# MECHANICAL PROPERTIES OF HIGH VELOCITY OXY FUEL THERMAL SPRAYS COATING.

By

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Dissertation submitted in partial fulfillment of the requirements of the Bachelor of Engineering (Hons) (Petroleum Engineering)

JANUARY 2015

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### **CERTIFICATION OF APPROVAL**

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A project dissertation submitted to the Petroleum Engineering Program Universiti Teknologi PETRONAS In partial fulfillment of the requirement for the BACHELOR OF ENGINEERING (Hons) (PETROLEUM ENGINEERING).

Approved by,

(Dr. SUBHASH KAMAL)

# UNIVERSITI TEKNOLOGI PETRONAS TRONOH, PERAK JANUARY 2015

### **CERTIFICATION OF ORIGINALITY**

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

(ANAS MA)

#### ABSTARCT

The microstructure and mechanical properties of High Velocity Oxy-Fuel (HVOF) sprayed  $Cr_3C_2$ -NiCr alloy coatings deposited on austenitic stainless steel AISI 304 were investigated. The splat morphology of the coatings is due to the deposition and re-solidification of successive molten or semi molten powder particles. The morphologies of the coatings were characterized by using the techniques such as optical microscopy, X-ray diffraction (XRD) and field emission scanning electron microscopy/energy-dispersive analysis. Average microhardness of the coatings at different loads were found to be in the range of 222.8–523 HV, respectively.

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## ABBREVIATION AND NOMENCLATURES

С	Carbon.
Cr	Chromium
F	Fluorine
HVOF	High velocity oxy fuel
Ni	Nickel
NLPM	Normal liter per minute
SEM	Scanning Electron Microscopy

C <sup>o</sup>	Degree Celsius
mm	millimeter
μm	micrometer
gf	gram force
HV	Hardness Value

#### **CHAPTER 1**

### **INTRODUCTION**

#### 1.1 Background of Study

Nowadays, with a variety of applications, mechanical components have to operate under tough conditions such as in jet turbine, pump impeller blades, high speed vehicles, coal and biomass fired boiler tubes and turbines blades and vanes operating in desert environments etc., where they are all subjected to solid particle wear. Thus from that surface modification is needed to protect them against several types of degradation. In order to protect and counteract them from wear problem there are few method, which are avoid the object contact from water and oxygen. Reduced the high-flow velocities by control the fluid velocity and in this study we are focused on coating the object by thermal spraying.

The small solid particles which carried by a flowing gas impacting the loss of material from the surface of a metal due to a sequence of mechanical actions which is called Wear, Erosion and corrosion.

Major advantages of thermal spray process are its extremely wide variety of materials that can be used to produce the coatings as well as its ability to produce the coating on the substrate without any significant heat input. In most cases, thermal spray is used due to its special ability to strip off and recoat the damaged or deteriorating coatings without making any changes to the dimensions or properties of the coated materials.

#### **1.2 Problem Statement**

Wear, erosion and corrosion is the process parameters involved influenced the mechanism of wear, erosion and corrosion. The major influences of the mechanism of wear, erosion and corrosion are properties of impacting materials, target materials and environment. In engineering applications they encountered degradation of metal due to solid/semisolid/irregular shape particle wear, erosion and corrosion, at room temperature. Many peoples do a research and aimed to develop the coating in order to decrease the wear erosion and corrosion problems.

Therefore, this project has been selected the thermal spray coating by high velocity oxy fuel (HVOF) process and conduct studies on micro-hardness by using Vickers Hardness test at different loads to investigated the Micro-hardness value and surface morphology of the specimens by Scanning Electrons Microscopy (SEM).

#### **1.3 Objectives**

- To study mechanical properties of thermal spray coating by High- Velocity Oxyfuel (HVOF) process.
- To carry out and analyze the surface morphology of austenitic stainless steel AISI 304 coating of Cr<sub>3</sub>C<sub>2</sub>-NiCr powder using a scanning electron microscopy (SEM).
- To carry out and analyze the micro-hardness at different loads by Vickers Micro-hardness tester.

#### 1.4 Scope of Study

The scope of this study is focused on the mechanical properties, micro-hardness and surface morphology of coated specimens. And study the coating with  $Cr_3C_2$ -NiCr powder specimen by high velocity oxy fuel (HVOF) process and using Vickers Hardness tester, Scanning Electron Microscopy (SEM) to analyze the micro-hardness and surface morphology of specimens.

#### **CHAPTER 2**

#### LITERATURE RIVEW

#### 2.1 Thermal spray coating

Thermal spray is widely used as wear, erosion and corrosion protective coatings for several types of industrial applicants which had been acknowledged as a suitable and applicable surface engineering technology.  $Cr_3C_2$  – NiCr has higher resistance of corrosion and oxidation as well having high melting point and maintaining high hardness, strength and wear resistance reach to highest temperature of 900°C. (Sukhpal, 2012).

There are many coating technique applied and select the best depend on the function requirement such as size, shape, and substrate. The coating process can be divided into 2, metallic and non-metallic, these materials are deposited in the form of molten and semimolten to provide condition for coating. (International Thermal Spray Association, N.D.).

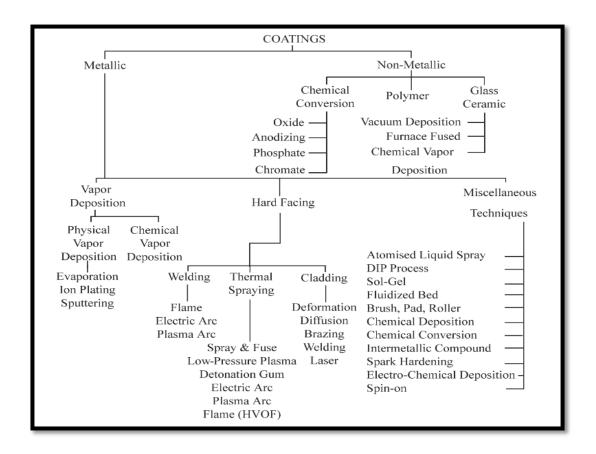


Figure 2.1: The coating technologies (Bhushan & Gupta, 1991).

The thermal spray coating is a method that produces varied range for various applications (Herman, 1996) hence there is a low application cost, less environment issues when compared to the others coating. Thermal spray coating technology is often considered (Singh, 2005).

Table 2.1: Comparison	n various type of therma	l spray processes.( Robert,
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1994).

		Feed	Surface		trate rature	Particle velocity		
Process	Materials	material	preparation	°C	°F	m/s	ft/s	
Powder flame spray	Metallic, ceramic, and fusible coatings	Powder	Grit blasting or rough threading	105-160	225-325	65-130	200-400	
Wire flame spray	Metallic coatings	Wire	Grit blasting or rough threading	95-135	200-275	230-295	700-900	
Ceramic rod spray	Ceramic and cermet coatings	Rod	Grit blasting	95-135	200-275	260-360	800-1100	
Two-wire electric-arc	Metallic coatings	Wire	Grit blasting or rough threading	50-120	125-250	240	800	
Nontransferred arc plasma	Metallic, ceramic, plastics, and compounds	Powder	Grit blasting or rough threading	95-120	200-250	240-560	800-1850	
High-velocity oxyfuel	Metallic, cermet, some ceramic	Powder	Grit blasting	95-150	225-300	100-550	325-1800	
Detonation gun	Metallic, cermet, and ceramic	Powder	Grit blasting or as-machined	95-150	225-300	730-790	2400-2600	
Super D-Gun	Metallic, cermet, and ceramic	Powder	Grit blasting or as-machined	95-150	225-300	850-1000	2800-3300	
Transferred arc plasma	Metallic fusible coatings	Powder	Light grit blasting or chemical cleaning	Fuses base metal	Fuses base metal	490	1600	

Proces	2222	Energy sources	Different Nomenclature
FIGUE	5505	Energy sources	Different Nomenciature
Low energy process	Flame spraying	Chemical	Oxyfuel gas-powder spraying
			Oxyfuel gas-wire spraying
			metallizing
	Arc spraying	Electrical	Electric arc spraying
			Twin-wire arc spraying
			metallizing
High energy process	Plasma spraying	Electrical	Atmospheric plasma spraying
			Vacuum plasma spraying
			Low pressure plasma spraying
			Water stabilized plasma
			spraying
			Inductive plasma spraying
	Detonation flame spraying	Chemical	D-gun
	High velocity	Chemical	HVOF spraying
	oxyfuel spraying		High velocity oxygen fuel
			spraying
			High velocity flame spraying
			High velocity air fuel

### Table 2.2 : The thermal spray process (Ajit,2012).

The coating substance could be in the form of powder, rod, wire or molten particle and it depend on its functional and application.

These processes are basically differentiated from each other on the basis of particle speed, flame temperature and spray atmosphere (Singh et al, 2007).

In order to understand the mechanism in thermal spray coating, The figure 2.2 shows the typical thermal spray process and coating.

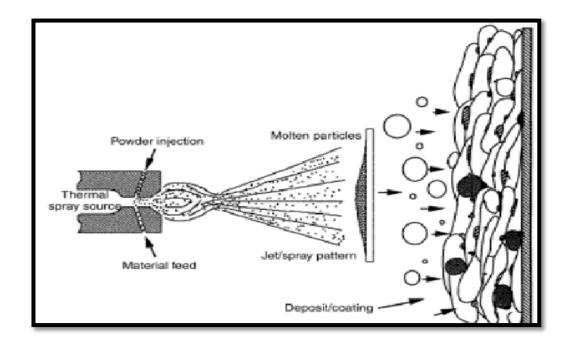


Figure 2.2 : Typical thermal spray process and coating. (Sidhu, 2012).

#### 2.2 High Velocity Oxy Fuel Thermal Spray.

The HVOF process is under the thermal spraying coating and can produced with lower porosity, lower frame temprature, higher hardness and higher jet velocity when compared to other process. (Wang&Shui, 2002).

The advantages of using HVOF process are higher density with lower porosity, improved particle bonding, improved wear and erosion resistance, lower surface oxidation due to less time in particle exposure and lower ultimate particle temperature where the others such as plasma, arc gun and HVOF with 16000 °C, 6000 °C and 3000°C respectively. (Stokes, 2005). And also stated that HVOF coating process is best quality than others standard plasma spraying process as we can see the figure 2.3 below. Where I is Hardness, II is Porosity, III is Oxide content, IV is Bond strength and V is Thickness.

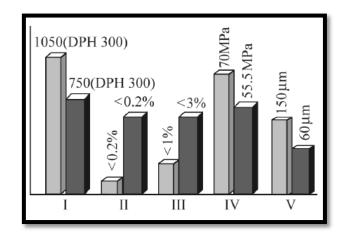


Figure 2.3: Characteristics of the HVOF and standard plasma-process coatings (Stokes,2005).

According to International Thermal Spray Association, The HVOF process apply a mixture of oxygen and fuel gases which are hydrogen,propane,propylene and kerosene.Product of burning are extended and expelled away over an orifice at high velocity in the comcustion chamber.Shock diamond offen mixture where they exiting the spray gun as shows in figure 2.4 below.

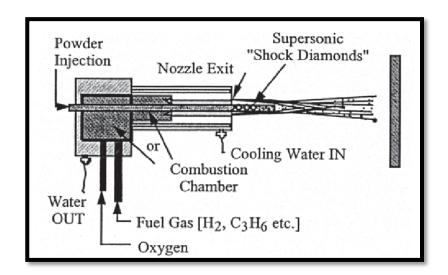


Figure 2.4 : HVOF thermal spray (graphic courtesy of Deloro Stellite Company,Inc).

#### 2.3 Scanning Electron Microscopy (SEM).

Regarding to the objective of this project, To analyze the surface morphology of eroded sample, the material characterization will be analyzed via the Scanning Electron Microscopy (SEM). Its uses a focused beam of high –energy eclectrons to produced a vary of signals at the surface of solid samples and to determine the erosion mechanism (Sukhpal,2012).

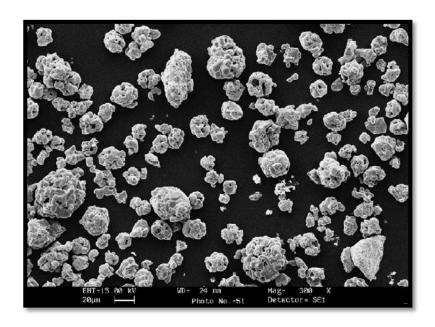


Figure 2.5 : SEM of Cr<sub>3</sub>C<sub>2</sub>–NiCr coating powder.(Kamal,2007).

The figure 2.5 shows the coating lower of  $Cr_3C_2$ -NiCr where we can see the size averaging from 5µm to 40µm by using SEM.

#### 2.4 Cermet Cr<sub>3</sub>C<sub>2</sub>-NiCr.

Cermet is a well-known class of materials that combine the positive properties of two groups of compound, ceramic (cer) and metals (met), being composed by a mixture of ceramic particles in a metal-matrix. It is ideally designed to have the optimal properties of both a ceramic, such as high temperature resistance and hardness, and those of a metal, such as the ability to experience plastic deformation. Furthermore, their high chemical stability makes them suitable to resist corrosion in aggressive solution/ condition.  $Cr_3C_2$ -NiCr has been proposed as one of the most suitable coating material to resist against wear in high temperature regime (800deg C).Although, it shows lower hardness than WC-Co, but it presents the advantage of a better corrosion resistance. Such a material is widely used in many industries, especially in chemical industry for its high capacity to resist a variety of corrosive media. With that capacity, it can give more advantages to industry and country in terms of economic.

#### 2.5 Wear

Wear characterization is one of the methods to identify the wear properties of a material surface. It is conducted to characterize the properties of one material while another surface is applied as the opposite surface. The term wear itself defines the interactions between two surfaces or more that causes the removal or deformation of surfaces material as a result of mechanical action on both surfaces. As a result of wear materials, the premature failure of engineering components can occur and affect a very high economic loss to industry and also to country.

The wear phenomenon can be found in our daily activities, such as in the piston cylinder components where the piston slides the cylinder which is called sliding wear and gears mechanism where the surface of gear tooth contacts each other which is also known as rolling contact.

In many mechanical components, the surface of material has been the critical region as it can be subjected to corrosion, oxidation, radiation, abrasion, or adhesion. In order to optimize the component and extend its life time, modification of surface properties requires to be performed. One of the most common methods to increase the surface properties of the component is by coating the base component with more suitable surface properties.



Figure 2.6 : Sliding wear of piston cylinder (acgo automotive corporation,2014)

A high wear-resistant material is one of the characteristics that are essential to many industries. Aircraft, petroleum, and machinery industry are the main industries that require a high-resistant material for their product. As such high-resistant material is used in the mechanical components, it can prolong the service life of the component, thus decreasing the running cost of the operation.

#### 2.5.1 Factors affecting wear

Numerous factors have been found to affect wear, such as those in shows in table below. And these can be grouped into material factors, conditions imposed by service, lubrication and environmental factors.

Table 2.3: Factors affecting the wear	

Temperature	Atmosphere
Load	Material Properties
Velocity	Lubrication
Contact area	Vibration
Shape	Sliding Distance

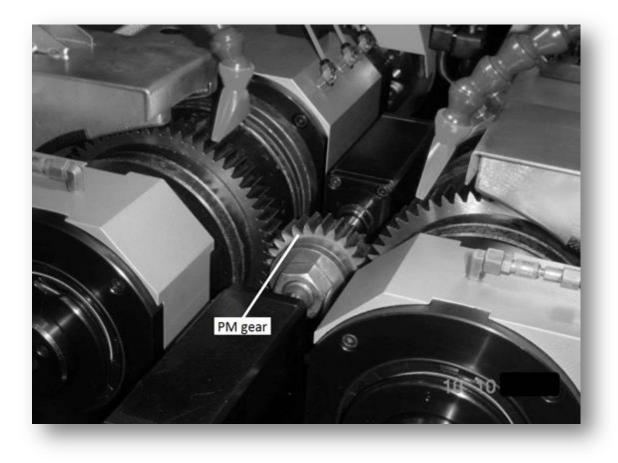


Figure 2.7 : Rolling contact in gear tooth ( Hagen Symposium, 2013)

### **CHAPTER 3**

### METHODOLOGY

### 3.1 Process Flow.

This methodology was scheduled and planned to ensure the activities perform well and smoothly. The process flow is very significant in order to reach the target within timeframe.

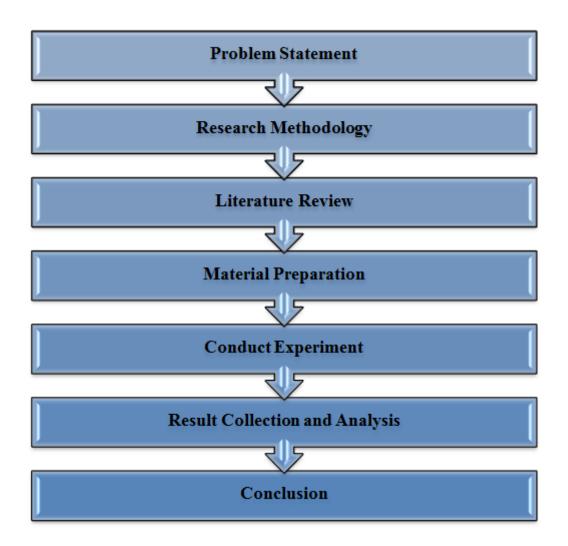


Figure 3.1: The sequence of work.

#### **3.1.1 Problem statement.**

The topic of the project was chosen by the student of final year and the supervisor was assigned. This step is focusing on statement and scope of the topic project.

#### 3.1.2 Research methodology.

At this step the topic of the project guided the way of the study. Then the more information that regarding to the project area will be learn and collected. All the information will be analyzed in the next step.

#### 3.1.3 Literature review.

At this step, the research will be done starting with literature review on the microstructure and wear characterization of HVOF thermal spray coating. Literature review will be study and analyzed through the related articles, journals, research papers, books and all other useful references.

#### 3.1.4 Material preparation

In order to conduct the coating and study the mechanical properties of HVOF thermal spray coating, a few specimens are conducted with parameters stated below in **table 3.1** 

Parameters	Details
Coating powder	Chromium carbide nickel chrome, (Cr <sub>3</sub> C <sub>2</sub> -25NiCr).
Substrate	Austenitic stainless steel AISI 304.
Deposition technique	High- Velocity Oxyfuel (HVOF).
Coating thickness	200 μm

### Table 3.1: Specimen fabrication parameters.

During the specimen fabrication process utilizing the spray parameters as listed in **Table 3.2**, the coatings thickness are constantly checked in order to obtain the range of thickness required in the study.

### Table 3.2: HVOF spray parameters

Parameters	Details
Fuel gas	CH4
Oxygen pressure (bar)	10.3
Fuel pressure (bar)	7.6
Air pressure (bar)	6.9
Oxygen flow rate, O <sub>2</sub> (NLPM)	279
Fuel flow rate, CH <sub>4</sub> (NLPM)	190
Air flow rate (NLPM)	361
Carrier gas flow rate, N <sub>2</sub> (NLPM)	12.5
Spray rate (g/min)	38 - 75
Spray distance (mm)	230
Deposit efficiency (%)	30

# 3.2 Project activities.



Figure 3.2: Cutting Specimens by Wire-Cutter.



Figure 3.3: 50x5mm AISI 304 specimens.



Figure 3.4: Specimen Sand Blasting.



Figure 3.5: Drying **Cr<sub>3</sub>C<sub>2</sub>-25NiCr powder**.



Figure 3.6: Coating by HVOF process.



Figure 3.7: Scanning Electron Microscopy.



Figure 3.8: Cutting specimen thru cross-section.



Figure 3.9: Vickers hardness testing.

## 3.3 Gannt chart and key milestone

## 3.3.1 Gannt chart and key milestone FYPI.

		Weeks												
Description of planning	1	2	3	4	5	6	7	8	9	10	11	12	13	14
Selection of Project Topic														
Preliminary Research Work														
Submission of Extended Proposal						*								
Proposal Defense														
Project work continues														
Preparation of the metal sample														
Preparation of chemical coating powder														
Submission of Interim Draft Report													*	
Submission of Interim Report														*

### Table 3.3: Gannt chart and key milestone for FYP I



\* Milestone

## 3.3.2 Gannt chart and key milestone FYPII.

	Weeks													
Description of planning		2	3	4	5	6	7	8	9	10	11	12	13	14
Project Work Continues														
Submission of Progress Report							*							
Project Work Continues														
Pre-SEDEX										*				
Submission of Draft Final Report											*			
Submission of Dissertation (soft bound)												*		
Submission of Technical Paper												*		
Viva													*	
Submission of Project Dissertation (Hard Bound)														*

## Table 3.4: Gannt chart and key milestone for FYP II

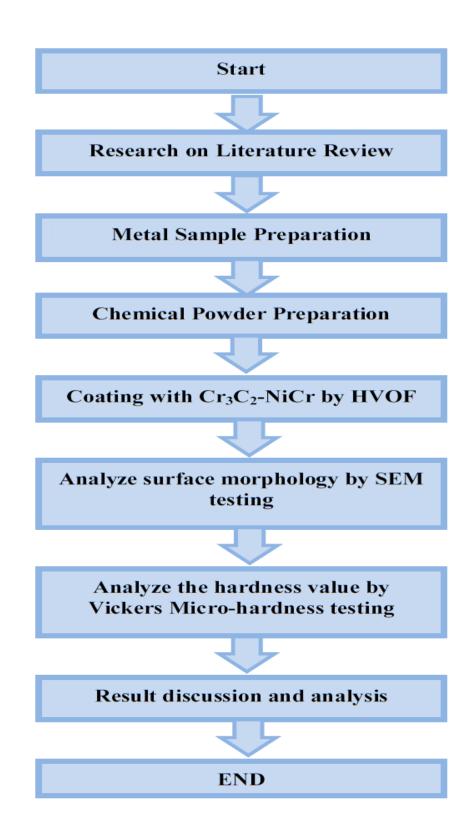


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Milestone

\*

3.4 Breakthrough Methodology.



#### **CHAPTER 4**

### **RESULTS AND DISCUSSION**

#### 4.1 Microhardness properties.

**Table 4.1** below recorded the hardness value of  $Cr_3C_2$ -NiCr coating at six different loads, which are 50 gf.,100 gf., 200 gf.,300 gf., 500 gf., and 1000 gf. Five readings are taken along cross-section of the coating thickness.

	MICRO-HARDNESS (HV)											
LOAD	1	2	3	4	5							
50	685.5	625.0	499.2	318.6	307.0							
100	397.1	381.1	290.2	243.8	241.2							
200	523.0	459.9	314.1	236.5	230.3							
300	389.4	350.9	286.6	248.6	231.4							
500	336.5	305.5	283.6	267.4	222.8							
1000	463.2	311.4	296.4	282.8	262.4							

### Table 4.1: Micro-hardness reading recorded throughout the cross section.

The recorded result were transferred to a graph as shown in **figure 4.1** where the hardness values result behaviour of the coating is can be clearly observed.

The  $Cr_3C_2$ -NiCr coating show great microhardness value of 685.5 HV at load of 50 gf. And can therefore be able to protect the substrate from external damage.

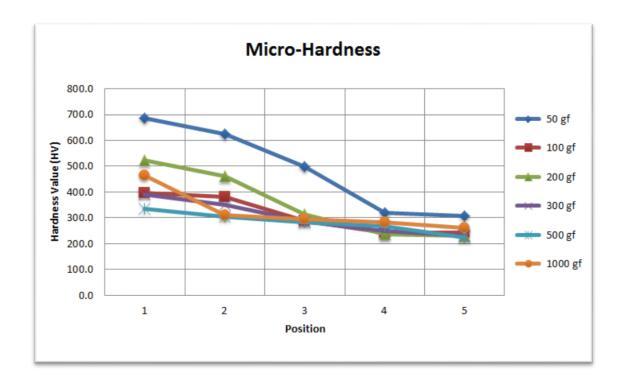


Figure 4.1: Microhardness Value across the coating cross section.

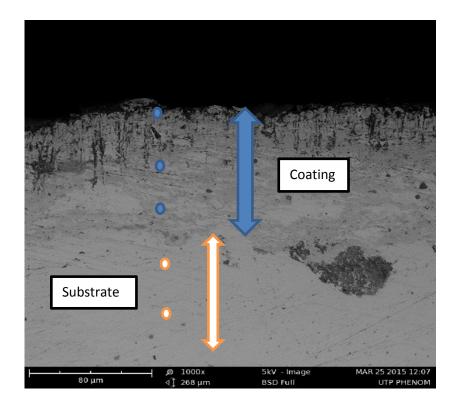
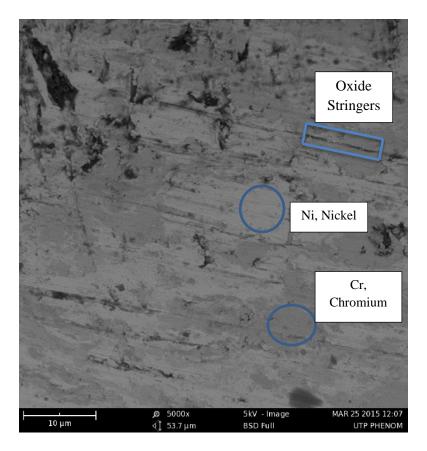


Figure 4.2: Position of the indentation made across the coating cross-section.

The hardness value of  $Cr_3C_2$ -NiCr coating obtained by HVOF process shows greater value than the other process of thermal spray coating. However, the result obtained in this project shows great correlation with studies by other authors also used HVOF process.

Position 1 at load of 50 gf. Shows a greatest hardness value along crosssection whereas the lowest hardness value along cross-section is at Position 5 at load of 500 gf. Therefore, the graph **Figure 4.1** shows slightly decreasing smoothly except at load 1000 gf. Due to the position indentation on Vickers Hardness and may be affected by the work hardening prior to HVOF process by the sand blasting work to surface of the substrate before coating.



#### 4.2 Microstructural Coating Overview.

Figure 4.3: Cross-section view of coating obtained by SEM at 5000x magnification.

The chromium carbide,  $Cr_3C_2$ -25NiCr coatings applied through HVOF process are uniform, dense and formed layered type of coatings. "Splats" are observed revealing the lamellar structure of the coatings. "Splats" is the term given to a single impacted particle in which many of the splats solidify and adhere to one another to form a continuous coating layer. This solidification and adhesion of the particles is what causes the lamellar structure to form and can be clearly viewed in the cross-section of the coating.

Oxide stringers, as shown in **Figure 4.3** are also observed constantly throughout the entire thickness of the  $Cr_3C_2$ -25NiCr coating. Oxide stringer is the observable dark, elongated line of phases that appears in the coatings parallel to the carbon steel substrate. These oxide stringers are produced through the interactions between the particles of the coating surfaces during the HVOF deposition process.

From **Figure 4.3** above, the dark grey area in the cross-section contains the element Carbon, C. and Chromium, Cr. So it can identified as the Chromium Carbide. Whereas the light grey area in the cross-section only contained Nickel, Ni. And chrome, Cr, which constitutes to the nickel chrome elements. Nickel chrome and chromium carbide did not fuse together but only melted and attached to each other. This phenomenon is what gives the dense and hard structure to the coatings.

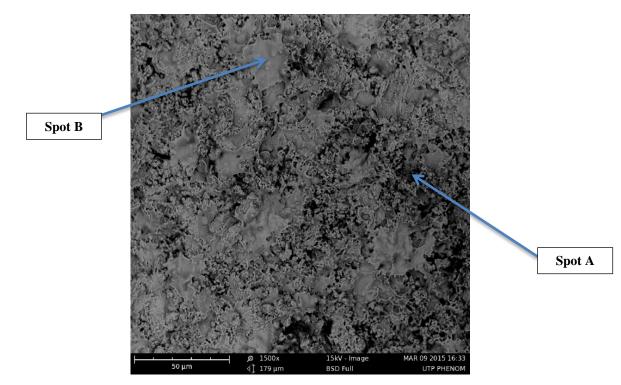


Figure 4.4: Element count across the coating mapped.

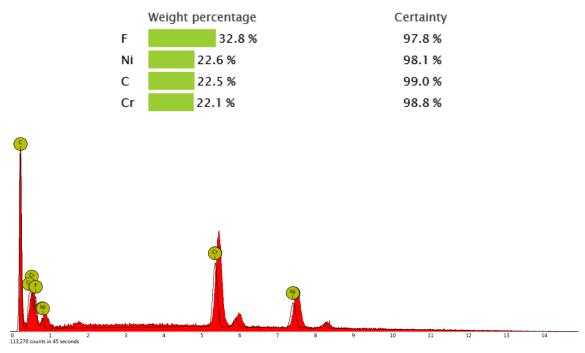


Table 4.2: the Cr<sub>3</sub>C<sub>2</sub>-25NiCr coating (Spot A).

Figure 4.5: the Cr<sub>3</sub>C<sub>2</sub>-25NiCr coating (Spot A).

Table 4.3: the Cr<sub>3</sub>C<sub>2</sub>-25NiCr coating (Spot B).

	Weight percentag	Certainty	
F		54.5 %	98.4 %
Cr	31.1 9	%	99.1 %
С	8.9 %		98.6 %
Ni	5.5 %		95.4 %

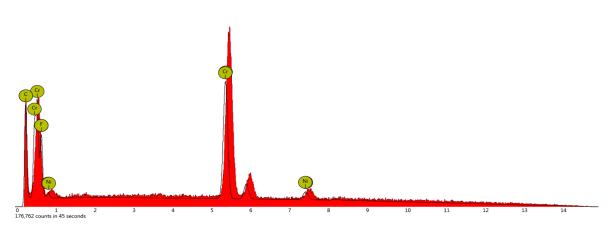


Figure 4.6: the Cr<sub>3</sub>C<sub>2</sub>-25NiCr coating (Spot B).

Element	Concentration (%)
Fluorine	43.65
Chromium	26.60
Carbon	15.7
Nickel	14.05

 Table 4.4: Percentage of element observed in coating.

From **Figure 4.4** above, shows the element that observed in the coatings from SEM. Which the Fluorine is major concentration composition (43.65 %) obtained. Followed by Chromium (26.60 %), Carbon (15.7%) and Nickel (14.05 %) respectively. As shown the percentage concentration in **Table 4.4** above.

The lighter grey (**Spot B**) area in the cross-section contains the element Carbon, C. Chromium, Cr. And a few element of Nickel, Ni. Whereas the dark grey (**Spot A**) area contain the element Carbon, C. Chromium, Cr. And Nickel, Ni, which constitutes to the nickel chrome element. Therefore this phenomenon is what gives the dense, hard structure and high hardness values to the coating.

# 4.3 Microstructure Specimen Overview.

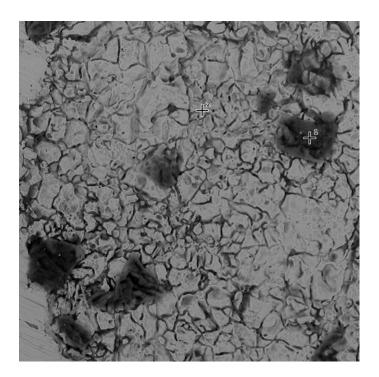
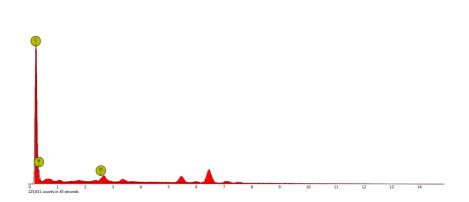
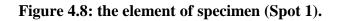


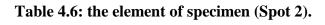
Figure 4.7: Element count across the mapped.



	Weight percentag	Certainty	
Ν		56.7 %	97.8 %
С	40.6 %		99.4 %
CI	2.7 %		96.5 %









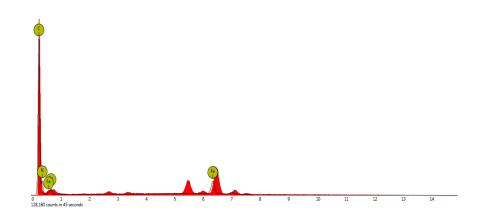


Figure 4.9: the element of specimen (Spot 2).

From **Figure 4.7** above, shows the element that observed in the specimen from SEM. Which the Nickel is major concentration composition obtained. Followed by Carbon, Iron and Chlorine. As shown the weight percentage concentration in **Table 4.5 and Table 4.6** above. Therefore this specimen is solid solution, with very low stacking fault energy and a high work hardening capability.

### CHAPTER 5

## CONCLUSION AND RECOMMENDATION

As in a conclusion, the author concludes that Wear Erosion and Corrosion problem is the most important issue in oil and gas industries and also in other factories. Therefore to overcome and counteract with this problem the author aim to study and do research regarding this problem in order to reduce the number of problem in industries.

From this study HVOF spray  $Cr_3C_2$ -25NiCr coating process shows great result in term of its mechanical properties, it shows better coating results compare to other type of thermal spray process such as Plasma spray, Detonation Gun and Flame sprayed. Based on literature reviews and the studies the conclusions are as follows:-

- HVOF technique is the best process when compare to others technique.
- HVOF technique is produces dense and lamellar structure of the coating.
- HVOF technique is produces better bounding, less degradation and economical.
- The Micro-hardness values of the coating obtained by Vickers Hardness are approximately Max at load 50 gf. And minimum at load 500 gf.

Recommendation on future studies on characterization and erosion-wear behavior at same coating powder and HVOF process. In addition for future recommended on this project is to conduct pin on disc tester and erosion test ring tester.

Objectives were achieved with the successful outcome of Micro-hardness values and studies surface morphology of the coating by HVOF process. The result of these studies will be helpful in finding the suitable process in surface engineering problem in oil and gas industries and also in other factories.

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### **APPENDIX**

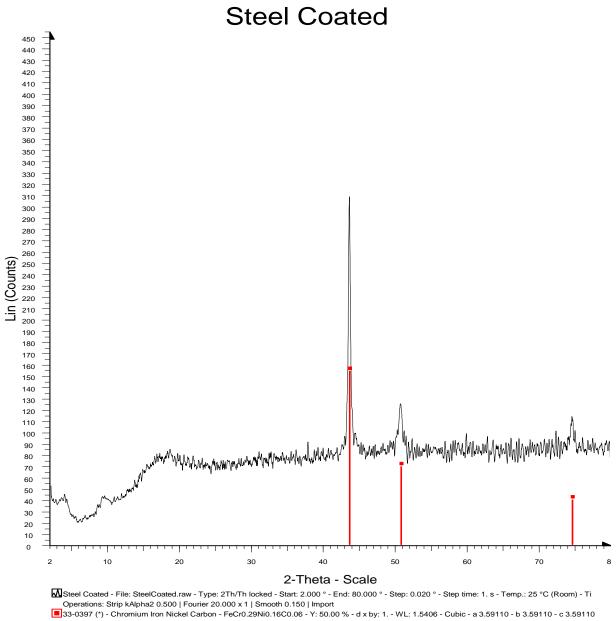


Figure A1: Coating obtained from XRD

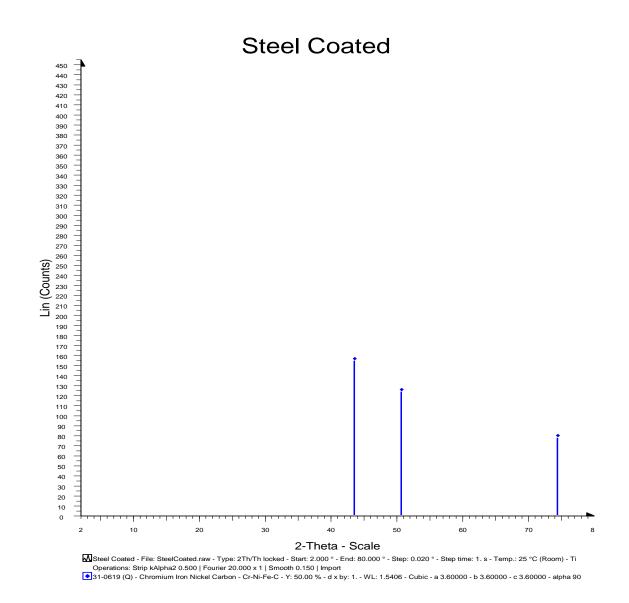


Figure A2: Coating obtained from XRD

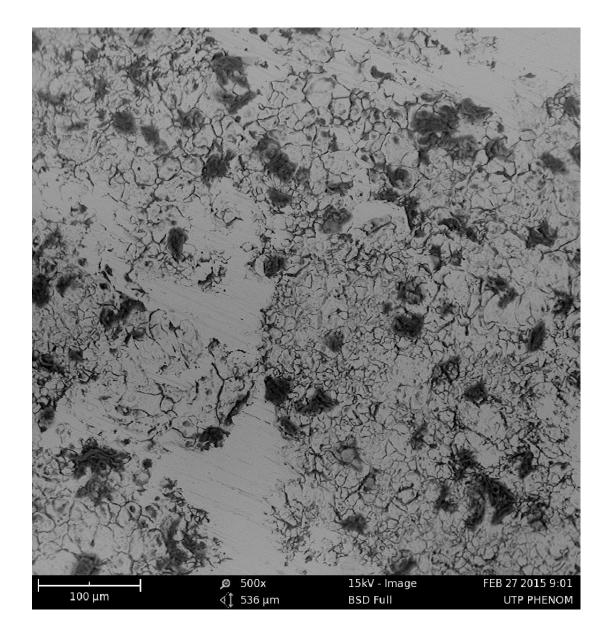


Figure A3: Sample metal Obtained by SEM at 500x magnification

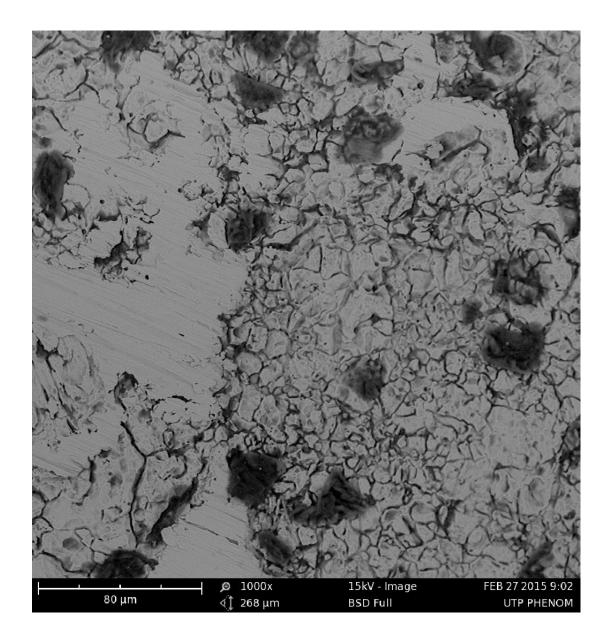


Figure A4: Sample metal obtained by SEM at 1000x magnification

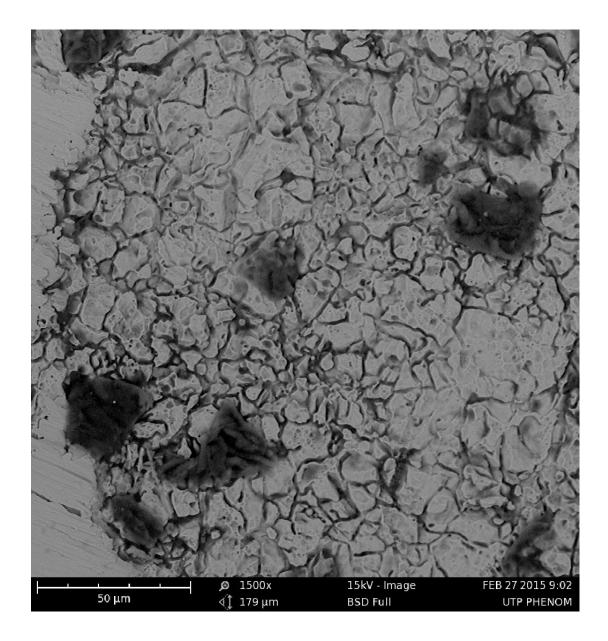


Figure A5: Sample metal obtained by SEM at 1500x magnification

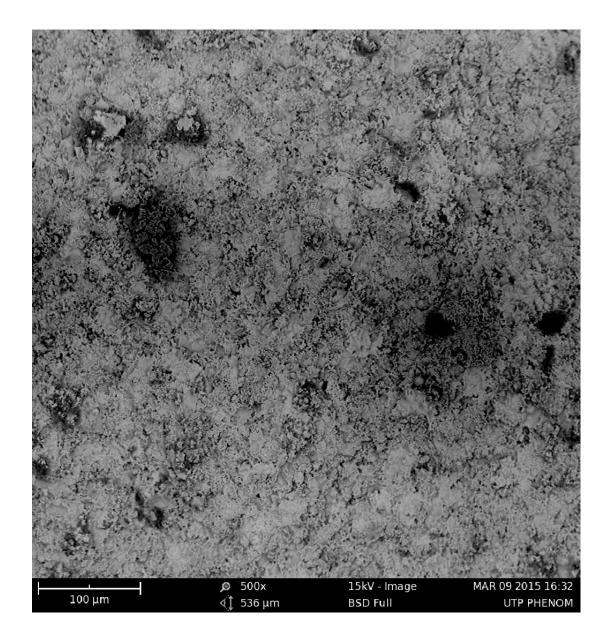


Figure A6: Cr<sub>3</sub>C<sub>2</sub>-25NiCr coating Obtained by SEM at 500x magnification

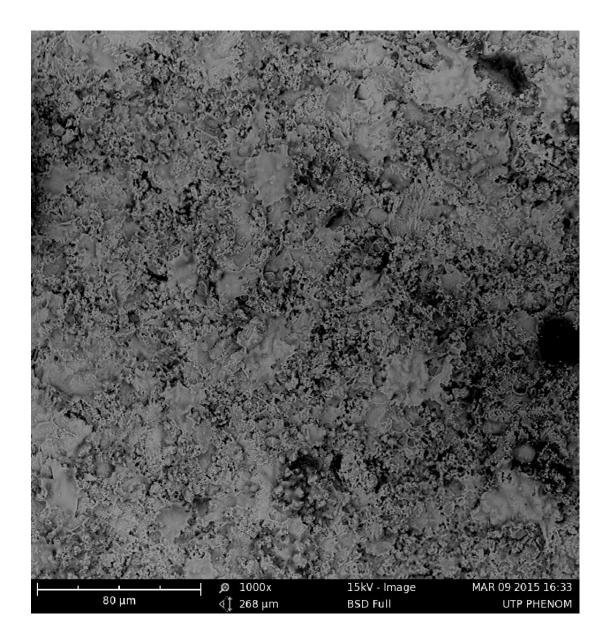


Figure A7: Cr<sub>3</sub>C<sub>2</sub>-25NiCr coating Obtained by SEM at 1000x magnification

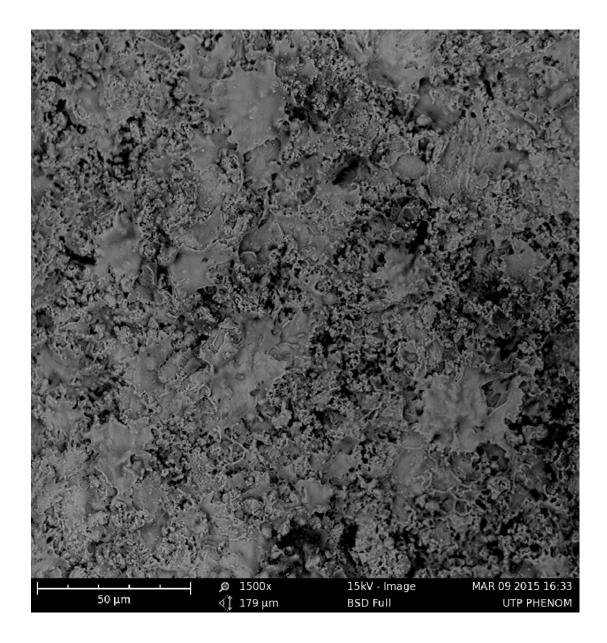


Figure A8: Cr<sub>3</sub>C<sub>2</sub>-25NiCr coating Obtained by SEM at 1500x magnification