FINAL PROJECT – TM184835

THE EFFECT OF THE HOLDING TIME ON THE FORMATION OF NIOBIUM-VANADIUM CARBIDE COATING ONTO AISI 420 BY THERMO REACTIVE DIFFUSION PROCESS

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TUGAS AKHIR – TM184835

PENGARUH PENAHANAN WAKTU TERHADAP PEMBENTUKAN FORMASI LAPISAN KARBIDA NIOBIUM-VANADIUM PADA AISI 420 DENGAN PROSES THERMO-REACTIVE DIFFUSION

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APPROVAL PAGE

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FINAL PROJECT

Submitted for fulfill one of the requirement for a bachelor's degree in the Department of Mechanical Engineering, Faculty of Industrial and Engineering Systems, Sepuluh Nopember Institute of Technology, Surabaya.

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THE EFFECT OF THE HOLDING TIME ON THE FORMATION OF NIOBIUM-VANADIUM CARBIDE COATING ONTO AISI 420 BY THERMO-REACTIVE DIFFUSION PROCESS

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ABSTRACT

. The deep drawing process had been widely used in automobile, aerospace, electronics, and allied industries to produce hollow parts. The quality of the deep drawing is determined by the dies properties especially their wear-resistant and hardness. Austenitic stainless steel AISI 420 is one of the material of choice for making these dies. In order to increase the wear resistance of the dies, surface treatment such as thermo-reactive diffusion (TRD) technique can be applied owing to their simplicity, low cost, and environment friendly process. TRD technique able to deposit hard layers of carbide, nitride, or carbonitride

In this research, TRD techniques was employed to AISI 420 with special attention given to the effect of holding time during TRD process to form niobium-vanadium carbide (NbVC) coating. The TRD process is carried out by the powder-pack method in a sealed steel container containing powder mixture of niobium and vanadium as master alloy, alumina (Al_2O_3) as an inert filler and ammonium chloride (NH_4Cl) as an activator. The samples were covered with powder mixture and then heated in the electric resistance furnace for 2, 4, and 6 hours at 1000˚C.

The crosssection analysis of the NbVC coating observed under microscopy shows that coating thickness increased with longer holding time with 6.95 μm measured in 6 hours holding time. The high hardness of 1333.2 HV also found in the longest *holding time of 6 hours. This high hardness is expected to contribute in the wear resistance characterisitic of AISI 420 dies.*

Key word : : Deep drawing process, thermo-reactive diffusion, niobium-vanadium carbide, AISI 420, hardness

THE EFFECT OF THE HOLDING TIME ON THE FORMATION OF NIOBIUM-VANADIUM CARBIDE COATING ONTO AISI 420 BY THERMO REACTIVE DIFFUSION PROCESS

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ABSTRAK

The Deep Drawing process telah banyak digunakan di mobil, dirgantara, elektronik, dan industri terkait untuk menghasilkan bagian berlubang. Kualitas gambar dalam ditentukan oleh sifat cetakan terutama tahan aus dan kekerasannya. Baja tahan karat Austenitik AISI 420 merupakan salah satu material pilihan untuk membuat cetakan ini. Untuk meningkatkan ketahanan aus pada cetakan, perawatan permukaan seperti teknik Thermo-Reactive Diffusion (TRD) dapat diterapkan karena kesederhanaan, biaya rendah, dan proses yang ramah lingkungan. Teknik TRD mampu mengendapkan lapisan keras karbida, nitrida, atau karbonitrida

Dalam penelitian ini, teknik TRD akan digunakan pada AISI 420 dengan perhatian khusus pada pengaruh waktu penahanan selama proses TRD untuk membentuk lapisan niobium-vanadium karbida (NbVC). Proses TRD dilakukan dengan metode powder-pack dalam wadah baja tertutup yang berisi campuran serbuk niobium dan vanadium sebagai master alloy, alumina (Al_2O_3) sebagai pengisi inert dan amonium klorida (NH_4Cl) sebagai aktivator. Sampel ditutup dengan campuran serbuk kemudian dipanaskan dalam tungku tahan listrik selama 2, 4, dan 6 jam pada suhu 1000˚C.

Analisis penampang lapisan NbVC yang diamati dengan mikroskop menunjukkan bahwa ketebalan lapisan meningkat dengan waktu tahan yang lebih lama dengan 6,95 µm yang diukur dalam waktu tahan 6 jam. Kekerasan yang tertinggi 1333.2 HV juga ditemukan pada holding time paling lama yaitu 6 jam. Kekerasan yang tinggi ini diharapkan berkontribusi pada karakterisitik ketahanan aus pada dies AISI 420.

Kata kunci: Deep Drawing Process, difusi termo-reaktif, niobium-vanadium karbida, AISI 420, kekerasan

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Surabaya, 21 Agustus 2020

Author
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CHAPTER I
INTRODUCTION

1.1 Research Background

Various methods have been employed to improve the formability of deep drawing process, usually represented by drawability. This was defined as the capacity of the material to assume the designed shape without losing its stability, avoiding fractures, achieve maximum forming force necessary. Sheet metal drawability is usually expressed by its limiting drawing ratio (LDR) which, for an axisymmetric cup, is defined as the ratio of the largest blank diameter that can be formed to the punch diameter. One main factor that affects the formability of the deep drawing process is the frictional force present between the blank and the forming tools. Several methods, such as having harder materials for the forming tools , better surface finish and the use of lubricants have been implemented with much success in reducing friction .

Another method that can be implemented into forming tools is introducing hard coating. Studies have shown that longer tool life, increased productivity, improved workpiece quality and reduced machining forces are experienced when cutting tools are coated with metal carbides, nitrides and oxides. Nevertheless, there are only limited study available on hard coatings for deep drawing tools even though the coatings had proven to increase the performance of forming and cutting tools.

There are several method available to introduce hard coating onto forming and cutting tools such as chemical vapor deposition (CVD), physical vapor deposition (PVD), lase cladding (LC), in situ synthesis (ISS) and thermo reactive diffusion (TRD). Although CVD, PVD, LC, ISS techniques provide excellent hard coating, these processes are considered expensive and sometimes complex, TRD method is considered as least expensive and simpler as compared to other methods. TRD method is process were a substrate is either immersed into molten borax (salt bath immersion method) or covered with mixture of powder (powder pack method) containing carbide forming element (CFE) such as chrominum, molydenum, vanadium, and niobioum.

Forming and Cutting tools usually using a cold working tool steel such as AISI 420 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 420 steel has high wear resistance and high hardness number properties. This is because AISI 420 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 420 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.

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 time on the thickness of the niobium-vanadium carbide layer resulting from the thermo-reactive diffusion process.
2. How is the relation between holding time and mechanical properties of the material that has undergone a hard coating process of niobium-vanadium carbide through a thermo-reactive diffusion process.

1.3 Objective of the Study

The objectives of this study are as follows:

1. Knowing the effect of the holding time on the thickness of the niobium-vanadium carbide layer resulting from the thermo-reactive diffusion process.
2. Knowing the hardness of the niobium-vanadium carbide layer affected by holding time of TRD process.

1.4 Scope of the Study

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

1. The TRD process is homogeneous on all sides of pack.
2. Composition comparison of niobium and vanadium is 1:1
3. The effect of NH_4Cl 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 development
2. Adding knowledge about the effect of thermo-reactive diffusion techniques, especially using niobium-vanadium based carbide.
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 Deep drawing process

Cup drawing or deep drawing is one of the widely used sheets of metal forming operations. Cup-shaped objects, utensils, pressure vessels, gas cylinders, cans, shells, kitchen sinks, etc are some of the products of deep drawing[7]. The deep drawing process is one of the important sheet metal forming processes. traditional deep drawing process by which a sheet of metal with a certain thickness is converted to a certain shape of geometry, the blank (undeformed sheet) is placed onto the die which has a cavity, and it pressed by punch into the die cavity. the schematic illustration of the deep drawing process and variable of it shown in Figure 2.1.

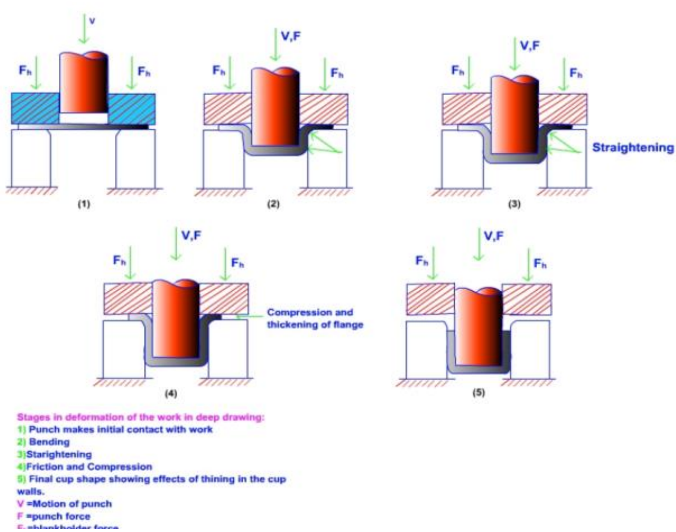

Figure 2.1 Cup drawing process sequence operation [7]

2.2 AISI 420 Martensitic Stainless Steel

Martensitic Stainless Steel is steel with a chromium content of between 11.5% to 18%, for example, steel of type 420, 440, and 501. This type of steel has magnetic properties, can be carried out cold and hot work easily, satisfactory machinability, especially with low carbon content, has good toughness, shows good corrosion resistance to weather, and some chemicals. This steel can achieve the best corrosion resistance when hardened from the recommended temperature.

The heat treatment process for martensitic steels is the same as ordinary carbon steels or low alloy steels, where maximum strength and hardness depend on carbon content. The main difference is that the high alloy content of this steel causes the transformation to be very slow, and hardenability is very high, the maximum hardness produced by air cooling. This steel is usually hardened by heating it above the transformation temperature range to 1010 ° C, then cooled using air or oil media. The temperature holding time should not be excessive to prevent excessive decarburization or grain growth. Tempering carried out on steel is usually carried out above 600 ° C. Higher tempering temperatures will cause carbide precipitation by decreasing corrosion resistance properties AISI 420 steel is tool steel for cold working which is often used in mechanical industries, usually as cutting and forming tools.. 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 420 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 420 steel is usually used as drawing and forming dies, cold drawing punches, blanking or stamping dies, and extrusion dies.

Table 2.1 Composition of AISI 420 steel based on ASTM F899[9]

%C	%Mn	%Si	%Cr	%P	%S
0.15	1.00	1.00	12.0-14.0	0.040	0.030

2.3 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 surfaces and soft interior is needed for several applications. One example is the gear that must withstand high pressure and fatigue. Also, 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. [10]

2.4 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 is a land or an empty side and there is energy for migrating atoms. The mechanism of diffusion is divided into two types, vacancy diffusion, and interstitial diffusion.

2.4.1 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 the 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.

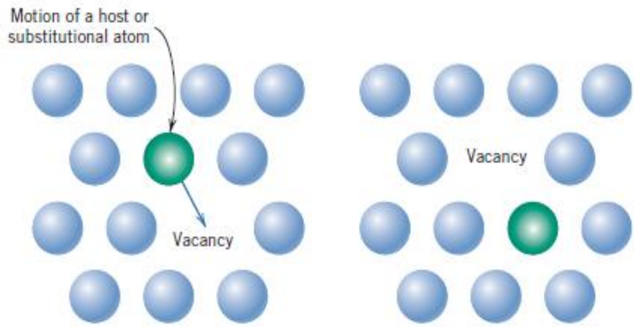

Figure 2.2 Vacancy Diffusion [18]

2.4.2 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. Most metal alloys have more frequent interstitial diffusion than vacancy diffusion. It is because interstitial atoms are smaller so they can move freely. Also, in the atomic structure, there are more interstitial than vacancy positions. [18]

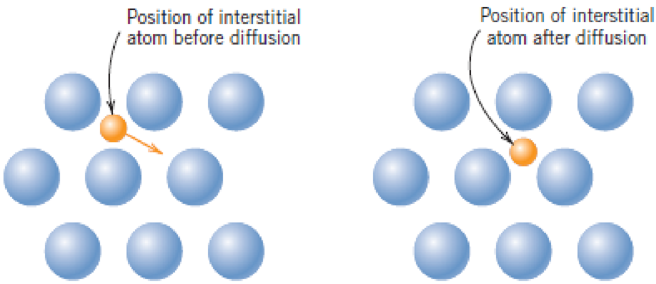

Figure 2.3 Interstitial Diffusion [18]

2.4.3 Thermo Reactive Diffusion (TRD)

Thermo-reactive Diffusion (TRD) processes are different from general surface hardening processes such as carburizing or nitriding. The TRD process is one of the diffusion hardening methods. In the TRD powder pack process, the component or tool to be coated is placed in a container containing a mixture of alumina powder and Ferro niobium (carbide forming element) and heated at a temperature of 850-1050 C for 2 to 10 hours [1]. In addition to the powder pack process, the TRD process can also be carried out on a salt bath with the main components of borax being liquefied at temperatures of 850-1050 C [4]. Carbide forming elements in the main element will diffuse and bind to the carbon in the steel and form a hard layer of carbide compounds with the help of activators. For the TRD powder pack process, the most commonly used activator is ammonium chloride (NH4Cl). These carbide compound layers generally have high hardness, good wear resistance, and good corrosion resistance [11]. Other carbide forming elements can also be added to form a carbide composite layer. Other elements that can be added include vanadium, chromium, niobium, and also titanium. To be able to distinguish between layers formed by the coating process and diffusion of carbide elements can be seen in **Figure** 2.4.

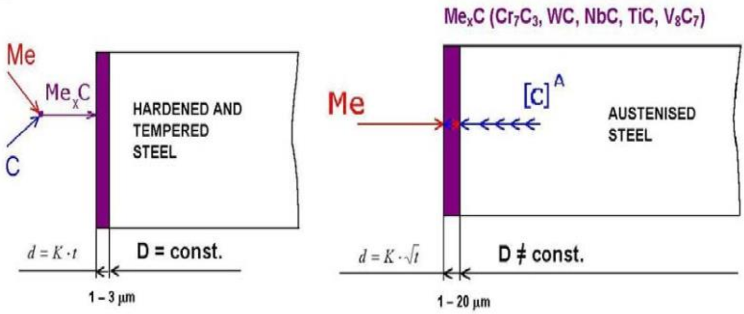

Figure 2.4 the basic difference between coating / diffusion and carbide diffusion scheme [4]

The TRD process starts with preheating at a temperature of 500-700 oC to reduce distortion and reduce heating time when TRD takes place. The TRD process itself is carried out at 850-1050 oC for 4-10 hours. Heating temperature conditions and holding time will greatly affect the thickness of the formed layer of niobium carbide. The thickness produced in the TRD process generally ranges from 2 - 20 µm.

After the TRD process, steel is generally cooled with air, salt, oil, or nitrogen to get a hard base metal so that the difference in hardness between the layers of niobium carbide and base metal can be reduced. This will increase the bonding ability between the hard layer of niobium carbide to base metal because of reduced stress.

To recover steel toughness and ductility after a rapid cooling process to increase the hardness of B base metal, steel is generally treated with tempering by reheating at 200-540 oC. The reheating process aims to eliminate the residual stress contained in steel due to the rapid cooling process.

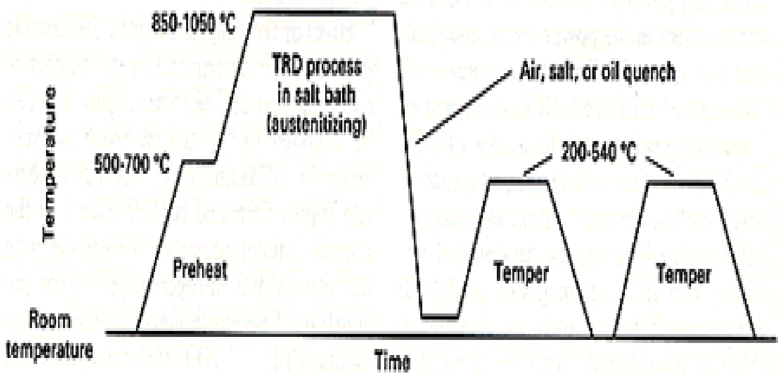

Figure 2.5 Thermo Reactive Diffusion (TRD) heat treatment scheme [1]

For the TRD process as a whole starting from sample preparation can be written as follows:

1. The steel that the TRD process wants to do is the preparation of sandpaper and polish which aims to remove surface oxides that can blocking the niobium from diffusing into the steel.
2. After the sample preparation is carried out the TD process is accompanied by reheating according to Figure. This reheating process aims to remove residual stress in steel due to the process fast cooling. This reheating process performed 3 times at 180 oC for 3-5 hours.
3. After washing using water, the sample is done polish before used in applications. This process aims to smooth the surface and aesthetics.

In the TRD process, the carbide-forming elements contained in the active ingredient in alumina pack powder will diffuse into the steel and bind to the carbon in the steel and then form carbides that have high hardness. The formation of carbide in the TRD process takes place through the stages:

1. The carbide forming elements added in powder form are evenly distributed in alumina media.
2. The ammonium chloride activator will help the formation of niobium ions and also cause the carbon on the steel surface to become active.
3. Carbon in steel becomes active and can bind to ions from carbide-forming elements so that it reacts on the steel surface to form a hard layer of carbide.

The carbide layer on the surface thickens due to a continuous reaction between carbon from steel and ions from the activation of carbide-forming elements as the TRD process progresses.

2.5 Previous Research

Soltani et al. [4] conducted TRD on AISI L2 using Nb as a master alloy. The temperature variations used are 900, 950, 1000, and 1050 ° C at 6 hours. Increasing temperature of the treatment leads to increase the thickness of NbC. Most researchers [1, 12, 13] have reported that the optimum process is about 1000 ° C. It has been reported that for the treatment temperature below 900 ° C, it takes a long time to ensure that the coating layer is formed. For the time variation used by Soltani et al. are 2, 4, 6, and 8 hours at 1000 ° C. The thickness of coating is represented in Figure 6 showing that the thickness of coating increase and the rates are decreased for 6 hours of treatment.

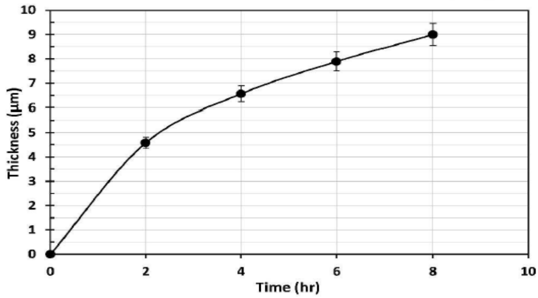

Figure 2.6 Thermo Reactive Diffusion (TRD) heat treatment scheme [1]

It has been reported that the optimum treatment time for process is about 2-7 hours [14]. The optimum treatment time should be determined according to the kind of substrate steel and treatment temperature. In general, the treatment temperature has more pronounced effect on coating growth compared to the effect of treatment time. Soltani et al. also experimenting about the effects of powder mixture. This TRD was carried out at a temperature of 1000 ° C for 6 hours of treatment. Figure 7 shows the thickness for different amounts of Fe-Nb and NH_4Cl.

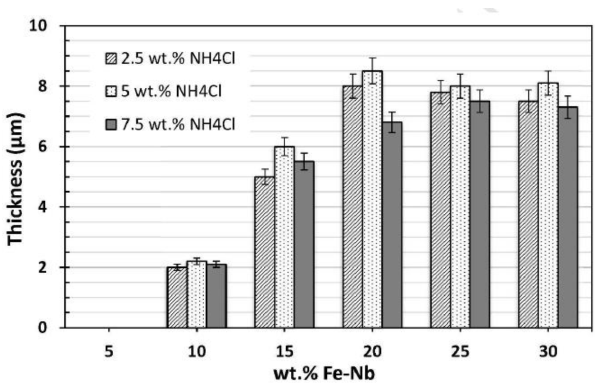

Figure 2.7 Effect of Powder Mixture (Soltani et al., 2017) [4]

It can be seen that Fe-Nb amounts more than 20 wt.% Have no significant effects on thickness and quality of coatings. Therefore, the optimum amount of Fe is required for the process is about 20 wt.%. This amount is in agreement with the study of Sricharoenchai et al. [15] that suggested 10 wt.% Fe-Nb as an optimum amount. On the other hand, by increasing the amount of NH_4Cl from 2.5 to 5 wt.%, The thickness of coating is increased but it decreases from 5 to 7.5 wt.% NH_4Cl. So, the optimum amount of NH_4Cl for the process based on Soltani et al. is 5 wt.%. So that overall for the results of optimal thickness coatings is at temperature treatment 1000°C with a time of 6 hours and powder mixture containing 5% wt NH_4Cl and 20 wt% Fe-Nb.

Castillejo et al. [16] conducted TRD using molten borax with added ferroniobium, ferrovanadium, and alumunium at temperatures of 1223, 1293, and 1363 K for 2, 3, 4, and 5 hours. AISI D2 tool steel is used as substrate. The dimension of the substrate were 15mm in diameter and 4 mm in thickness. The coatings were produced using salth bath formed by molten borax ($Na_2B_4O_7$) 8 wt. % ferroniobium (FeNb), 8 wt. % ferrovanadium (FeV), and 3 wt. % aluminum (Al). Figure 8 shows the cross section from SEM result of vanadium-niobium obtained at temperatures of 1363 and 1223 K for a deposite time of 5 hours for both coatings. The coatings have thicknesses of 19.3±3.2 µm and 9.6±0.8 µm, respectively. Higher values of thickness were obtained at the higher temperature.

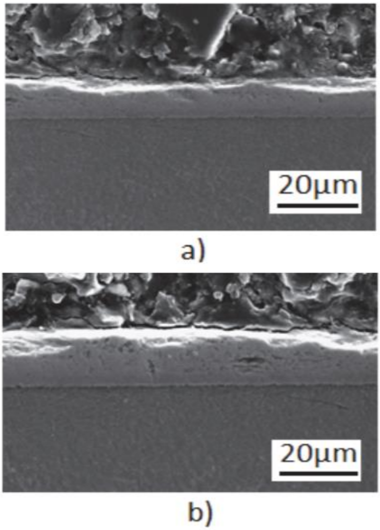

Figure 2.8 SEM Image of a) Niobium-Vanadium Carbide at 1223 K and 5 hours. b) Niobium-Vanadium carbide at 1363 K and 5 hours. [16]

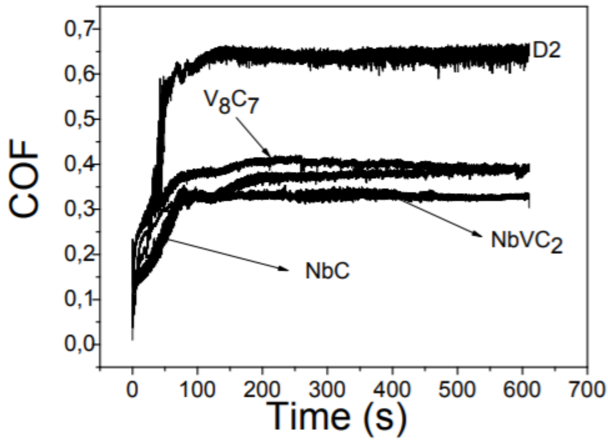

Figure 2.9 Friction Coefficient in Carbide Coating on AISI D2 Steel [16]

Figure 2.9 **Friction Coefficient in Carbide Coating on AISI D2 Steel [16]** shows the curve of the coefficient of friction as a function of the duration of wear testing for binary and ternary carbides systems deposited on AISI D2 steel using ball-on-disk test. From the figure it can be established that the coefficient of friction (COF) of the binary carbides is less by about a factor two compared to the COF substrate. In the ternary carbide systems, the COF values exhibit little change as a function of the percentage of vanadium, the coating with less vanadium having a lower COF value.

Sen U. [17] was evaluate tribological properties of niobium carbide coated AISI 1040 steel. In coating process, TRD by pack method was performed at the temperatures of 800, 900, and 1000 °C for 1 to 4 hours. A pin sample was prepared by coating the substrate material, AISI 1040 plain carbon steel**.** Niobium carbide coating was carried out in an electrical resistance furnace under atmospheric pressure and then left to cool in open air. Wear test of the niobium carbide coated and uncoated steel sample were performed via AISI D2 steel disk (780 HV) by the configuration of a flat end of pin vs. disk for 1 km, under the load of 15 N and 30 N at the sliding speeds of 0.5, 1, 2 and 5 m/s in the atmospheric condition (62% relative humidity).

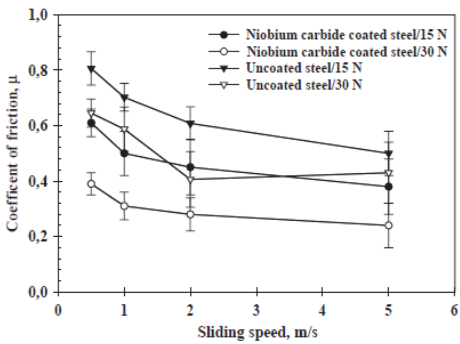

Figure 2.10 Coefficient of Friction of Niobium Carbide Coated and Uncoated AISI 1040 Steel

Figure 2.10 presents the coefficient of friction of niobium carbide coated and uncoated AISI 1040 steel with sliding speed under 15 N and 30 N load values. In this figure, the coefficient of friction of niobium carbide coated AISI 1040 steel is about 75% of that of uncoated steel. Furthermore, the coefficient of friction of uncoated and coated steel decrease with increase in sliding speed and load values. Thus, friction coefficient values exhibit significant load and speed dependency.

2.6 Research Variation

In this research, the author will investigate the effect of holding time of TRD process made by powder pack methods onto coating formation and hardness properties.

The variation of the holding time itself means that the holding time that is carried out will be different after the material is entered into the furnace for the heat treatment process. The time that was varied was the time after preheat.

The variation of the holding time that will be carried out in this research is detention for 2 hours, 4 hours, and 6 hours with a temperature of 1000^{o}C. Taking the temperature of 1000^{o}C itself is seen from previous research which shows that the optimum temperature for the TRD process is 1000^{o}C. After determining the temperature and holding time, then next is the mixture powder for the TRD process itself in which consists of NH_4Cl, Al_2O_3, FeNb, and FeV where the presentation taken from previous research shows that the most optimal mixing is 5% NH_4Cl 20% CFE (Fenb Fev) and the rest. is Al_2O_3.

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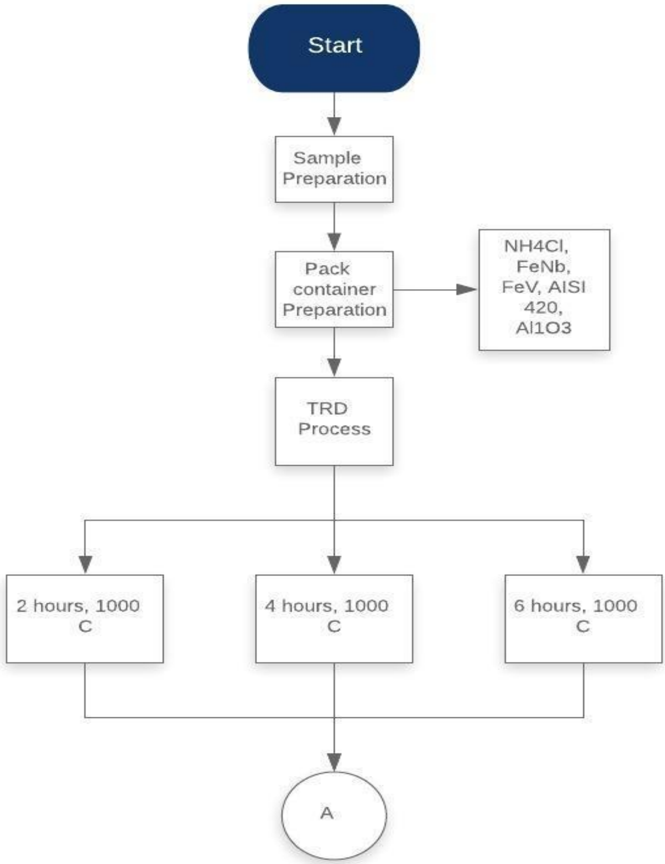
CHAPTER III
METHODOLOGY

3.1 **Experiment Flowchart**

The experiment of this study is summarized in the flow chart as shown in Figure 3.1 below.



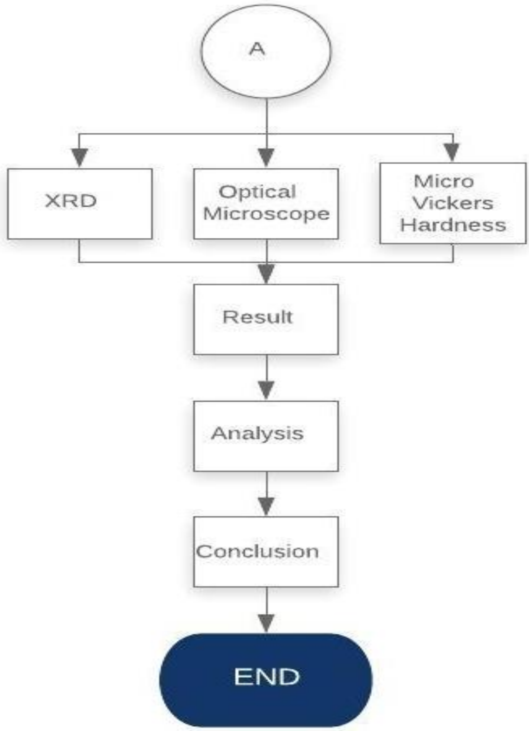


Figure 3.1 Experiment Flowchart

3.2 Experimental Overview

Substrates with the dimensions 0.5 cm × 3cm (height × width) were made of AISI 420 steel bars. Samples were then grinding by SiC abrasive papers up to 2000 mesh. 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 (Al_2O_3) as inert filler and ammonium chloride (NH_4Cl) as activator. The samples were placed in a box with a powder mixture and then put into the furnace at 1000°C for 2 hours,4 hours, and 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.

3.3 Materials and Tools of Experiment

3.3.1 Materials

3.3.1.1 AISI 420 Austenitic stainless steel as Base Metal

The composition of AISI 420 from the test at PT. Barata Indonesia is shownt in Table 3.1 below.

Table 3.1 AISI 420 Chemical Composition

%C	%Si	%Ni	%Cr	%Mo	%S	%Mn	%P
0.35	1.24	0.31	13.808	0.314	0.003	0.59	0.019

3.3.1.2 FeNb and FeV as Master Alloy

Ferroniobium and ferrovanadium are chosen as master alloy bcause they can form hard layers of coating on the surface of AISI 420. The composition of FeNB and FeV is shown in Table 3.2 and Table 3.3.

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_2O_3 as Inert Filler

The Al_2O_3 is chosen as inert filler because it has high temperature resistance with a melting point at 2072 C and boiling point at 2977 C. Al_2O_3 also does not react with ionized FeNB and FeV so it does not interfere the TRD process.

3.3.1.4 NH_4Cl as Activator

The activator used in this study is NH_4Cl. The function of the activator itself is to break down FeNb and FeV to bind to the carbon in 420 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

Metal cutting is done to adjust the dimensions of the sample to the place for testing to be carried out after treatment. The cutting tool that will be used is a wire cutting because it has a high level of cutting precision. This treatment is carried out in the Mechanical Engineering Department FTIRS ITS Surabaya.

3.3.2.2 Electric Resistance Furnace

Electric Resistance Furnace is a device used to increase the temperature of the desired object in the device. This tool already has automatic control to adjust the expected temperature, holding time and the time needed to reach the expected temperature.

Specification :

Brand: Nabertherm
Type: N 11 / HR
Made: Germany
Number: 211108
T max: 1250 °C
The year 2009

Voltage: 400 V / 3 Phase
Frequency: 50/60 Hertz
Current: 13,6-13,9 Ampere
Power: 5.5 kiloWatt

Figure 3.3 Electric Resistance Furnace

3.3.2.3 Grinder-Polisher

This tool can be used for grinding and polishing before testing to other method so that the surface is smooth and clean from impurities. But for polishing itself is done manually so that the surface is really smooth and flat.

Specification :
Brand: Metkon
Type: Gripo 300-1V
Made: Turkey Voltage: 220
Volts Frequency: 50 Hertz
Power: 550 Watts

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 3.5connected 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 1000x to 2500x according to the needs of the observer.

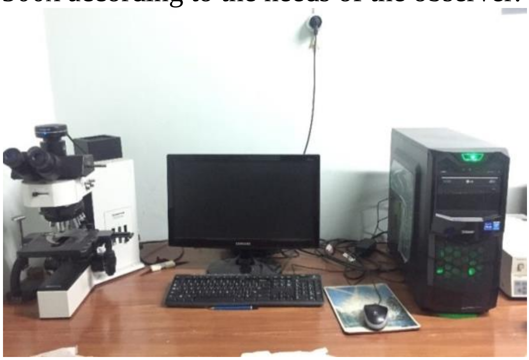


Figure 3.5 Optical Microscope

3.3.2.5 X-Ray Diffraction (XRD)

X-Ray diffraction or XRD is used to identify the substrane formed in the hard layers of FeNB an FeV. XRD testing is done in Material and Metallurgy engineering department FTIRS ITS Surabaya.

3.3.2.6 Microvickers Hardness Tester

This test is carried out to prove that the test sample that has been carried out by the hard coating process with Thermo Reactive Diffusion has a harder surface than the test sample that has not been carried out the process.

Data collection was carried out in the Department of Mechanical Engineering of FTIRS ITS Surabaya with indenter microvickers. The test was carried out on the hard layer area of Niobium Carbide formed, the interface layer area and the base metal area with each of the 5 data averaged for the hardness value of each region.

Specification :

Brand: Shimadzu Corporation
Model: HMV-2T
Production Number: 344-04152-12
Serial Number: I63033800469
Made: Japan
Power: 100 VAC, 50-60 Hertz, 300 VA

Figure 3.6 Microvickers Hardness Tester

3.4 Procedure of Conducting Research

The following is the procedure for conducting research evaluating niobium-vanadium carbide coatings on AISI 420 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 420) using wire cut. The dimension is 3.5 mm for the length 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.% NH_4Cl, and 75 wt.% alumina. Put the substrate inside them.

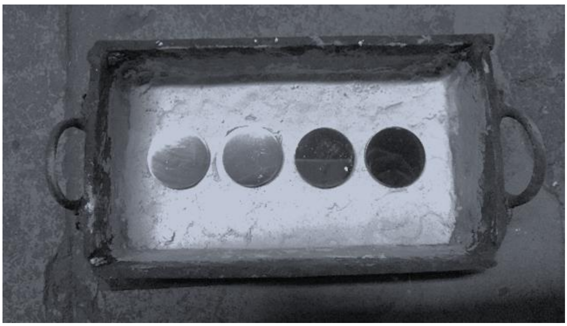


Figure 3.7 Inside the Reaction Pack

- TRD treatment on test pieces according to the desired holding time 2 hours, 4 hours, and 6 hours with temperature of each holding time is 1000°C.

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, and microstructure using optical microscope.
- Analyze test results.
- Make conclusions from the analysis

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.% NH4Cl and 75 wt.% Al2O3. This research will explore the effect of holding time variation of the TRD process onto vanadium-niobium coating formation on the surface of AISI 420. The samples were covered with powder mixture and then heated in the electric resistance furnace at holding time 2 hours, 4 hours, and 6 hours at 1000^{o}C. 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 coating chemical composition.

4.2 Chemical Composition Result

Testing og chemical composition was carried out using the electron spectrometer GVM-514S owned by PT. BARATA INDONESIA. the following is table 5 which is the result of spectometer test for AISI 420 :

Table 4.1 Chemical composition result

Chemical composition	Spectrometer result (%wt)	ASTM F899 standard(%wt)
C	0.35	0.16 (*min*)
Mn	0.59	1.00 (*max*)
P	0.019	0.040 (*max*)
S	0.003	0.030 (*max*)
Si	1.24	1.00 (*max*)
Cr	13.808	12.0-14.0
Mo	0.314	-

N	-	-
Ni	0.31	1.00 (*max*)

From the results of the chemical composition test obtained showed that the chemical composition of stainless steel AISI 420 is better than the ASTM F899 standard due to silicon content of more than 0.24 wt% of the standard which is a maximum of 1.00 and has molybdenum content of 0.314 wt%. This affects the mechanical properties of the material because the levels of silicon and molybdenum present in the material can increase the strength and hardness of AISI 420.

4.3. X-Ray Diffraction Test

X-ray Diffractionn testing was carried out to determine the chemical composition of treated specimens. Testing uses XRD with 2Ɵ varying from 10° to 80°. the following is the result of XRD Pattern for the treated specimen.

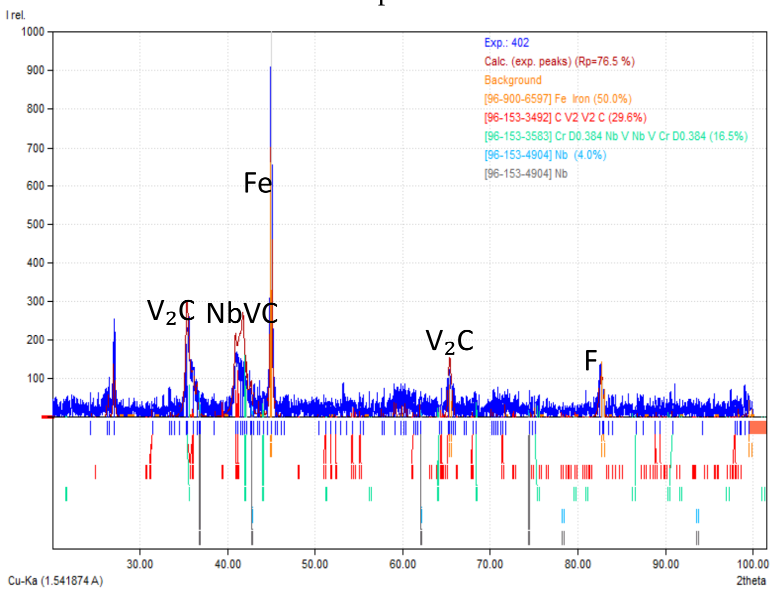
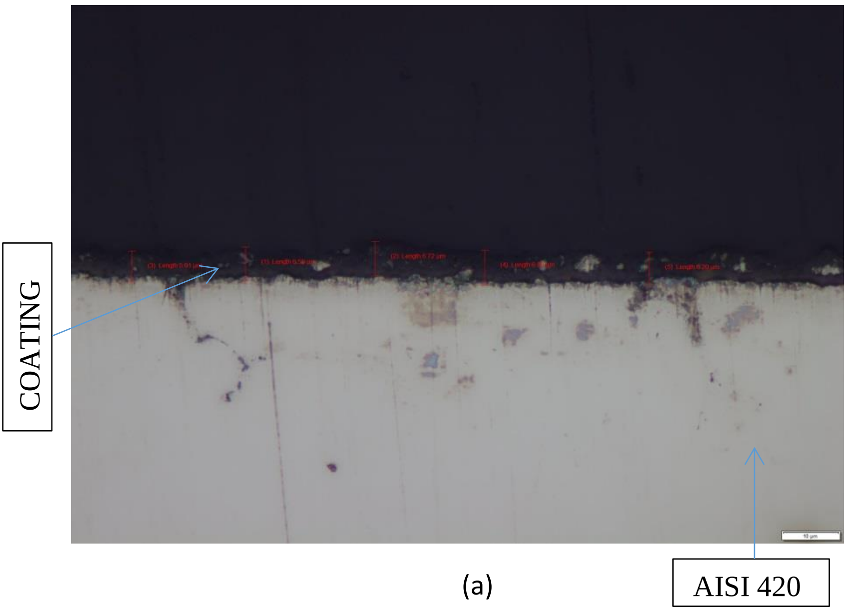


Figure 4.1 XRD pattern of holding time analyse at match!3 software

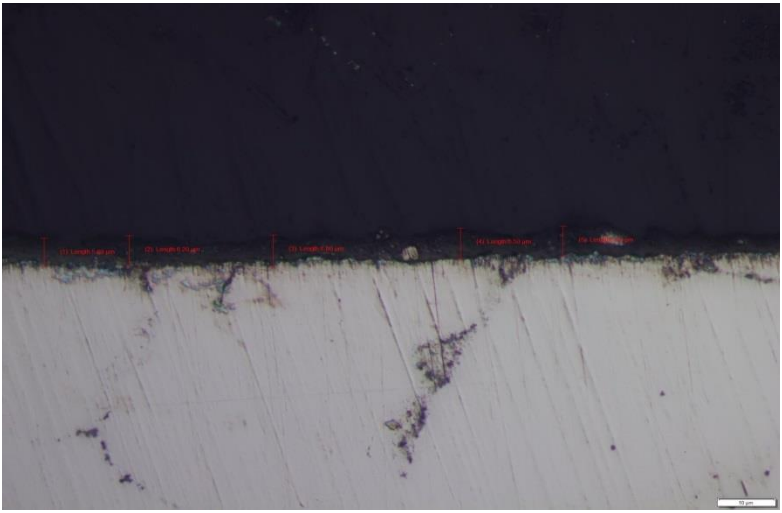
Data from the results of x-ray testing carried out in the Material and Metalllurgy department, then analyzed using match!3 software to find out what chemical substances were obtained from the test results. From the graph in theFigure 4.1shows that the x-ray diffraction test proves that there is a reaction of NbVC and V2C elements. The test results data prove that the coating formation that occurs is the result of the reaction of the elements niobium and vanadium.

4.4 Thickness Test Result

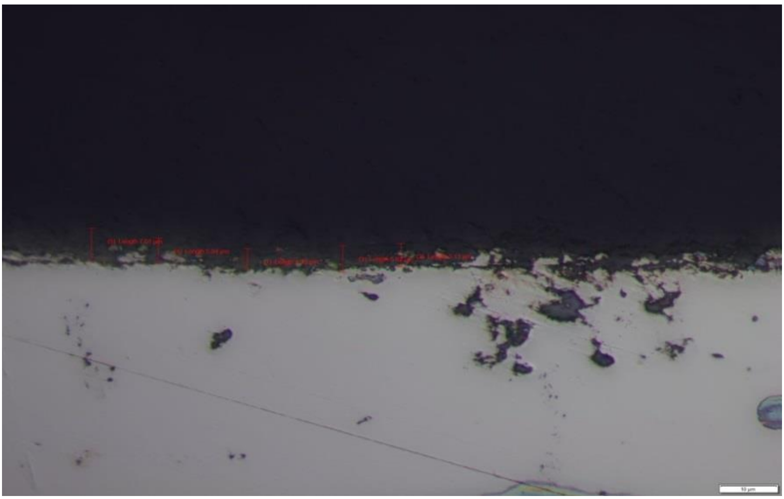
Observation of the coating thickness results from the TRD process was carried out using an Olympus optical microscope with 200x,500x,and 1000x magnification. The specimens prepared for observation are coating area NbVC as a result of TRD with variations at holding time 2 hour, 4 hour, and 6 hour at 1000°C. The following are the results of observations on each specimen.



(a)



(b)



(c)

Figure 4.2 Coating thickness observation with 500x magnification. (a) 6 hours holding time TRD process, (b) 4 hours holding time TRD process, (c) 2 hours holding time TRD process

The XRD results show that there is niobium vanadium carbide in aAisi 420, so it can be concluded that the layer formed around the speciment is a niobium vanadium carbide coating as shown in the Figure 4.2 above. The formation of a coating layer around the specimen has a difference in thickness, especially in each variation, therefore to calculate the thickness it will be seen from the average thickness value of several points taken, with a total of 15 points measuring the thickness of each variation as shown in the table below.

Table 4.2 Thickness value of TRD porcess

point	2 hours	4 hours	6 hours
1	4.452730429	6.496606692	5.693655303
2	5.109690657	6.715593434	6.204624369
3	5.620659722	5.912642045	6.861584596
4	5.036695076	6.861584596	6.496606692
5	7.007575758	6.204624369	6.423611111
6	5.474668561	8.759469697	7.445549242
7	7.737531566	6.35061553	6.35061553
8	5.620659722	6.861584596	6.715593434
9	6.204624369	6.788589015	6.204624369
10	5.182686237	5.912642045	5.912642045
11	7.883522727	5.109690657	7.299558081
12	6.35061553	5.182686237	8.029513889
13	3.941761364	5.620659722	7.810527146
14	3.065814394	5.255681818	8.175505051
15	2.627840909	5.109690657	8.759469697
Average	5.421138468	6.209490741	6.958912037
Standard Deviation	1.51855539	0.96187597	0.914909377

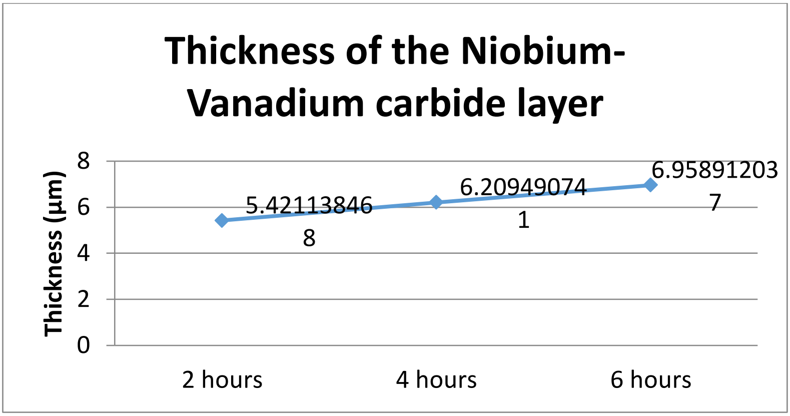
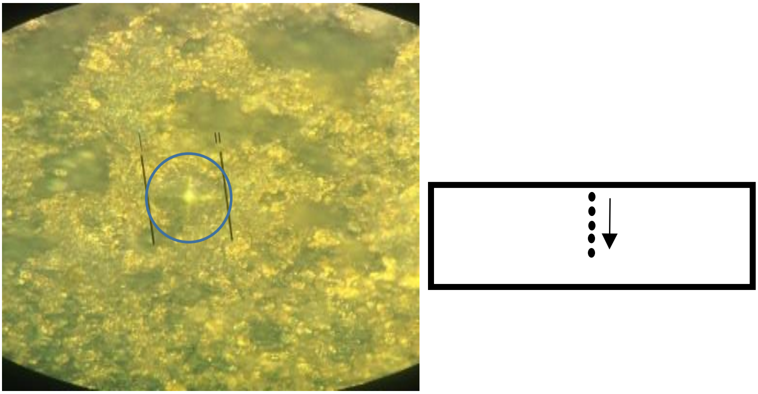


Figure 4.3 average thickness of the carbide layer coating

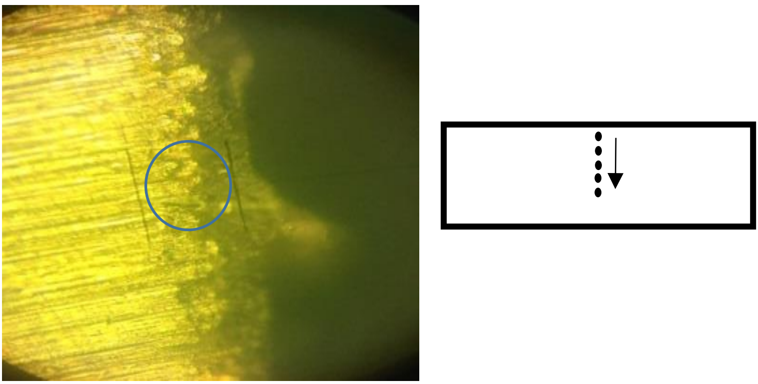
From the table above, it can be seen the thickness value of each variation carried out, namely the variation of holding time at 2 hours 4 hours and 6 hours. It can be seen that the highest thickness is in the holding time of 6 hours with an average value 6.95 µm followed by the holding time of 4 hours with average thickness value 6.20 µm, and finally the holding time of 2 hours with average thickness value 5.42 µm.. From the results above, it can be seen the effect of the length of time holding on the thickness of the coating where with a longer time the coating will be thicker due to the diffusion process that effect the binding time of the carbide forming element that occurs in the powder pack when heat treatment is carried out which has a longer time to bind.

4.5 Hardness Test Result

Micro-Vickers hardness testing was carried out on all specimens with a load of 0.5 HV in the carbide layer formed on the side of the test specimen and in the center of the specimen which did undergo TRD testing 10 times on each specimen. an example of microhardness testing on AISI 420 can be seen in the Figure 4.4. An example of data obtained from a Micro-Vickers hardness testing machine is shown in the Table 4.3 and Figure 4.5 shows the average results of the AISI 420 Micro-Vickers hardness test.



(a)



(b)

Figure 4.4 (a) and (b) example of indentation result of speciment with TRD treatment with 2 hour, 4 hour, and 6 hour holding time variation

Table 4.3 Micro Vickers Hardness result

Point	Hardness value (HV)			
	Non	TRD 2 hours	TRD 4 hours	TRD 6 hours
1	498	1009	987	1147
2	501	1102	1103	1223
3	543	890	856	1298
4	505	901	1043	1320
5	554	1103	925	1301
6	563	900	974	1487
7	547	1092	1036	1389
8	569	805	1156	1352
9	560	763	925	1404
10	546	1203	906	1411
average	538.6	976.8	991.1	1333.2
Standard Deviation	26.97818	145.86127	93.66898	98.574056

From the data above, it is found that the greatest hardness value in the untreated specimen is 569 HV with an average 528.6 HV. The greatest hardness value obtained in the specimens treated with a hold time of 2 hours is 1203 HV with an average value of 976.8 HV. on specimens with a holding time of 4 hours had the greatest value 1156 HV with an average value 991.1 HV. The greatest hardness value on the specimen with a holding time of 6 was 1487 HV with an average 1333.2 HV. The comparison of the average hardness values between each variation can be seen in the **Figure 4.5**

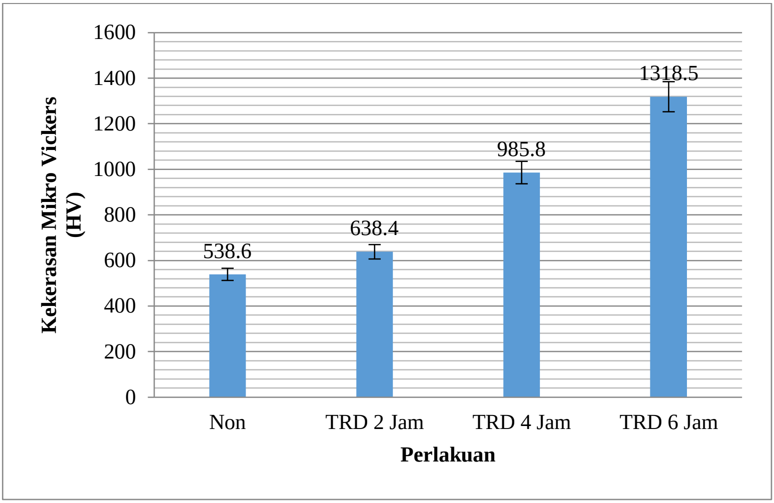


Figure 4.5 average value of micro hardness result

The untreated specimen has the lowest hardness value compared to the hardness value of the treated specimen. The results above show that the TRD process has the highest value of hardness with a holding time of 6 hours followed by the hardness value of the holding time of 4 hours and finally the hardness value of the holding time of 2 hours. in theory, it shows in general coating formation processing that, the longer the holding time, the hardest the carbide layer is formed, and in this research, it does show conformity to the theory with the highest hardness values found at the longest holding time and the trend shown there was no decrease in the mean of hardness value of the niobium-vanadium carbide formed. the longer the holding time in the TRD process itsels also effect the homogenity and density fro the carbide layer that is form because it will have the longer time to bind the CFE or niobium and vanadium to the speciment so the homogenity and density will be higher at the 6 hours than at 4 hours or 2 hours, therefore the hardness value will be higher at the longer time.

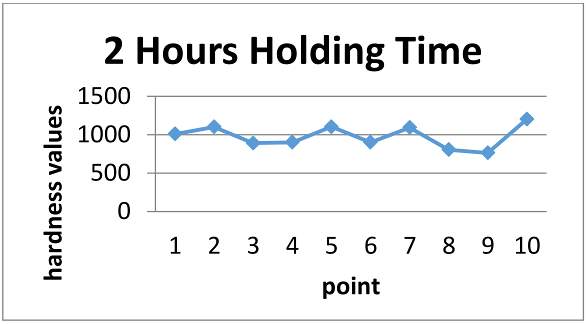


Figure 4.6 Hardness value for 2 hours of holding time

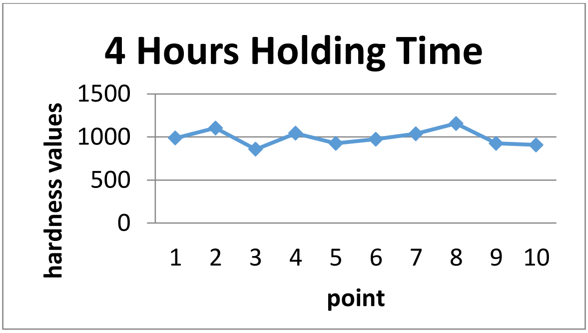


Figure 4.7 Hardness value for 4 hours of holding time

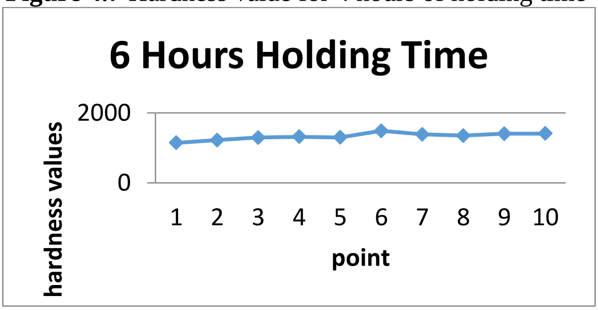


Figure 4.8 Hardness value for 6 hours of holding time

The image above shows the rising and falling trends of each indentation point taken in the Micro-Vickers hardness test carried out on the treated specimen. the difference in hardness values at each point is influenced by the presence of a depleted zone or a carbon-poor area at the indentation point taken. The amount of powder mixture also affects the precense of the depleted zone or a carbon-poor area obtained, the percentage of powder mixture used in this research is 10% nb 10% v 5% NH_4Cl and 75% Al_2O_3 with a content of 222 grams of Al_2O_3, 29.6266 grams of niobium, 29.6266 of vanadium, and 14.813 of NH_4Cl with the grade as stated, with more of the defleted zone, the homogenity and density of the coating will be low therefore at 2 hours holding the hardness value trend that is show more unstable than the 6 hours holding time because with high homogenity the hardness value at each point taken will be close amount at each other. The hardness value for each specimen is as shown in the table and if the grade is multiplied it will affect the hardnessand homogenity obtained with the same material.

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CHAPTER V
CONCLUSION AND RECOMMENDATION

5.1 Conclusion

From the research conducted, the conclusion can be drawn regarding the effect of the holding time on the formation of niobium-vanadium carbide coating onto AISI 420 by Thermo Reactive Diffusion process as follow :

1. The thickness of the layer of Niobium-Vanadium carbide (NbVC) formed on the surface of the specimen is affected by the increased of the holding time given Thermo Reactive Diffusion process with the result of the holding time of 6 hours with an average thickness value of 6.95 µm followed by the holding time of 4 hours with average thickness value 6.20 µm, and finally the holding time of 2 hours with average thickness value 5.42 µm.
2. Longer holding time resulted in higher hardness as observed in 2, 4 and 6 hours providing 976.8 $HV_{0.5}$, 991.1 HV, 1333.2 HV respectively. This value is more than double of uncoated sample which had hardness of 528.6 HV.

5.2 Recommendation

After doing research, a number of suggestion are obtain in order to get more comprehensive result in future reasearch, namely :

1. Thermo Reactive Difussion research in needed with variation of holding time between 7 hour and above in order to get most optimal time to do the process.
2. To get a better value of hardness andcoating thicness, the composition of the mixed powder in a similar study needs to be added so that the more formed niobium-vanadium carbide layer.

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