A COMPARATIVE STUDY ON WEAR AND CORROSION BEHAVIOUR OF TUNGSTEN CARBIDE-NICKEL (WC-Ni) AND TUNGSTEN CARBIDE-COBALT (WC-Co) HIGH VELOCITY OXY-FUEL (HVOF) FOR CARBON STEEL BLADE

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The report submitted as partial fulfilment of the requirement for the award of the Degree of Master of Mechanical Engineering

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AUGUST 2017

Dedicated to my beloved parents, sisters, brothers and for those who have contributed to me in order for my completion of this thesis.

Thank you for the support and encouragement.

ACKNOWLEDGMENT

I would like to express my deepest appreciation to my project supervisor, Dr Zakiah Binti Kamdi, who has guided me with care. She has shown the attitude and the substance of a genius: excitement in regard to guidance and invested her full effort in guiding me to achieve the goal. Without her supervision and constant help this project would have not been possible.

Furthermore, a word acknowledges also goes to my co-supervisor, Dr Abdul Latif Bin Mohd Tobi for allowed me to experience master project under his supervision, which encouraging my research and for allowing me to grow as a research scientist. Your advice on both research as well as on my career have been priceless. Thank you for the ideas, encouragement, and understanding on my research project from Material Laboratory's assistant engineer (FKMP), Mr. Anuar and Mr Termizi. All instruction and guidance were much appreciated, which was allowing me to move forward.

I would also like to show my gratitude to the Tioxide (Malaysia) Sdn Bhd for sharing their knowledge and pearls of wisdom with us during the course of this research. This research was financially supported by Knowledge Transfer Programmed (KTP) project under grant no 1483.

Not to forget our parents, my families and friends to give their spirit and encouragement to me finish this final project until the end. Especially, to them given all the energy will be remembered forever.

ABSTRACT

Nowadays, the demand of high wear and corrosion resistance of the components in various industry is increasing from time to time. Therefore, high velocity oxy-fuel (HVOF) thermal spray was introduced to protect machine components from wear and corrosion, to restore worn components and to improve the durability of the components. HVOF is one of the process of depositing a material layer over a base metal or substrate with characteristics of high flame velocity and moderate temperature. The main purpose of this present study is to characterize the structure of the tungsten carbide 10 wt.% nickel (WC-10Ni) and tungsten carbide 12 wt.% w cobalt (WC-12Co) coating deposited by means of HVOF thermal spray onto a continuous digester (CD) blade that made up from carbon steel. The morphology and chemical composition of the coating were characterized by scanning electron microscope (SEM), electron dispersive spectrometer (EDS), and x-ray diffraction (XRD). The hardness test was carried out by using Vickers micro-hardness tester with load of 490.3 mN (0.05 HV). The wear and corrosion behavior and mechanism for both coatings was compared. Three body wear test was carried out in term of weight loss and electrochemical test was performed in acidic media (mixture of sulfuric acid, H_2SO_4 and ilmenite) to obtain the corrosion rate of the coating. From the result, it shows that WC-12Co coating has finer grain size that is around 2.3 µm. WC-12Co has higher wear resistance due to high volume friction, low mean free path, high hardness and lower porosity distribution compared to WC-10Ni. Besides, the formation of secondary phase, W_2C also affected the hardness of both coating, where this phase is harder than WC phase. For corrosion test, WC-12Co shows good corrosion resistance with small differences of corrison rate with WC-10Ni, that is only 0.7016 mm/y. As a conclusion, WC-12Co HVOF coating shows high potential on replacement of CD blade.

ABSTRAK

Pada masa kini, permintaan yang tinggi terhadap komponen yang memiliki rintangan haus dan kakisan dalam pelbagai industri semakin meningkat dari semasa ke semasa. Oleh itu, bahan bakar oksigen berhalaju tinggi (HVOF) diperkenalkan untuk melindungi komponen mesin dari haus dan kakisan, untuk memulihkan komponen yang rosak dan memperbaiki ketahanan komponen. HVOF adalah salah satu proses pengenapan saduran bahan ke atas logam asas atau substrat dengan ciri-ciri halaju nyalaan tinggi dan suhu sederhana. Tujuan utama kajian ini adalah untuk mencirikan struktur tungsten karbida 10 wt.% nikel (WC-10Ni) dan tungsten karbida 12 wt.% kobalt (WC-12Co) saduran yang diendap melalui semburan haba HVOF ke atas bilah cernaan berterusan (CD) yang diperbuat daripada keluli karbon. Morfologi dan komposisi kimia saduran dicirikan oleh mikroskop pengimbas elektron (SEM), spektrometer penyebaran elektron (EDS), dan pembelauan sinar x-ray (XRD). Ujian kekerasan dilakukan dengan menggunakan penguji kekerasan mikro Vickers dengan beban 490.3 mN (0.05 HV). Tingkah laku dan mekanisme haus dan kakisan telah dibandingkan bagi kedua-dua saduran. Haus tiga jasad telah dijalankan dalam bentuk kehilangan berat dan ujian elektrokimia dilakukan dalam media berasid (campuran asid sulfurik, H_2SO_4 dan ilmenit) untuk mendapatkan kadar kakisan saduran. Dari hasilnya, ia menunjukkan bahawa lapisan WC-12Co mempunyai saiz butir yang lebih halus iaitu sekitar 2.3 µm. WC-12Co didapati menghasilkan rintangan haus yang lebih tinggi disebabkan oleh isipadu geseran yang tinggi, laluan bebas purata yang rendah, kekerasan yang tinggi dan pengagihan keliangan yang lebih rendah berbanding dengan WC-10Ni. Selain itu, pembentukan fasa sekunder, W_2C menjejaskan kekerasan kedua-dua saduran, di mana fasa ini lebih keras daripada fasa WC. Untuk ujian kakisan, WC-12Co menunjukkan rintangan kakisan yang baik dengan perbezaan kadar kakisan yang rendah dengan WC-10Ni, iaitu hanya 0.7016 mm/y. Sebagai kesimpulan, saduran WC-12Co HVOF menunjukkan potensi tinggi sebagai penggantian bilah CD.

CONTENTS

TITLE PAGES	i
STATUS COMFIRMATION	ii
FRONT PAGE	iv
DEDICATION	v
ACKNOWLEDGMENT	vi
ABSTRACT	viii
ABSTRAK	ix
CONTENTS	x
LIST OF FIGURES	xiv
LIST OF TABLES	xviii
LIST OF ACRONYMS	xix
LIST OF APPENDICES	xx

CHAPTER 1 INTRODUCTION

1.1 Background of study 1

1.2 Problem statements 4

1.3 Objectives 6

1.4 Scopes of study 6

1.5 Significant of study 7

CHAPTER 2 LITERATURE REVIEW

2.1 Introduction 8

2.2 Hardfacing technique 9

2.2.1 Welding 11

2.2.2 Laser cladding 12

2.2.3 Thermal spraying 12

2.3 High velocity oxy-fuel (HVOF) 13

2.4 Tungsten carbide (WC) 16

2.4.1 Cemented carbide 17

2.4.1.1 Tungsten carbide-cobalt (WC-Co) 18

2.4.1.2 Tungsten carbide-nickel (WC-Ni) 20

2.5 Corrosion 21

2.5.1 Type of corrosion 22

2.5.1.1 Galvanic corrosion 22

2.5.1.2 Pitting corrosion 23

2.5.2 Electrochemical test 24

2.6 Wear 29

2.6.1 Wear behaviour and mechanism 30

2.6.2 Archard’s law 34

2.7 Phase identification analysis 35

CHAPTER 3 METHODOLOGY

3.1 Description of project 36

3.2 Selection of powder 38

3.3 Preparation before coating 39

3.4 Preparation of sample 43

3.4.1 Sectioning the blade 44

3.4.2 Cold mounting 45

3.4.3 Grinding 46

3.4.4 Polishing 46

3.5 Characterization analysis 47

3.5.1 Volume fraction and mean free path analysis 47

3.5.2 Grain size, thickness and microstructure analysis 48

3.5.3 Porosity analysis 49

3.5.4 Phase analysis 49

3.6 Microhardness analysis 50

3.7 Corrosion analysis 51

3.8 Wear analysis 55

3.9 Surface roughness analysis 58

CHAPTER 4 RESULT AND ANALYSIS

4.1 Introduction 60

4.2 Characterization of powder and coating 60

4.2.1 Microstructure analysis 61

4.2.2 Elemental composition analysis 63

4.2.3 Cross section and thickness analysis 64

4.2.4 Phase identification analysis 67

4.3 Porosity analysis 68

4.4 Microhardness analysis 70

4.5 Wear analysis 72

4.6 Corrosion test 76

CHAPTER 5 CONCLUSION

5.1 Conclusion 80

5.2 Recommendation 82

REFERENCES 84

APPENDIXS 94

LIST OF FIGURES

FIGURES		PAGES
1.1	Wear and corrosion area of the blade after three months' operation	2
2.1	Categories of thermal spraying	13
2.2	HVOF coating device	14
2.3	Common temperature with particle velocity for various thermal spray systems	15
2.4	Cross sectional SEM micrograph WC-12Co coating layers; a) low magnification, b) high magnification	19
2.5	Galvanic corrosion method	23
2.6	Pitting corrosion mechanism of carbon steel; a) low magnification of pit, b) high magnification of pit	24
2.7	Electrochemical test setup	25
2.8	Potentiodynamic polarization curves of the coating layers in the aerated 5 wt.% H_2SO_4 solution	26
2.9	Tafel curve; a) WC-9Ni and b) WC-6Co	27
2.10	SEM and BSED micrograph of surfaces of both coatings after saline corrosion test; (a), (b), (c) WC-Co; (d), (e), (f) WC-Co-Cr; (b) is magnified image of holes in black circles	27
2.11	Wear processes and their characteristics	29
2.12	Graph hardness vs. grain size	31

2.13	Hardness vs. abrasion rate in WC-Co composites	32
2.14	SEM micrograph of the worn surface; (a) WC-17Co; (b) WC-10Co-4Cr; (c) WC-12Co; (d) Cr_3C_2-25NiCr	33
2.15	XRD patterns of powders and corresponding HVOF coatings	35
3.1	Methodology flow chart	37
3.2	Carbon steel blade	39
3.3	Procedure for HVOF thermal spray	40
3.4	Grit blasting machine	41
3.5	The blade after the blasting	41
3.6	Blade after coating process	42
3.7	EDM wire-cut machine model Mitsubishi, Japan	44
3.8	The blade's sectioning position	45
3.9	Buehler grinder machine model BUEHLER, USA	46
3.10	Polisher machine model BUEHLER, USA	47
3.11	Optical microscope model Nikon, Japan	47
3.12	Scanning electron microscope model JEOL, Japan	48
3.13	Porosity analysis using image J software	49
3.14	X-ray diffraction machine model Bruker, USA	50
3.15	Vickers micro hardness model Shimadzu, Japan	51
3.16	Electrode set up	52
3.17	Corrosion potentiostat model WonaTech, Korea	54
3.18	The procedure for the wear test	55
3.19	Modified grinder machine model Metkon, Turkey	56
3.20	Surface roughness tester model Mitutoyo SJ-400, Japan	59

4.1	SEM micrographs of WC-10Ni powder; a) low magnification, b) high magnification	61
4.2	SEM micrographs of WC-12Co powder; a) low magnification, b) high magnification	61
4.3	Microstructure of WC-10Ni coating; (a) Secondary Image; (b) BSE image	62
4.4	Microstructure of WC-12Co coating; (a) Secondary Image; (b) BSE image	63
4.5	Microstructure of WC welding method	63
4.6	EDS Analysis for powder; (a) WC-10Ni; (b) WC-12Co	64
4.7	EDS Analysis for coating; (a) WC-10Ni; (b) WC-12Co	64
4.8	Cross sectional micrograph coating; (a) WC-10Ni; (b) WC-12Co	65
4.9	Sectioning carbon steel blade after coating	65
4.10	Average thickness for both coating	66
4.11	XRD analysis for WC-10Ni	68
4.12	XRD analysis for WC-12Co	68
4.13	The porosity of coating; (a) WC-10Ni; (b) WC-12Co	69
4.14	Average porosity for both coating	69
4.15	Indention of the (a) substrate; (b) coating	70
4.16	Graph hardness of the substrate and coating	70
4.17	Comparison of hardness and porosity of coating	71
4.18	Graph wear volume against sliding distance	73
4.19	Wear rate against hardness	73
4.20	Wear micrograph of WC-10Ni coating; (a) 1000 m; (b) 2000 m; (c) 3000 m; (d) 4000 m	75

4.21	Wear micrograph of WC-12Co coating; (a) 1000 m; (b) 2000 m; (c) 3000 m; (d) 4000 m	75
4.22	Tafel graph for WC-10Ni and WC-12Co coating	76
4.23	Surface micrograph after corrosion test of WC-10Ni coating; a) low magnification, b) high magnification	78
4.24	Surface micrograph after corrosion of WC-12Co coating; a) low magnification, b) high magnification	79

LIST OF TABLES

TABLES		PAGES
2.1	Common materials and purpose for hardfacing	10
2.2	Various processes for hardfacing technique	11
2.3	WC properties	17
2.4	Co properties	18
2.5	Properties of Ni	20
2.6	Coating characteristics	33
3.1	Chemical composition of powder	38
3.2	Characteristics WC-10Ni and WC-12Co powder	38
3.3	Chemical composition of medium carbon steel	39
3.4	Parameters of HVOF thermal spray	43
3.5	Parameters for electrochemical test	54
3.6	Specification of SiC	55
3.7	Moh's hardness	56
3.8	Times for sliding distance	57
4.1	Tafel curve average data for WC-10Ni and WC-12Co coating	77

LIST OF ACRONYMS

WC-10Ni	-	Tungsten carbide- 10 wt.% nickel
WC-12Co	-	Tungsten carbide- 12 wt.% cobalt
HVOF	-	High velocity oxy-fuel
CD	-	Continuous digester
SEM	-	Scanning electron microscope
EDS	-	Electron dispersive spectrometer
XRD	-	X-ray diffraction
WC	-	Tungsten carbide
Ni	-	Nickel
Co	-	Cobalt
SMAW	-	Shielded metal arc welding
EDM	-	Electrical discharge machining
W_2C	-	Tungsten dicarbide
W	-	Tungsten
SiC	-	Silicon carbide

LIST OF APPENDICES

APPENDIX		PAGES
Table A1	Data of weight loss and wear volume of WC-12Co	93
Table A2	Data of weight loss and wear volume of WC-10Ni	94
Figure A1	Surface Roughness Profile of WC-10Ni	95
Figure A2	Surface Roughness Profile of WC-12Co	96
Figure A3	Mapping analysis of WC-10Ni	97
Figure A4	Mapping analysis of WC-12Co	97
Table A3	Average thickness of WC-10Ni coating	98
Table A4	Average thickness of WC-12Co coating	100

CHAPTER 1

INTRODUCTION

1.1 Background of study

Nowadays, weld hardfacing had been evolved rapidly and are applied in various industries including nuclear, steam power plants, agriculture and railways, and even in aerospace components (Kirchgaßner *et al.*, 2008). These process not only capable to reduce the cost but also hold promise in improving the life service of machine parts by fabricating or rebuilding (Pradeep *et al.*, 2013). However, this hardfacing techniques also brings along together some crucial problems such as wear, erosion and corrosion which relating to the some application of hardfacing material and its composition of the chemical for a certain application (Shibe & Chawla, 2013). Wear and corrosion are worth investigating due to its problems represent a large portion of the costs, for example in oil and gas producing companies. Garcia *et al.* (2010) reported that the economic costs linked to the corrosion of natural gas sweetening (CO_2 corrosion) and oil refining plants range between 10% and 3% of the maintenance budget. While, in Canada alone, wear is estimated to cost $ 2.5 billion a year (Borle *et al.*, 2015). Engineering components such as in petrochemical, power and aerospace suffer surface degradation primarily due to wear. Both wear and corrosion problems are usually connected with operating problems and equipment maintenance, which leading to recurrent partial and even total process shutdown, resulting in severe economic losses.

Tioxide (Malaysia) is one of the Hunstman's pigments division that are the leading global producer of specialist titanium dioxide (TiO_2) pigments. This pigments primarily deliver whiteness, brightness and opacity to a vast range of products; form coatings and polymers to cosmetics and food. They also help to increase the longevity of products. However, during the TiO_2 manufacturing process, continuous digester (CD) blade that used to mixture sulphuric acid (H_2SO_4) and ilmenite exposed to wear and corrosion. CD blade was made up from medium carbon steel and coated with hard weld deposit (tungsten carbide, WC) based materials. The problem is due to the very corrosive (acid), erosive (ilmenite) environment and also operate at high temperature due to exothermic reaction. Thus, the blade use for the current setup can only last for about six months. Figure 1.1 shows the blade that wear and corrode after three months of operation. Therefore, the components need replacement which costs money and downtime of the equipment. Researchers have identified that by replacing the component frequently, it caused the working parameters of facility disorganize, productivity decreased and the energy consumption increased (Arsic *et. al.*, 2015). Moreover, wear and corrosion often combine to cause aggressive damage in a number of industries (Davis, 2014).

Figure 1.1: Wear and corrosion area of the blade after three months' operation

So, as modern method is quite effective in enhance the corrosion and wear resistance, the option is to change the hardfacing techniques from hard weld to thermal spraying process with WC-based materials. This technique is totally different from hard welding where the heated/melted materials are sprayed onto the surface at high velocity. The thermal sprayed cermet coatings (WC-based) are widely used to restrain the components from corrosion and wear in most industries such as aerospace, petroleum and steel metallurgy (Oksa *et al*., 2011).

High Velocity Oxy Fuel (HVOF) is widely used in groups of thermal spraying and have been extensively used for WC feedstock powder in order to obtain good bond strength, higher density and less decarburization. This is due to the lower temperature (3000 ℃) and higher velocities (400 m/s -1000 m/s) experienced by the powder particles (Lopez & Rams, 2015) compare to other thermal spray technique like vacuum/low pressure plasma (VPS/LPPS) and atmospheric plasma (APS). In order to achieve better wear and corrosion rate, adhesion and porosity of the coating are important properties. Thus, HVOF method are preferred for producing coating with high adhesion and low pores. The main feature of the HVOF method is it is able to produce dense coatings with small amount of degradation, oxidation of materials, and transformations of the phase due to short dwell time of the particles in a relatively cold flame (Oksa *et al*., 2011).

Apart from that, as a based material, WC has been thought of as one of a key factor in controlling wear and corrosion resistance. Content, size and distribution of the WC particles take decisive influence on the wear characteristics. Woezel *et al.* (2003) has reported the wear resistance increases with rising of WC content and reducing the carbide particle size of the powder. Over the years, WC have also proven their superiority in great number of other tooling and engineering applications due to their properties including high hardness, great abrasion or wear resistance, high modulus of elasticity, high compressive strength and low thermal expansion compared to other carbide (Woezel *et al.*, 2003).

Nowadays, cemented tungsten carbide has been introduced to improve the performance of the WC, where it consists of the hard carbide phase embedded in a ductile metallic matrix, referred to the binder phase such as cobalt (Co) and nickel (Ni). For this carbide, components properties are superimposed where the carbide phase produce high hardness and wear resistance. Meanwhile, the binder with ductile feature contributes to toughness and strength. It is often referred as hard metals for excellent combination of hardness and toughness (Su *et al.*, 2015). WC-Co based materials are preferred due to their superior abrasive and erosive wear resistance. Besides, their remarkable mechanical properties such as high hardness, excellent high temperature strength, good corrosion resistance and chemical and thermal stability during high temperature operations made them commercially very attractive (Saheb *et al.*, 2014). It has been reported that, these mechanical properties are depending on the development microstructure in the sintered parts which is governed by several factors like binder phase content, distribution of the carbide particle size, hardness and volume fraction of the carbide. Thus, nanostructure materials in the form of coatings have been popular to impart wear resistance of various industrial components (Zafar & Sharma, 2016). Other than that, with high corrosion resistance, nickel has received the most attention as an alternative binder to cobalt due to its similarity in structure and properties (Tarragó *et al*., 2015). There are several reason choose nickel as cement for carbides, for example it has high melting point, no structural defects, high resistance to corosion and oxidation, high hardness and strength (Miranda, 2004). Due to these superior mechanical properties, WC-Ni is effective in applications requiring a highs resistance to corrosion.

As many coating enhancements have been reported by researchers, more understanding on the characteristics, wear and behaviour of the HVOF thermal spraying is needed, so that this technique could be commercialized for various industrial applications in the near future. In this research, the application of HVOF process coated with WC based material on the CD blade that was made up from carbon steel was analysed as a potential method.

1.2 Problem Statement

Many industries faced the common problems which is the surface of components were exposed to deterioration, wear and corrosion. For example, in automotive industry, a substantial proportion of the disk brake rotors that is fabricated from gray cast iron with its relatively low cost and desirable properties such as low melting point and good fluidity.

However, due to their poor resistance to wear and corrosion under severe operating conditions, gray cast irons are unlikely to be satisfactory rotor materials without a different alloy system or a coating as potential material for friction system (Samur & Demir, 2012).

As in Hunstman Tioxide industry, an issue of wear and corrosion is also unavoidable. During manufacturing TiO_2 pigment process, there are two shafts with blades that are counter rotating to stir the mixture of ilminite and H_2SO_4 inside the continuous digester in order to produce raw TiO_2 pigment. The blade was fabricated from cast iron carbon steel and coated with hard weld deposit WC exposed to wear and corrosion. This problems is due to the environment during TiO_2 manufacturing process, where it is very corrosive (acid), erosive (ilmenite) and also it operated at high temperature due to the exothermic reaction (operating temperature around 135℃-150℃). Therefore, the lifetimes of the blades are short where the mean time between failure (MTBF) is only six months. The Hunstman spent so much expense to build and refurbishment the blades every time. Moreover, this industry is single line production base plant, where this industry forced to delay the production rate due to the downtime of the components.

Although, the surface of blade implemented by using hard facing (welding) method, the wear and corrosion is inevitable. Therefore, this study was designed to investigate the most favorable coatings material to increase wear and corrosion resistance, hence improve the performance of the blade by using another appropriate method, that is HVOF thermal spraying. This technique has been chosen due to its properties such as high density (low porosity) due to greater particle impact velocities, good bond strength and less decarburization, which leads to enhancement of wear and corrosion resistance. Other than method, based material is also important key in controlling wear and corrosion. With excellent combination of high hardness, high wear and corrosion resistance and high toughness, WC-Co and WC-Ni has good opportunity for replacement materials for the blade.

1.3 Objectives

The present study focuses on the influence of HVOF coating with cemented WC based material on the CD Blade. Therefore, the objectives of the research are:

I. To characterise different HVOF coating (WC-12Co and WC-10Ni) on potential replacement coating for continuous digester blade.

II. To compare the wear and corrosion on behaviour and mechanism for both WC-12Co and WC-10Ni coatings on continuous digester blade.

1.4 Scope of Study

a) Preparation of HVOF cermet WC based coatings:

I. CD blade (medium carbon steel) used as the samples. (CD blade from Hunstman).

II. Powders of WC-12Co and WC-10Ni was used as material based coating.

III. Aluminium oxide use to grit blast the sample before thermal spray.

IV. Diamond jet (sulzer metco) was used to spray the powder to the sample

V. Wire cut 0.20 mm needed for cutting the sample using electrical discharge machining (EDM) wire cut machine.

b) Samples characterisation:

I. Cross section and surface morphology of samples observed by using optical microscope (OM) and scanning electron microscope (SEM).

II. Elemental composition of the coating determined by energy dispersive spectrometer (EDS).

III. The phase of the coating identified by using x-ray diffraction (XRD) and analysed by EVA software.

c) Microhardness, wear and corrosion test:

I. Micro hardness of the coating and substrate tested by Vickers microhardness equipped with diamond pyramid indenter with loads of HV 0.05 (490.3 mN).

II. The wear of coating tested using weight loss method by modified grinder machine with SiC slurry.

III. Wear test carried out with load of 20N/mm in four different sliding distance that is 1000 m, 2000 m, 3000 m and 4000 m.

IV. Electrochemical test was used to test the corrosion behaviour using AC potensiostat/ galvanostat by ACM instruments in mixture of sulphuric acid (H_2SO_4) and ilmenite solutions.

V. Electrode that were used in electrochemical test are platinum (Pt) wire gauze as counter electrode, reference electrode by using silver/ silver chloride (Ag/AgCl) and working electrode (sample).

1.5 Significant of Study

The main purpose of this study are to enhance the wear and corrosion resistant of the CD blade, other than to increase the performance and production rate of the industry. The problem of wear and corrosion are caused by abrasion of blade and the reaction between the material and its surrounding. The environment in continuous digester is one of the factor of the blade to wear and corrode, where it stirs the mixture of sulfuric acid (corrosive) and ilmenite (erosive). Thus, the hardfacing HVOF coating used to improve the quality and performance of blade. At the end of the study, the result will benefit present needs of industry and significantly for further study.

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

Coatings has been evolved nowadays to protect the components against corrosion and wear from physical and chemical interaction with its environment. Wear and corrosion problems are relevance in a wide range of applications in industrial as they resulting in the degradation and eventually failure for both components and systems (Fauchais & Vardelle, 2012). Various technologies such as hardfacing used to deposit the appropriate surface protection that can resist under specific conditions, where it normally distinguished by coating thickness. As Shipway & Hutchings (1995) stated in their paper, the problem of coating damage generally relied on the coating material and its thickness, the interface properties, the material of the substrate and the test conditions.

The use of thermal sprayed coatings of wear and corrosion resistant to protect an underlying substrate has received much interest over the years. This is due to the expectation that very low porosity coatings can be prepared using the HVOF spraying process. This is a consequence of the higher particle velocities with the relatively lower particle temperatures obtained with HVOF compared to the other thermal spraying processes (Oksa *et al*., 2011).

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