechanical Engineering Department UGM.

### 3.4 Procedure of Conducting Research

The following is the procedure for conducting research evaluating niobium-vanadium carbide coatings on AISI D2 produces through TRD:

- Collect sources (book, journal, etc.) as research references.
- Preparing research tools and materials by considering the methods used, namely thermo-reactive diffusion process.

- Assembly and welding of the reaction pack according to the specified dimensions.
- Cut the substrate (AISI D2) using wire cut. The dimension is 3.5 mm for the diameter and 0.5 for the thickness.
- The preparation of substrate is performed through metallographic polishing with 120, 240, 320, 400, 600, 800, 1000, and 2000 sandpaper.
- Fill the reaction pack with powder mixture which consists of 10 wt. % Fe-Nb, 10 wt. % Fe-V, 5 wt. %  $\text{NH}_4\text{Cl}$ , and 75 wt. % alumina. Put the substrate inside them.

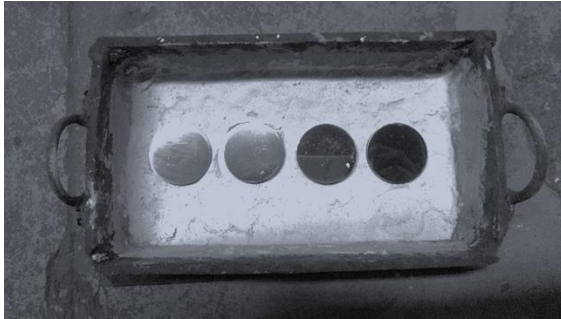


Figure 3.7 inside the Reaction Pack

- TRD treatment on test pieces according to the desired temperature is  $950^{\circ}\text{C}$ ,  $1000^{\circ}\text{C}$  and  $1050^{\circ}\text{C}$  with holding time of each temperature is 6 hours.



Figure 3.8 Reaction Pack in the Furnace

- Let the samples are left to cool down in open air.

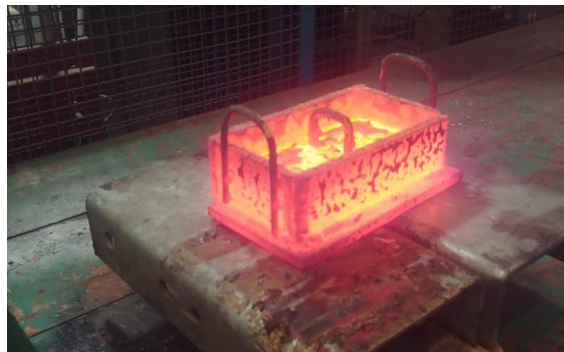


Figure 3.9 after TRD Process

- Preparing samples for topographic, elemental and mechanical testing.
- Testing samples using XRD, hardness test, microstructure using optical microscope, and wear test.
- Analyze test results.
- Make conclusions from the analysis.

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## **CHAPTER IV**

### **RESULT AND DISCUSSION**

#### **4.1 Introduction**

The TRD process is carried out by powder-pack method in a sealed steel container containing powder mixture 10 wt.% Fe-Nb, 10 wt.% Fe-V, 5 wt.%  $\text{NH}_4\text{Cl}$  and 75 wt.%  $\text{Al}_2\text{O}_3$ . This research will explore the effect of temperature variation of TRD process onto vanadium-niobium coating formation on the surface of AISI D2. The samples were covered with powder mixture and then heated in the electric resistance furnace at temperature of 950, 1000 and 1050 °C for 6 hours. Upon finishing the process, the sample is allowed to cool in the open air. The coating cross section was analyzed using Optical Microscopy. The hardness measurement also performed on the same sample. XRD was utilized to investigate the coating chemical composition. The wear of the coatings are assessed using revolving tribological test under dry sliding condition.

#### **4.2 XRD Test Result**

Chemical composition testing was carried out to determine the chemical composition of untreated and treated specimens. Testing uses XRD with  $2\theta$  varying from 10° to 80°. The following is the result of XRD pattern of the specimen.

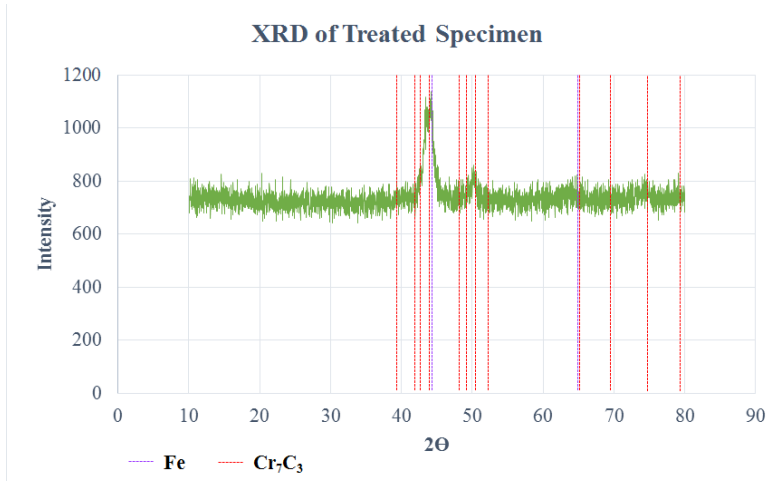


Figure 4.1 XRD Patterns of Treated Specimen

The XRD pattern of treated specimen is shown in Figure 4.1. The results showed that neither niobium carbide nor vanadium carbide was found. The carbide found is chromium carbide ( $\text{Cr}_7\text{C}_3$ ). XRD testing is also performed on specimen untreated. The following are the results.



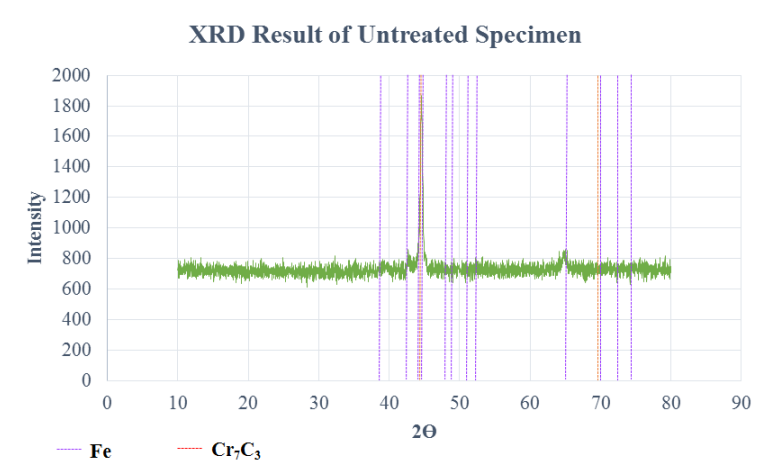






Figure 4.2 XRD Pattern of Untreated Specimen

The XRD pattern of untreated specimen is shown in Figure 4.2. The results show that there are chromium carbides ( $\text{Cr}_7\text{C}_3$ ) at several peaks. This result is the same as the content found in treated specimens.

### 4.3 Microstructure Test Result

To prove the results of XRD testing, microstructure testing is needed. Microstructure observation was carried out using an Olympus optical microscope. The first observation is to find out whether there is coating formed or not. The following is the observation of each specimen.

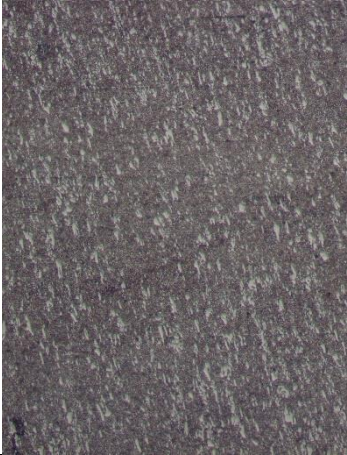
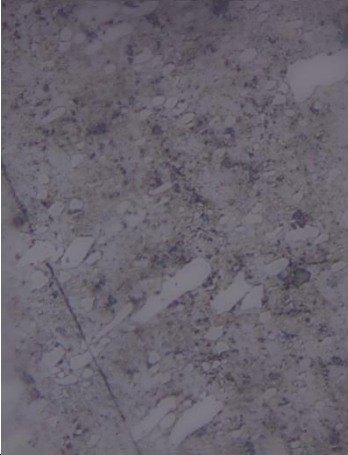
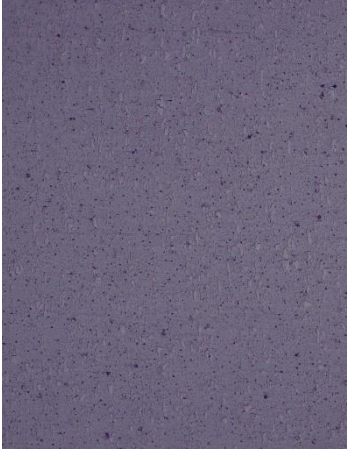
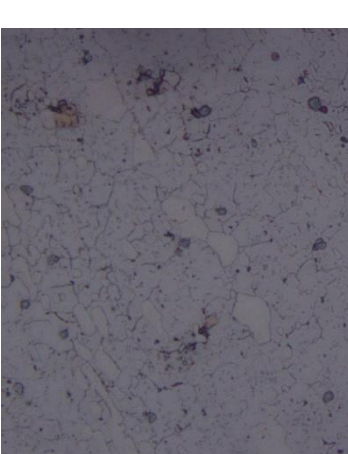
Table 4.1 Coating Observation Results from Thermo Reactive Diffusion with the magnification of 50x and 100x

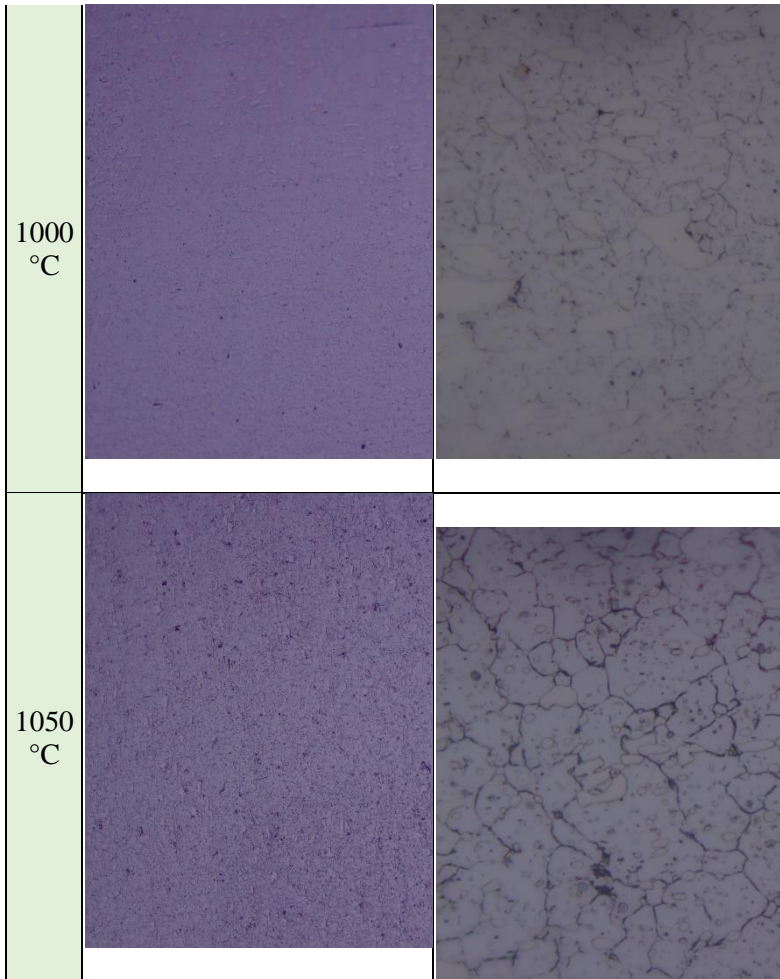
Treat ment	Magnification 50x	Magnification 100x
950° C		
1000 °C		



The observation results shown in Table 4.1 do not indicate the presence of a coating formed on AISI D2 substrate. This reinforces the XRD results in Figure 4.1 where there is no vanadium-niobium carbide. Further observation of the microstructure is needed to analyze the causes of the formation of nioboium-vanadium carbide on the surface of AISI D2 substrate. The following are observations of microstructure in untreated and treated specimens where the specimens have been etched first.

Table 4.2 Microstructure Observation Result of Etching Specimen

Treat ment	Magnification 100x	Magnification 1000x
Untre ated		
950° C		



The microstructure observations showed that in untreated and treated specimens there were carbides. Carbide here is chromium carbide ( $\text{Cr}_7\text{C}_3$ ) according to the results obtained from XRD testing. The presence of chromium carbide ( $\text{Cr}_7\text{C}_3$ ) in untreated specimen makes carbon supply to form carbides with

niobium-vanadium to be less so that carbide coating is not formed. Untreated specimens are purchased in annealed condition which are they have  $(\alpha + FeC) + Fe_3C + (M_7C_3)$  phase. To change the chromium carbide phase to austenite phase, a heat treatment with a temperature of more than  $1300^{\circ}C$  is required according to the picture of the phase diagram in Figure 4.3.

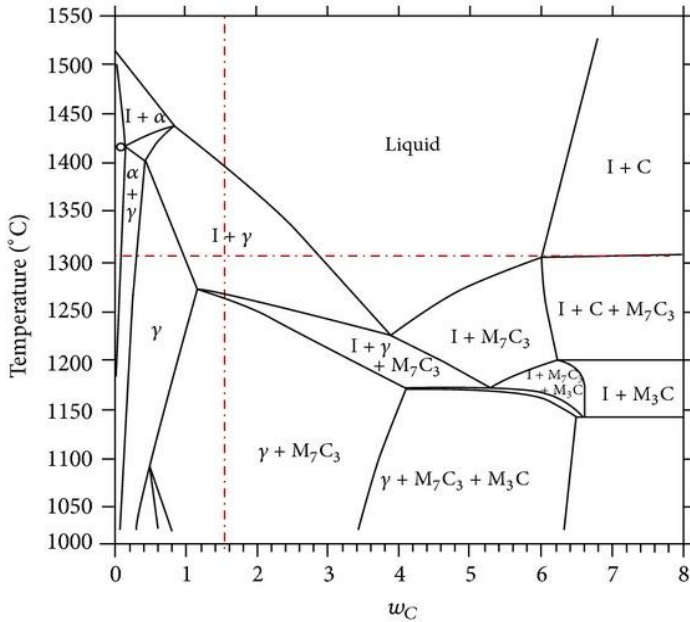


Figure 4.3 Phase Diagram of AISI D2 Tool Steel [25]

Other factors that can cause non-formation of niobium-chromium carbide coating are competing factors between niobium, vanadium and chromium to bind to carbon to form carbides. The higher the value of free energy formation, the reaction cannot occur spontaneously [27]. According to calculation on Table 2.2 VC has highest energy to form and  $Cr_7C_3$  has lowest energy to form. So that the carbide formed only on the substrate does not form a coating. Figure 4.1 showed that the content of  $Cr_7C_3$  is increase. It

is because the solubility of austenite is decrease then the chromium and carbon in the austenite will diffused interstitially. The carbon and chromium will form carbide as presipitation in the specimen.

#### 4.4 Hardness Test Result

Microhardness testing was performed to determine the hardness of the AISI D2 tool steel and specimen after TRD treatment with variations in the heating temperature of 950°C, 1000°C, and 1050°C. Tests carried out as many as 10 points in each specimen with a load of 0.5 HV. The following are the results of microhardness testing on each specimen.

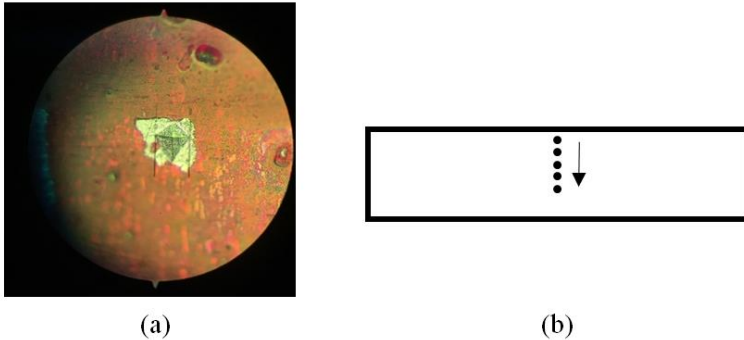
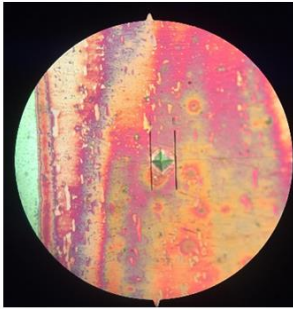
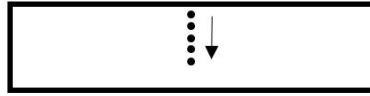


Figure 4.4 (a) Indentation Result of AISI D2 Tool Steel  
Untreated  
(b) Indentation Position of AISI D2 Tool Steel Untreated



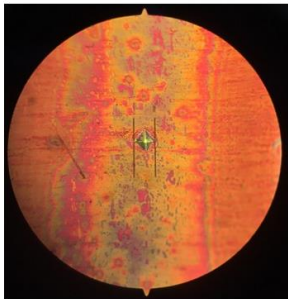
(a)



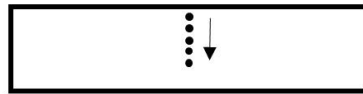
(b)

Figure 4.5 65(a) Indentation Result of Specimen with TRD Treatment at 950°C Heating Temperatures

(b) Indentation Position of Specimen with TRD Treatment at 950°C Heating Temperatures



(a)

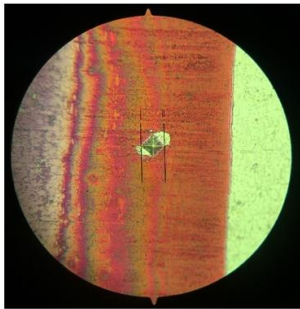


(b)

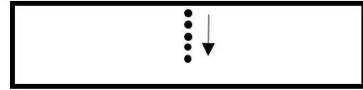
Figure 4.6 (a) Indentation Result of Specimen with TRD Treatment at 1000°C Heating Temperatures

(b) Indentation Position Specimen with TRD Treatment at 1000°C Heating Temperatures





(a)



(b)

Figure 4.7 (a) Indentation Result of Specimen with TRD Treatment at 1050°C Heating Temperatures  
(b) Indentation Position of Specimen with TRD Treatment at 1050°C Heating Temperatures

Table 4.3 Micro Vickers Hardness Result

Point	Untreated	950	1000	1050
1	278	869	858	776
2	306	836	850	794
3	259	807	828	792
4	255	793	791	781
5	281	771	732	777
6	283	782	780	787
7	297	765	770	756
8	275	732	759	756
9	250	743	739	749
10	294	884	766	774
Average	277.8	798.2	787.3	774.2
Standard Deviation	18.6118242	51.0355	44.21928	15.73249

From the test, the hardness number of each specimen was obtained. Untreated specimens have the smallest hardness number which is the differences are far enough to treated specimens. The highest hardness is obtained from the treatment at a temperature of 950°C which the average is equal to 798.2HV. The value of hardness increases because of the hardening process. [26]

#### 4.5 Wear Test Result

Wear testing was assessed to determine wear resistance from the results of TRD treatment with temperature variations of 950°C, 1000°C, and 1050°C compared to untreated. The tool used in wear testing has an output rotation speed of 729.3 rpm and the load of 62,328N. Wear testing was performed out on both sides of each specimen in 16 minutes. Wear scars obtained from the test were observed using an Olympus optical microscope to find wear length. The following is an observation using an optical microscope.

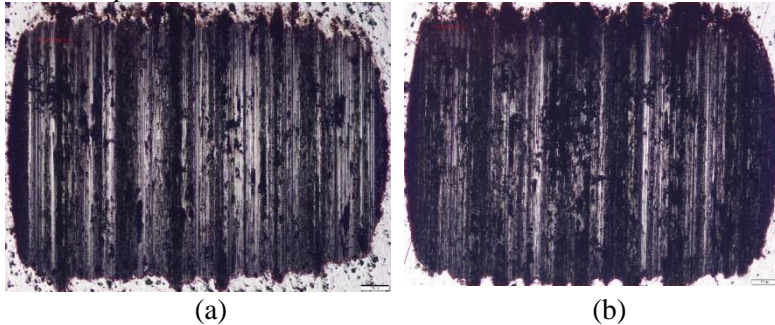


Figure 4.8 Wear Scars Result of AISI D2 Tool Steel on Side (a) A  
(b) B

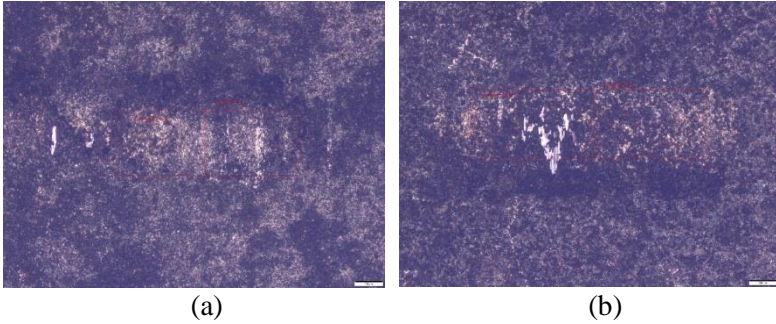


Figure 4.9 Wear Scars Result of Specimen with TRD Treatment at 950°C Heating Temperatures on Side (a) A (b) B

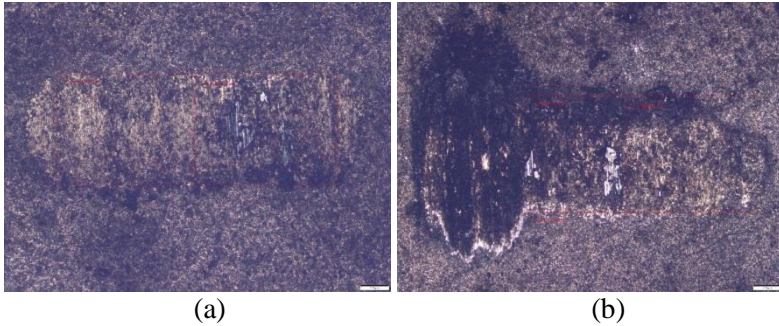


Figure 4.10 Wear Scars Result of Specimen with TRD Treatment at 1000°C Heating Temperatures on Side (a) A (b) B

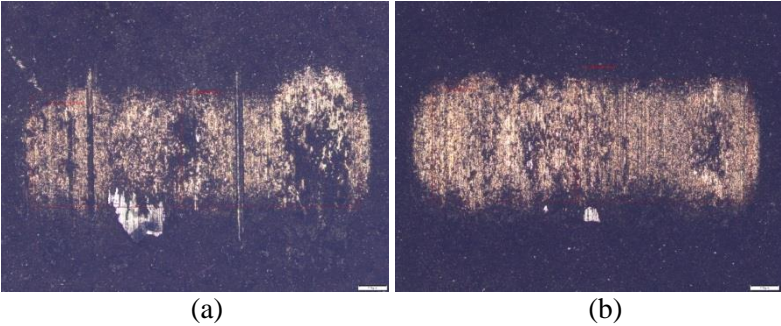


Figure 4.11 Wear Scars Result of Specimen with TRD Treatment at 1050°C Heating Temperatures on Side (a) A (b) B

Volume loss is calculated using Eq. 1. The results can be seen in Table 4.4 below.

Table 4.4 Wear Test Result

Material	Treatment	Width		Volume Loss		
		Side A	Side B	Side A	Side B	Average
AISI D2	Untreated	1.92E+00	2.13E+00	2.53E-01	3.48E-01	0.30025
	950°C	7.82E-01	7.97E-01	1.71E-02	1.81E-02	0.01757
	1000°C	8.26E-01	8.49E-01	2.02E-02	2.18E-02	0.02101
	1050°C	8.21E-01	8.69E-01	1.98E-02	2.34E-02	0.02163

From the results of the study obtained volume loss values from each specimen. Volume loss is defined as wear from the specimen. The more volume loss, the wear resistance of the specimen gets lesser. Table 4.4 shows that the treatment specimen at 950°C has the smallest average volume loss which is equal to 0.01757 mm<sup>3</sup>. While the specimens at the temperatures of 1000°C and 1050°C have almost the same volume loss, which is equal to 0.02101 mm<sup>3</sup> and 0.02163 mm<sup>3</sup> respectively.

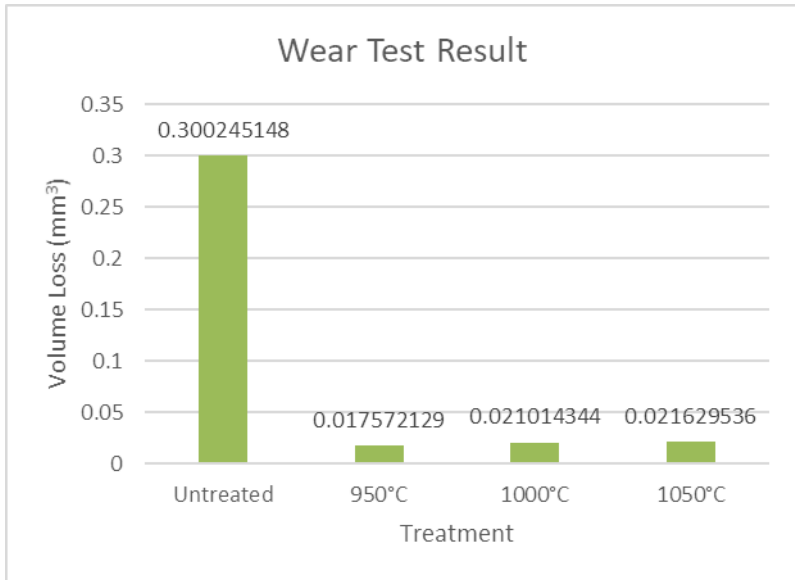


Figure 4.12 Comparison of Wear Test Result of Coated and Uncoated

Figure 4.11 shows the volume loss comparison between treated and untreated specimens. Untreated specimens had the highest volume loss of 0.30025 mm<sup>3</sup> compared to TRD treatment specimens. Treated specimen at 950°C has 17 times less volume loss than untreated specimen. While the treated specimens with the temperatures of 1000°C and 1050°C have volume loss of 14 times compared to untreated specimen. This proves that the wear resistance of specimens is increased due to the treatment. The wear test results are in accordance with the hardness number. Hardness is the ability of specimens to resist indenting loads. Based on the hardness number, specimen with treatment of 950°C has the highest hardness number.

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## **CHAPTER V**

### **CONCLUSION AND RECOMMENDATION**

#### **5.1 Conclusion**

Thermo reactive diffusion were done on AISI D2 steel. The microstructure, hardness, chemical composition and wear properties of treated and untreated AISI D2 substrate were studied. Based on the obtained results, it can be concluded:

1. The XRD and microstructure test results did not find any niobium-vanadium carbide coating on AISI D2 substrate. In untreated specimens found chromium carbide ( $\text{Cr}_7\text{C}_3$ ), so there is no sufficient supply of carbon to form niobium-vanadium carbide when the thermos reactive diffusion process takes place. In addition, the chromium carbide ( $\text{Cr}_7\text{C}_3$ ) has the lowest free energy of formation compared to NbC and VC. So that the carbide formed only on the substrate does not form a coating.
2. Untreated specimen has hardness number of 277.8 HV. After TRD treatment, the hardness number is increase. For heating temperature variation of 950°C, 1000°C, and 1050°C have hardness number of 798.21HV, 787.3HV, 774.2HV respectively. The value of hardness rises due to the hardening of the AISI D2 specimen. The highest wear resistance owned by heating temperature variation of 950°C and the lowest wear resistance owned by the untreated specimen. This value is in accordance with the hardness number.

#### **5.2 Recommendation**

After doing the research, there were some suggestions to obtain more comprehensive information, namely:

1. Before the TRD process is carried out, the AISI D2 tool steel material needs to be heated until the  $\text{Cr}_7\text{C}_3$  phase turns into austenite



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