

**STUDY OF FRICTION STIR WELDING TOOL'S COATING MATERIAL
(TiAIN) FOR TUNGSTEN CARBIDE COBALT**

By

Haikal bin Kamaruddin

24766

Dissertation submitted in partial fulfilment of
the requirement for the
Bachelor of Mechanical Engineering (Hons)

JANUARY 2020

Universiti Teknologi PETRONAS

32610 Seri Iskandar,

Perak Darul Ridzuan.

CERTIFICATE OF APPROVAL

**STUDY OF FRICTION STIR WELDING TOOL'S COATING MATERIAL
(TiAlN) FOR TUNGSTEN CARBIDE COBALT**

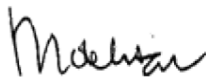
By

HAIKAL BIN KAMARUDDIN

24766

A project dissertation submitted to the
Mechanical Engineering Programme
Universiti Teknologi PETRONAS
in partial fulfilment of the requirement for the
BACHELOR OF ENGINEERING (Hons)
(MECHANICAL)

Approved by:

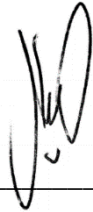


Ir. Dr Mokhtar Awang
Project Supervisor

UNIVERSITI TEKNOLOGI PETRONAS,
TRONOH, PERAK
JANUARY 2020

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 done by unspecified sources or persons.



HAIKAL BIN KAMARUDDIN

ABSTRACT

This project aims to compare and see the performance of the material of tungsten carbide cobalt (WC-Co) with a different coating (6%, 9% and 12%) with and without the coating (titanium aluminium nitrate (AlTiN) for friction stir welding (FSW). The proposed material uses two kinds of an experiment which is Vickers hardness test and scratch test data to compare and see the material performance. The higher the cobalt content, the higher the hardness of the materials. In the other hand, the adhesion of coating will decrease as the higher the cobalt content in the materials. The reason why the performance of the material needs to be compared because each of the material carries a different value of hardness and adhesion strength. In order to find a very strong material that can withstand sudden heat change and excessive friction, these materials need to be tested in term of strength and adhesion strength. The coating is essential because it can prolong the material life, and it can act as a lubricant to the friction stir welding tool. The strong the adhesion of the coating with the material, the longer the material life span.

ACKNOWLEDGEMENT

First and foremost, praised be to Allah, most merciful and the Most Beneficent, who grant the author to complete this research within the given time. I would like to give sincere appreciation to my project supervisor, Ir Dr Mokhtar Awang, who had to provide me with a lot of information regarding my FYP topic and also help me to deal with external party in a process to complete this project. Furthermore, I want to thank him for the time he spent with me on, commenting, evaluating and sharing valuable information throughout the project.

I also would like to thank my parent for their constant support mentally and physically to me in completing this project. Not forgetting Mr Amirul Amin Khalili, Postgraduate student of Universiti Teknologi PETRONAS (UTP) for his advice, discussion and guidance during this research. Also to Dr Sattar from Universiti Malaya for guiding me when I was conducting my experiment there.

I would like to give special thanks to all the staff in Universiti Teknologi PETRONAS and Universiti Malaya who give their full commitment and cooperation to assist me to operate the machine in the lab. Also, a lot of thanks to Mrs Amnani and my fellow friends who give me moral support throughout this project.

Last but not least, I would like to thank Mechanical Department of Universiti Teknologi PETRONAS, UTP for the opportunity to take part in this Final Year Project where a lot of experience and knowledge have gained throughout this project.

TABLE OF CONTENTS

CERTIFICATION OF APPROVAL	i
CERTIFICATION OF ORIGINALITY	ii
ABSTRACT	iii
ACKNOWLEDGEMENT	iv
LIST OF FIGURE	vii
LIST OF TABLE	viii
CHAPTER 1: BACKGROUD STUDY	
1.1 Background Study	1
1.2 Problem Statement	2
1.3 Objective	3
1.4 Scope of Study	3
CHAPTER 2: LITERATURE REVIEW	
2.1 Introduction	4
2.2 Development of FSW	4
2.3 Development of FSW tool	5
2.4 Modifying of FSW tool	5
2.5 Development of Tungsten Carbide as FSW Tool	8
2.5.1 FSW Tools	9

CHAPTER 3: METHODOLOGY

3.1 Process Flow of Methodology	11
3.2 Experiment Work	12
3.2.1 Material Preparation	12
3.2.2 Tools Preparation	13
3.3 Mechanical Testing	13
3.3.1 Hardness Test	13
3.3.2 Adhesion Test	15

CHAPTER 4: RESULT AND DISCUSSION

4.1 Introduction	17
4.2 Optical Microscope Result	19
4.3 Hardness Test Result	23
4.4 Scratch Test	26

CHAPTER 5: CONCLUSION AND RECOMMENDATION **29**

REFERENCES **30**

LIST OF FIGURES

- FIGURE 1: *Vickers Principle*
- FIGURE 2: *Example of Light Microscopic Micrographs of Scratches on Samples A and B*
With different material
- FIGURE 3: *Uncoated Rectangular Tungsten Carbide*
- FIGURE 4: *Coated Rectangular Tungsten Carbide*
- FIGURE 5: *Uncoated Cubic Tungsten Carbide*
- FIGURE 6: *Coated Cubic Tungsten Carbide*
- FIGURE 7: *surface of the WC-Co 6%*
- FIGURE 8: *surface of the WC-Co 6% TiAlN*
- FIGURE 9: *surface of the WC-Co 9%*
- FIGURE 10: *surface of the WC-Co 9% TiAlN*
- FIGURE 11: *surface of the WC-Co 12%*
- FIGURE 12: *surface of the WC-Co 12% TiAlN*
- FIGURE 13: *The Scratch on The Wc-Co 6% Material*
- FIGURE 14: *The Scratch on The Wc-Co 9% Material*
- FIGURE 15: *The Scratch on The Wc-Co 6% Material*

LIST OF TABLES

- Table 1: *Parameter of scratch test*
- Table 2: *Microhardness test result of WC-Co 6%*
- Table 3: *Microhardness test result of WC-Co 6% TiAlN*
- Table 4: *Microhardness test result of WC-Co 9%*
- Table 5: *Microhardness test result of WC-Co 9% TiAlN*
- Table 6: *Microhardness test result of WC-Co 12%*
- Table 7: *Microhardness test result of WC-Co 12% TiAlN*
- Table 8: *Scratch test result*

CHAPTER 1

INTRODUCTION

1.1 BACKGROUND STUDY

Friction stir welding (FSW) was introduced to the world in 1991 at The Welding Institute (TWI) in the UK. FSW is a new type of welding by using heat from friction to fuse two metal together. The rotation of the pin and shoulder of the FSW tool will heat the workpiece by friction; the heat of the friction will then bind the workpiece together [2] [3] [4] [5]. Initially, this type of welding is using soft material like aluminum as its base material. Because of this type of welding look promising, more study and research is made to increase the performance of FSW for high strength material [2] [3] [4] [5]. Because of that, a new type of material was introduced to make the FSW tool more durable and last long. One of the materials that are known for this type of welding is Tungsten Carbide (WC). It is a high strength material commonly used in FSW because it is durable and cheaper compare to the other high strength material. Because of that, to increase the strength for the material, Titanium Aluminum Nitrate (TiAlN) is added to the WC as a coating to act as a lubricant and to strengthen the material [3]. Besides TiAlN, Diamond-like carbon (DLC) also act as coating and the hardness be compared to the diamond. When compare between DLC and TiAlN, DLC is more durable and stronger than AlTiN plus can prolong the material life much longer. Another type of material that is used in FSW is Polycrystalline cubic boron nitride (pcBN) and one of the high strength materials that are used in FSW. This material is very strong and can be compared to diamond strength. However, when compared in high temperature, the hardness of pcBN is exceeding diamond [3]. Because of this characteristic, pcBN is one of the most preferred material in FSW but the downside with this material is the price is high. On top of that, the coating is an important aspect to FSW tools in order to keep the material strong, durable and lifelong. Chemical Vapour Deposition (CVD) is one of the coating processes where volatile precursor reacts to form a solid coating on a heated substrate where it transports by vapour phase at the reaction chamber. This process required high temperature and a lot of material can be deposited by using this method which is metal, sulphides oxide and carbide [6]. The reason why tungsten carbide is chosen in FSW because of the characteristic of this material,

which can withstand sudden change in temperature and loading during the welding process [7]. Also, WC can be mixed with other material to form stronger material like Co, Fe, and Ni.

1.2 PROBLEM STATEMENT

Friction stir welding is initially used in soft metal such as aluminium and its alloy. The performance of this type of welding is impressive from commercial welding. It is because the result of this type of welding is outstanding due to the surface of the welding process is nearly smooth. Also, the bond between the metal is stronger than the mainstream method. Because of that, many applications start using FSW to join the metal such as aircraft structure, production of train care bodies and production of a fuel tank barrel.

Metal matrix composites (MMC) comprise of metal alloy strengthened with ceramics. Metal matrix composites are widely used in many fields and have a high demand for aerospace applications because it has better mechanical properties than metal alloys. Conventional welding on MMC may be developed porosity and cracking [8]. The tool that is usually used in FSW for MMC is a steel tool, and it has some drawback.

The main drawback is that the current tool tends to undergo rapid and severe tool wear due to the contact between the tool and the stronger particle reinforcement [8]. It is vital to preserving the tool as the development of void could occur as the progressive wear of the tool removes the features that help the material to stir during the FSW process [8]. In order to decrease the wear rate, the material needs to be stronger than aluminium. In this project, the material that been chosen is tungsten carbide cobalt coated with titanium aluminium nitrate.

1.3 OBJECTIVE

- To study the relation between cobalt content and material hardness
- To investigate the adhesion strength of TiAlN coating on different substrates of cobalt content (6%, 9% and 12%)

1.4 SCOPE OF STUDY

The focus of this study is to compare the most suitable and durable cobalt content to the tungsten carbide (WC). This is because some content does not suitable with tungsten carbide, which will make the adhesion between them not last long and will make the tools wear faster. Tungsten carbide with a mixture of 6%, 9% and 12% cobalt content will undergo hardness test and adhesion test to see the durability of the material and also to the adhesion strength for each of the cobalt content. Also, the tools that will be used in this experiment is only a cube of WC-Co 6%, 9% and 12% and not the FSW probe/pin.

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

Many researchers have been experimented on Friction Stir Welding (FSW) tools with different material to see the performance of the material on high strength materials. Although there are many types of research on this topic, there is little knowledge of Tungsten Carbide Cobalt (WC-Co) with different content on FSW. This paper will focus on the performance of WC-Co with different content which is 6%, 9% and 12% and the adhesion strength of WC-Co with this cobalt.

2.2 Development of FSW

A FSW tool is one of the essential components in order to complete the process. The common tool usually consists of the round shoulder and cylindrical pin, which will rotate to create heat to the workpiece [8]. This heat produced from the friction will soften the workpiece; this softened material will move around to form a joint when it cooled. The advantage of FSW is that it can prevent the common problem in fusion welding, which is liquation cracking, solidification and loss of volatile alloying element [8]. This advantage is the reason why the widespread of FSW in soft alloy and aluminium. However, the FSW process is exposed to sudden change temperature and high stress when it comes to high strength material such as steel or titanium alloy that will limit the application of FSW to the commercial application because it will increase the cost and decrease the life span of the FSW tool.

2.3 Development of FSW tool

Initially, aluminium alloy is used in FSW before the high strength material is introduced. However, Aluminium matrix composite (AMC) is more durable than aluminium alloy when compared in term of wear resistance, fatigue life, temperature resistance and high strength to weight ratio. The other thing that makes the AMC is better than aluminium alloy is AMC has higher mechanical properties and because of that AMC have a higher demand in aerospace industry. According to Patthi et al. [9], FSW end product produce no visible defection the two plate are welded together as typical onion flow is observed after welding and there is no additional phases to show the retention of the material using the H13 steel tool. The welding process does not produce sufficient heat to cause phase changes, as there is no real proof of phase changes between the base metal and the welded zone. This study has shown that FSW can weld AMC perfectly without porosity and cracking.

2.4 Modifying of FSW tool

FSW tool have two main part which is shoulder; to generate heat and to prevent expulsion and pin; to penetrate into the material, stir and sweep the plasticised material with rotation [10]. Steel is commonly used in FSW tool, but it has very low resistance to wear after each welding. The wear rate is the major problem of FSW on Metal Matric Composite (MMC) as the tools steel experiences great wear rate when contact between the tool with the reinforce particle during welding. The wear rate is the crucial problem on MMC as the tool experience major wear rate due to contact between tool and base material during welding work. According to Ashish et al [11], the tool's wear rate is faster at the start of the FSW process because the material and tool is still cold and the weld forces are higher. The welding speed also play the part in Wear rate of the tool due to the maximum wear rate at the beginning of the welding process. The lower the speed the higher the tool's wear rate. When the material is hotter, the force needed is less and will decrease the wear rate. The wear rate of the material will be affected by 3 factors, rotation rate, transverse speed and distance welded. In order to promote the stirring of material and to reduce the likelihood of deformation, it is important to maintain the shape of the tool. Wear rate is the amount of material loss from the tool after welding the material. There are many methods of

determining the wear rate. Based on Prater et al [12], wear rate of the tool is measured by measuring the changes in the weights of the tool. After each welding pass, the mass of the tools will be measured to calculate the percentage of wear. The equation for finding the wear shown below:

$$\frac{m_i - m_f}{m_i} \times 100\% = \% \text{ wear}$$

Where m_i , is the initial mass and m_f is final mass.

This method of measuring the wear rate could be done as it will indicates clearly the percentage of wear of the tool. But the disadvantage of this measurement is the measuring machine must have small precision as it will affect the result of the experiment.

The second method in which the wear rate is determined by using the variation of the diameter. After welding the samples, the tools are put into 5% of sodium hydroxide (NaOH) to eliminate the aluminium that stick to the tools. Then the tools were photograph using digital camera mounted in the optical microscopic system. The wear rate of the tool is measure by equation below.

$$\text{variation} = \frac{d_o - d_f}{d_o} \times 100\%$$

$$\text{wear rate} = \frac{\text{variation}}{\text{travel distance}}$$

Whereby d_o is the initial diameter and d_f is the final diameter. This method needs to use optical microscopic system to get the images of each tool. Using this method, the location of the maximum wear rate could be identified easily as the minimum diameter is the part of the tool that experience highest wear rate.

The third method is by using imaging software develop by Prater [12]. After each welding, the probe was submerged into the solution of water and Sodium Hydroxide to remove the aluminium that stick to the probe. Then the images of the probe were imported into imaging software. Wear of the probe is calculated by comparing the pre-weld and post-weld images of the probe. The percentage tool loss is calculated by the reduction of the cross-sectional area. 1 cm square grid is used to determine the area of each image of the probe.

The fourth method is by comparing the tool image weight. Prado et al [13] calculates the wear rate by calculating total volume consumption. The nib was photographed vertical to the axis before and after welding. A series of photographs were obtained with a fixed magnification and the outline of each nib cut out of standard photographic printing. The cut-out print was weighted and compared to the rate of change from the initial tool nib. The percentage tool wear was obtained by calculating the loss based on the original projection of the tool. The percent tool lost is shown below:

$$\text{percent tool loss} = \frac{\text{original tool image weight} - \text{specific tool image weight}}{\text{original tool image weight}} \times 100\%$$

Among all the methods, the change of mass and area are the most practical method and these methods must be done correctly to prevent any error.

There are 4 methods to modify the FSW tools on MMC. Each of the alternatives is used to change the wear rate of current tools. In order to able to replace the current tools, tools material must achieve several characteristics in choosing the tool for FSW tool which include

- Wear resistance
- Has a good thermal fatigue strength when undergoing repeated thermal cycle
- No harmful effect to the base
- Has low thermal coefficient of thermal expansion
- At ambient and elevated temperature, it has good strength, dimensional stability and creep resistant
- To prevent damage during plunging and dwelling, it has good fracture toughness

One of the ways to improve the wear rate is by coating the tools. According to Contorno et al, AlSiTiN and AlSiCrN coating on steel tools were analysed to detect the changes in wear rate of the coated and uncoated tools in AMC in 20% of Silicon Carbide. The parameter for this study was 1000rpm, tool feed is 300mm/min and tilt angle of 2 degree. From this study, these coating made no difference of wear resistance of the tools as some parts of the coating was completely removed due to glue effect of the coating with the tools. The reduction of thickness can reduce glue effect of the coating of the tools. Meanwhile

Uzun[19] used TiAlN coating on Tool Steel to weld the AMC with 25% of Silicon Carbide, with rotational speed and travel speed were 800rpm and 120mm/min. In the EDX maps, there are only carbon silicon and aluminium material in the specimen which means that the coating is not even wear. Therefore, it is important to have the suitable coating and thickness in order to reduce the wear rate of the tool.

2.5 Development of Tungsten Carbide as FSW Tool

The development of tungsten carbide (WC) in FSW is still ongoing because of the characteristic of WC. The researcher such as Rai et al [8] and Selvam et al [3] found that the WC is not sensitive to sudden change in temperature and load during welding.

Choi et al [24] shows that the extreme wear had occurred at the tungsten carbide cobalt alloy tools between the edge and pin centre after welding on steel plate by using rotation speed is 1600 rpm and transverse speed is 15cm/min. In order to reduce the wear rate of the tool and produce a more last longer tool, the tool could possibly be initially shaped with a steady-state geometry. There are three potential mechanisms that cause the wear of the tools, oxidative wear of tungsten carbide, and a fatigue of the cobalt binder and a formation of a ternary W–Fe–O compound.

Liu et al [13] has studied tungsten carbide cobalt alloy tool on Al-Si alloy matrix and 30% volume of silicon carbide by using rotation speed is 2000rpm and 1500rpm, and transverse speed is 25-150mm/min. Liu discovered that the wear rate of the tool varies at each location and the minimum wear occurs in the upper half-part of the pin and maximum wear occur at the other half part near the shoulder. From this study, the maximum wear rate is occurred at the beginning of the welding. The welding rate is inversely proportional to the welding speed. The welding rate fluctuates along the travel distance of the welding. For the first 1.27m, the wear rate decreases as the travel distance increase and after 1.27m the wear rate increase as the travel distance increase.

Liu et al [25] has continued studied the tungsten carbide cobalt alloy tool on aluminium matrix composite with 30C% silicon carbide using constant welding rate at 2000rpm and increasing welding speed which is from 25mm/min to 150mm. Lie has concluded that all parameter can be used to produce a defect free joint. The welding speed has important effect on the weld joint variously and higher welding speed could increase the process of agglomeration of the silicon carbide which leads to inconsistence of deformation at the upper part and lower parts of the joint.

According to Batalha[26] by using tungsten tool coated with nitride (Al, Cr)N coating on titanium alloy at using the rotation speed is 1500, rpm and transverse speed is 50mm/min, the tungsten carbide tool had undergone wear and the coating was totally damage after the FSW processing. There is no residual trace of its components throughout the worn tool, so that the real causes of the coating layer could not be detected.

In this study, tungsten carbide cobalt (WC-Co) is coated with titanium aluminium nitrate (TiAlN) is used. Prater et al [12] claimed that the life of the tool can be increase by using the harder material in FSW. The higher the hardness ratio of the material, the wear rate will be decreased. As the material can be stopped from fractured, material is been coated with TiAlN can be effective in preventing the wear. This coating act as lubricant so that it will reduce the friction while FSW process is been in progress. For cemented carbide tools (WC-Co), micrograin tools have poor wear performance compared to submicrograin structure. It is because tinier particles can be worn more easily by abrasive action. Rough grains appear to resist the abrasive wear of FSW tools.

2.5.1 FSW Tools

Initially, FSW is made for the soft material like aluminium and soft alloy because of it soft and easy to weld by this method. When performance show by this method is impressive, FSW is move to the next level where it is now welding a high strength material. However, the problem with this material is that the tools use for FSW is wear drastically compared to aluminium and soft alloy. Because of that, selecting the right tools for FSW is one of the important things in welding for high strength materials.

The common tools that are used for high strength material are tool steel, W based tools and Polycrystalline cubic boron nitride (pcBN) tools.

I. Tools steel

Material such as magnesium alloy, brass and composites of the metal matrix are typically welded using steel devices. Lee et al. (2009) have discussed Al-Mg welded alloy with lower carbon steel in lap joint configuration using tool steel as a tool material without extreme wear by placing the softer alloy Al-Mg on top of the steel plate and avoid direct contact with the steel plate of the tool. The harder workpiece is often positioned on the advanced side in the butt joint configuration and the tool is offset slightly from the butt interface to the softer workpiece.

II. W based tools

Commercially, pure tungsten (cp-WC) is durable at high temperature, but at ambient temperature, it also has low toughness and wears faster when used as a medium for titanium alloys and FSW steel. Cp-W exposure to 1200 °C excess temperature can cause cp-W to recrystallise and break down at ambient temperature when cooled. Adding rhenium to it can reduce the transition from ductile to brittle. Tungsten carbide (WC) is very good because it can withstand up to 1650 HV and the material can also withstand sudden temperature changes. It is also one of FSW's favourite material that is easy to get and low cost. [3]

III. Polycrystalline cubic boron nitride (pcBN) tools

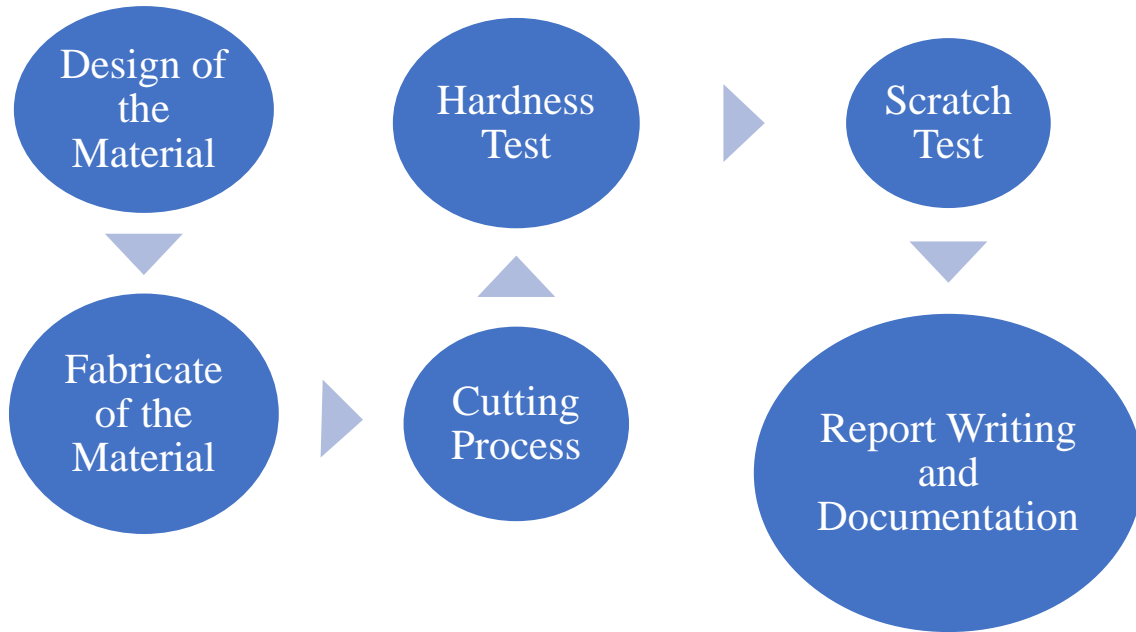
pcBN is durable because it can stand elevated temperature while remain the material strength and hardness and also have high-temperature stability. It is one of the preferred tool materials for FSW of a hard alloy such as Ti alloys and steels. Even with all the good characteristic, the downside of these materials is the cost is very expensive [3].

CHAPTER 3

METHODOLOGY

3.1 Process Flow of Methodology

The methodology of the Final Year Project is summarize in the form of flow process below:



The process flow of methodology start with the design of the material which is rectangular shape with the dimension of 8cm x 1cm x 1cm. This shape has been chosen because the main objective of this study is to study the hardness and adhesion strength of FSW material. With this shape, it will be easier to conduct an experiment for the material. The dimension for this material need to be done and finalize before send it for fabrication. After the dimension of the material is finalize, the drawing will be sent to Microcarbide Sdn Bhd to produce the material. After that, the rectangular material need to be cut cube size with dimension of 1cm x 1cm x 1cm. that is because the scratch test need a smaller size of material in order to conduct the testing. Then, mechanical testing were done to study the morphology of the material. The test include hardness test and scratch test. When the result is analyzed, further study for the experimentation need to be done by comparision of research paper. Last but not least, all report must be written and documented on every data that have been collected.

3.2 Experiment Work

Before starting this experiment, some preparations need to be done to ensure that the experiment is running smoothly.

3.2.1 Materials Preparation

For material preparation, the tools for this experiment is ordered from manufacturer due to lack of material provided in UTP and no specific equipment for fabrication of tools. Tools that are used in this experiment are WC-Co 6%, WC-Co 6% TiAlN, WC-Co 9%, WC-Co 9% TiAlN and WC-Co 12%, WC-Co 12% TiAlN. The first step for testing is the readiness of the material in order to execute the project plan smoothly and on time. This material is coated by using Physical Vapor Deposition (PVD) process. In this process, the material which is WC-Co was evaporated by bombardment with ions by sputtering process. At the same time, a reactive gas (gas that containing carbon) was added and formed a compound with the metal vapor where was deposited on the rectangular shape WC-Co as a thin coating. To get the uniform coating, the material needs to be rotate at constant speed with different axes.

3.2.2 Tools Preparation

The tests that need to be done on the material is the Vickers hardness test and friction/scratch test. Before the testing stated, the material are cut into cube size with dimension of 1cm x 1cm x 1cm. That is because some experiment like scratch test only fit smaller size of material. After cutting process is done, to use the Vickers equipment for hardness test, student need to book the equipment by using the UCS system. For scratch test, UTP did not have the machine for this test. The student need to find external resources in order to proceed with the testing. To ensure the experiment going smoothly, early preparation or booking needs to be done.

3.3 Mechanical Testing

During this study, some mechanical testing has been done to the cube of WC-Co to study the morphology of the material. The testing that involve is hardness test and scratch test.

3.3.1 Hardness test

The hardness test is to measure the resistance of the material to permanent deformation such as indentation, abrasion, wear, and scratch. The importance of hardness test is to show the relationship between hardness and material properties. This test is preferred because it is nondestructive, easy and straightforward. The hardness test that will be doing in this experiment is the Vickers hardness test.

Vickers hardness test

Vickers hardness test is the test to see the hardness of the material through indentation. Vickers test is versatile because it can be used with any metal due to the indentation tool that Vickers using is diamond pyramid indenter. The basic principle of this test is to observe the material hardness to resist plastic deformation made by diamond pyramid indenter. With that indentation, the surface area of the indentation is measured horizontally and diagonally. The unit that is used in Vickers hardness test is HV also known as Vickers Pyramid Number or DPH also known as Diamond Pyramid Hardness. This value shows the hardness of the material, the higher the HV value, the higher the hardness of the material.

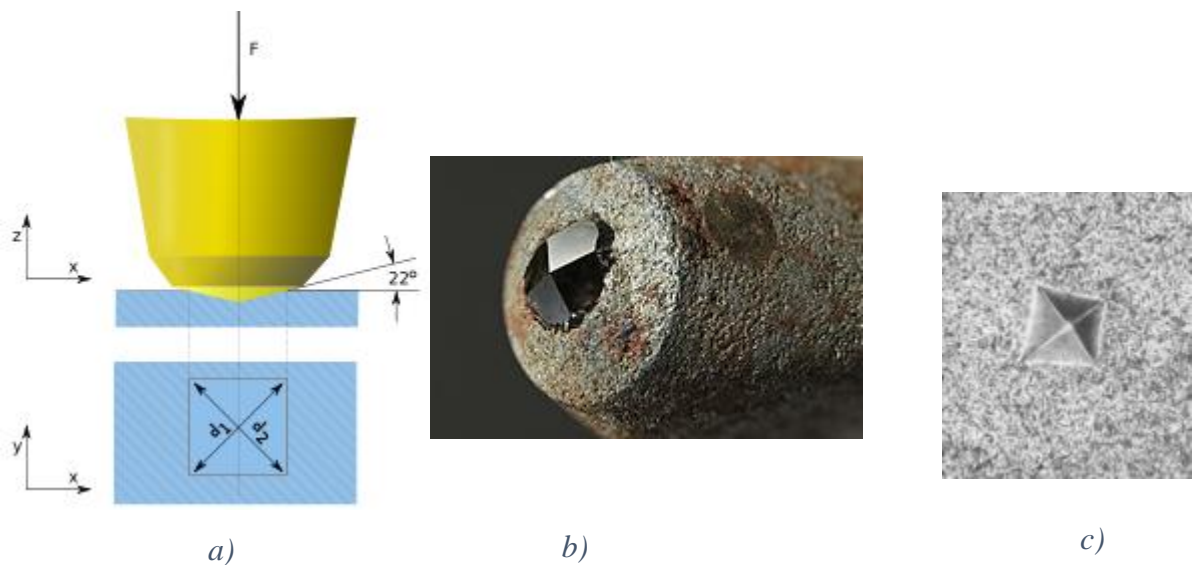


Figure 2: Vickers principle a) the diameter of diamond b) the diamond indenter shape c) the indentation on the surface of the material

The test use constant force and constant dwelling time at all the testing to make sure the reading can be compared with other material. Below are the equation and parameter used in the testing.

$$HV = 1.8544 \frac{F}{d^2} \text{ (Equation 3)}$$

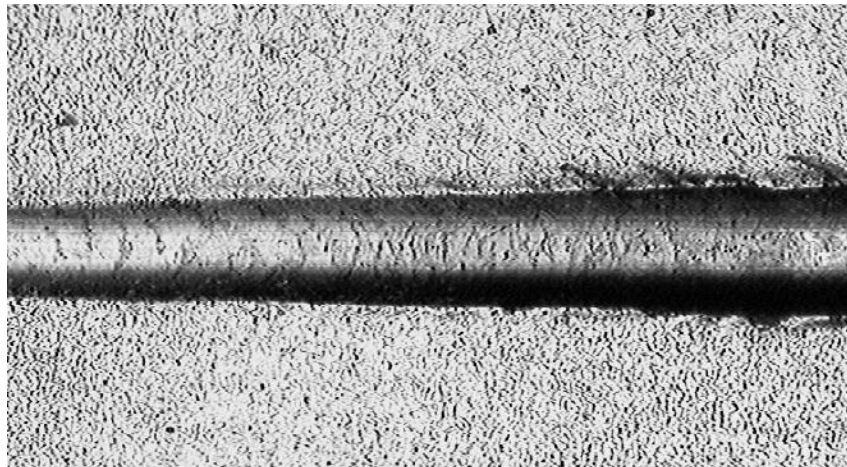
$$d = \frac{d_1+d_2}{2} \text{ (Equation 4)}$$

Parameter	Value
Test force (N)	1000
Dwelling time (s)	15

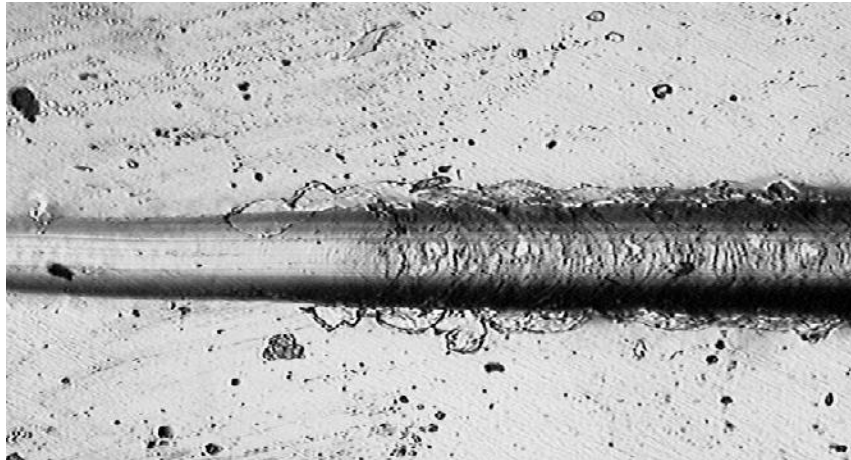
Table 6: Parameter of Vickers Test

3.3.2 Adhesion test

An adhesion test is the testing to see how good the adhesion of the coating with the material. It is important because some material cannot stick well with the coating. Even the material is strong, but without the help of coating, the wear time of the material will be faster compared to coated material. In this project, the scratch test is used to see the adhesion strength by using the friction of the diamond indenter against the sample. This friction will show the critical scratch load and critical displacement of the sample surface. With this result, the adhesion strength of the material can be see and compare with other material.



Sample A



Sample B

Figure 3: Example of light microscopic micrographs of scratches on samples A and B with different material

Figure 3 shows the scratch mark on the microscopic view on the sample with different material. In this figure, the morphology of the material can be seen clearly also can determine the strength of the adhesion of the coating material. The experiment use constant load and max length which is 10 000 mN and 1000 μm . With constant parameter is set, the result can be observe and compare with other material. Below are the parameter of the scratch test.

Table 1: Parameter of scratch test

Parameter	Value
Load (mN)	10 000
Displacement (μm)	1000
Preload Force (mN)	5

CHAPTER 4

RESULT AND DISCUSSION

4.1 INTRODUCTION

The drawing of the tungsten carbide cobalt content 12% (WC-Co 12%) material needs to be finalized before submitting it to supplier. The material shape is rectangular (80mm x 10mm x 10mm) and it consist of coated with titanium aluminium nitrate (TiAlN) and uncoated. The supplier had manufactured the material according to the drawing and dimension. Figure 4 and 5 shows the rectangular tungsten carbide coated and uncoated and for Figure 6 and 7 shows the cubic tungsten carbide, coated and uncoated.

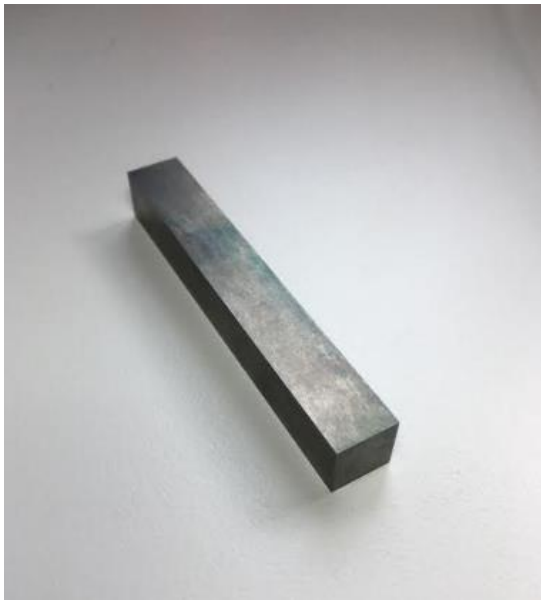


Figure 4: Uncoated Rectangular Tungsten Carbide



Figure 5: Coated Rectangular Tungsten Carbide



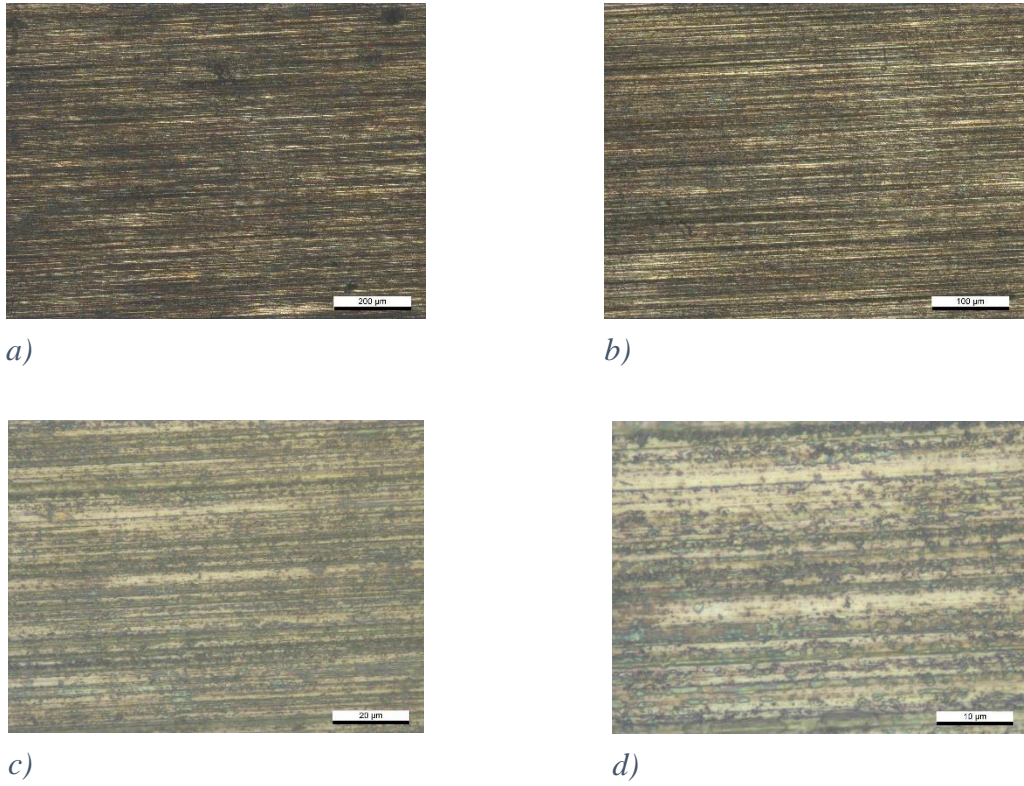
Figure 6: Uncoated Cubic Tungsten Carbide



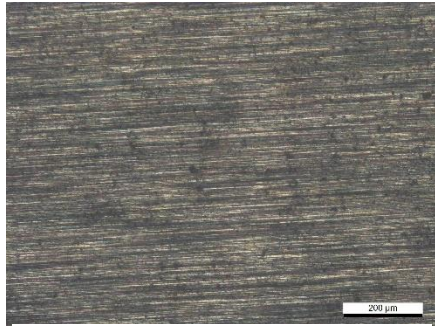
Figure 7: Coated Cubic Tungsten Carbide

4.2 OPTICAL MICROSCOPE RESULT

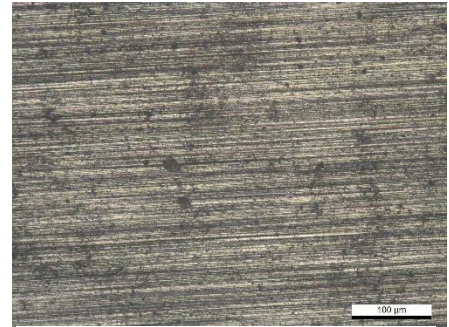
Before the experiment on the material is perform, the surface condition of the material is taken by using optical microscope because the surface of the material need to be observed. Figure 8, 9, 10, 11, 12 and 13 shows the picture of the surface if the materials with different type of magnifying glass.



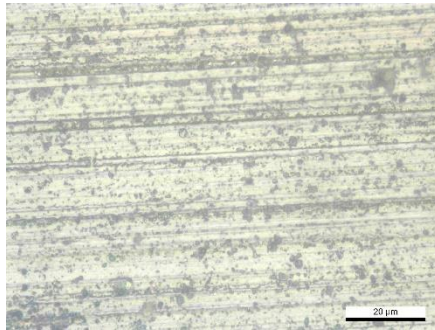
*Figure 8: Picture above shows the surface of the WC-Co 6%
a)50X b)100X c)500X d)1000X*



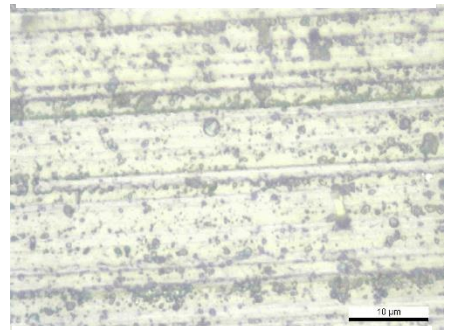
a)



b)

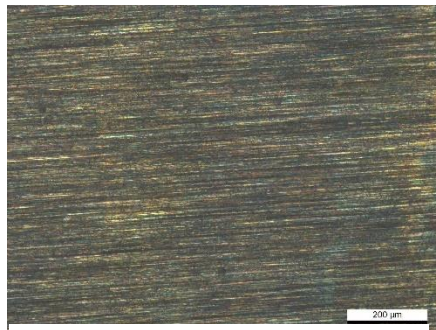


c)

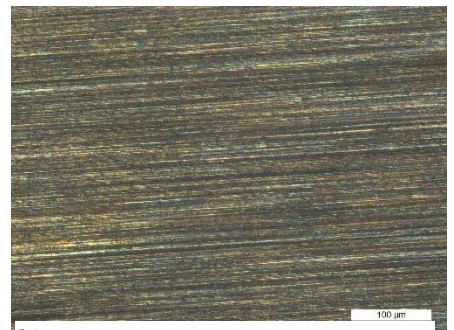


d)

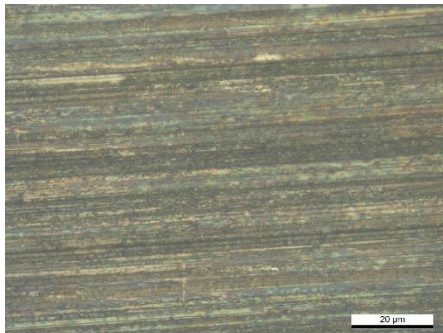
Figure 9: Picture above shows the surface of the WC-Co 6% TiAlN
a)50X b)100X c)500X d)1000X



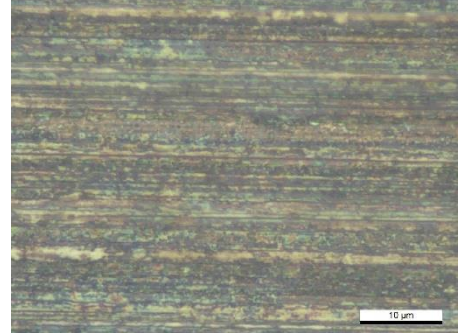
a)



b)

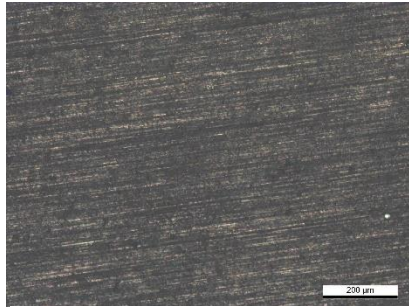


c)

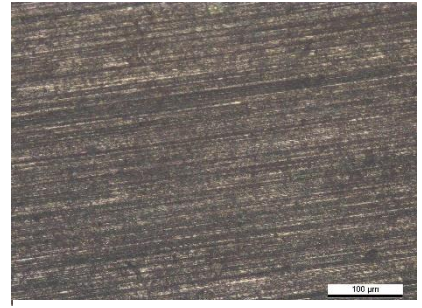


d)

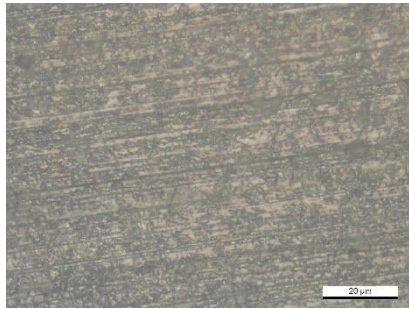
Figure 10: Picture above shows the surface of the WC-Co 9%
a)50X b)100X c)500X d)1000X



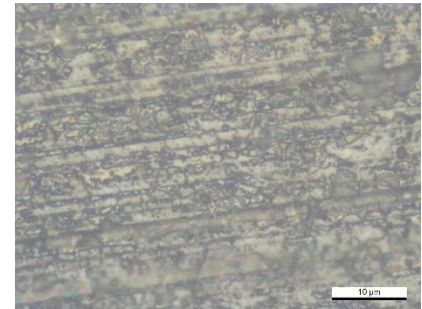
a)



b)



c)



d)

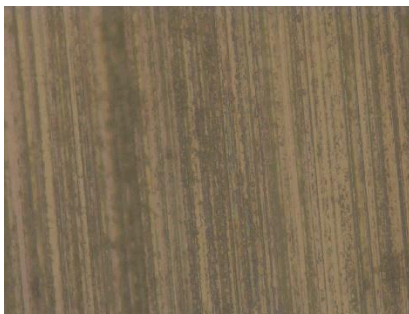
Figure 11: Picture above shows the surface of the WC-Co 9% TiAlN
a)50X b)100X c)500X d)1000X



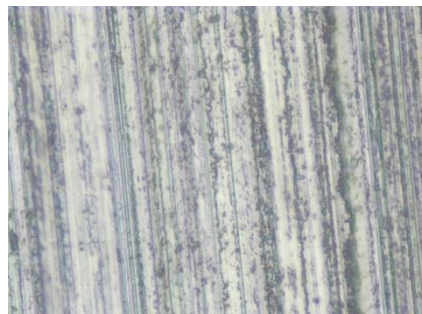
a)



b)

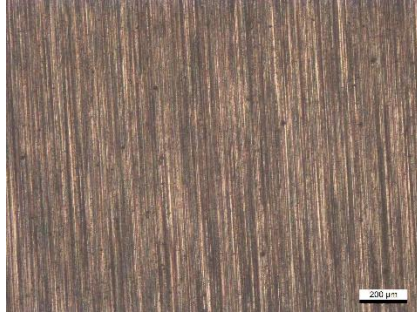


c)



d)

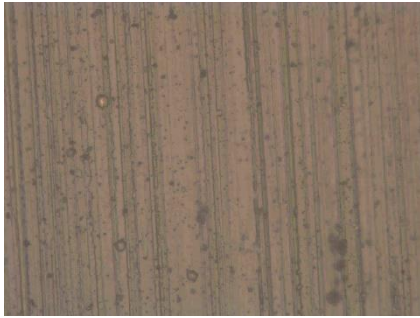
Figure 12: Picture above shows the surface of the WC-Co 12%
a)50X b)100X c)500X d)1000X



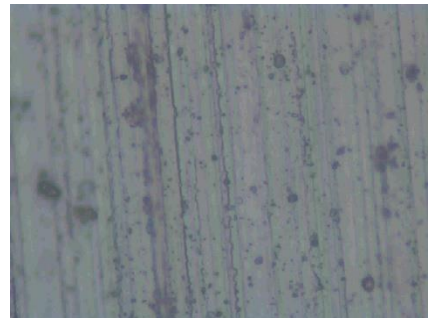
a)



b)



c)



d)

*Figure 13: Picture above shows the surface of the WC-Co 12% TiAlN
a)50X b)100X c)500X d)1000X*

4.3 HARDNESS TEST RESULT

Each material of tungsten carbide had been tested by using Vickers Hardness Machine in UTP. The result of the test is shown in the table below.

Table 2: Microhardness test result of WC-Co 6%

No	D1 (μm)	D2(μm)	F(gf)	HV
1	35.52	34.16	1000	1619.4
2	35.31	32.15	1000	1629.9
3	35.36	32.73	1000	1599.9
4	33.55	34.17	1000	1617.4
Average				1616.7

Table 3: Microhardness test result of WC-Co 6% TiAlN

No	D1 (μm)	D2(μm)	F(gf)	HV
1	31.63	27.93	1000	2091
2	29.83	27.92	1000	2224.1
3	31.68	26.81	1000	2168.2
4	29.08	30.68	1000	2077
Average				2140.1

Table 4: Microhardness test result of WC-Co 9%

No	D1 (μm)	D2(μm)	F(gf)	HV
1	33.23	33.23	1000	1678
2	34.26	32.22	1000	1677.9
3	32.23	34.23	1000	1678.9
4	35.33	31.10	1000	1680
Average				1678.7

Table 5: Microhardness test result of WC-Co 9% TiAlN

No	D1 (μm)	D2(μm)	F(gf)	HV
1	29.37	30.71	1000	2055
2	29.33	30.85	1000	2048.1
3	29.48	30.73	1000	2046.1
4	31.10	29.10	1000	2046.8
Average				2049

Table 6: Microhardness test result of WC-Co 12%

No	D1 (μm)	D2(μm)	F(gf)	HV
1	30.14	35.45	1000	1724.2
2	29.24	35.72	1000	1757.8
3	29.30	35.74	1000	1753.5
4	31.78	33.74	1000	1727.9
Average				1740.7

Table 7: Microhardness test result of WC-Co 12% TiAlN

No	D1 (μm)	D2(μm)	F(gf)	HV
1	31.35	24.85	1000	1692.6
2	31.67	34.85	1000	1676.3
3	31.42	34.82	1000	1690.5
4	33.98	32.88	1000	1659.3
Average				1679.7

According to Rei et al. [8], the hardness of WC is very good which is at 1300-1600 HV. The result of the Microhardness Test of tungsten carbide is higher than the expected result because the machine lighting is not bright enough and will make it harder to see the indentation at the surface of the material. Because of this, the reading will not be accurate and will result in the reading to be varied.

4.4 SCRATCH TEST

Table below shows the result of the critical scratch of the tungsten carbide cobalt (6%, 9% and 12%) that coated with titanium aluminium nitrate. According to M.S Raguveer et al [1] the adhesion strength will be decrease as the cobalt content increase. Below are the result from scratch test on WC-Co (6, 9, and 12%).

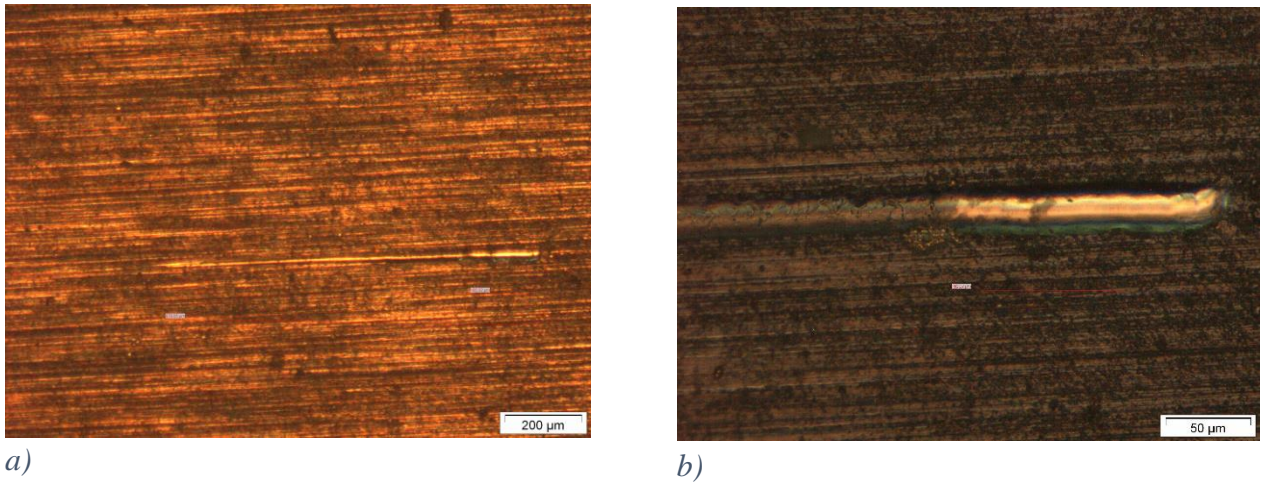
Table 8: Scratch test result

Sample coating with TiAlN	Load applied (mN)	Total distance (μm)	Critical scratch load (mN)	Critical distance (μm)
WC-Co 6%	10 000	1000	8494.60	823.10
WC-Co 9%	10 000	1000	6903.93	678.81
WC-Co 12%	10 000	1000	6572.14	648.61

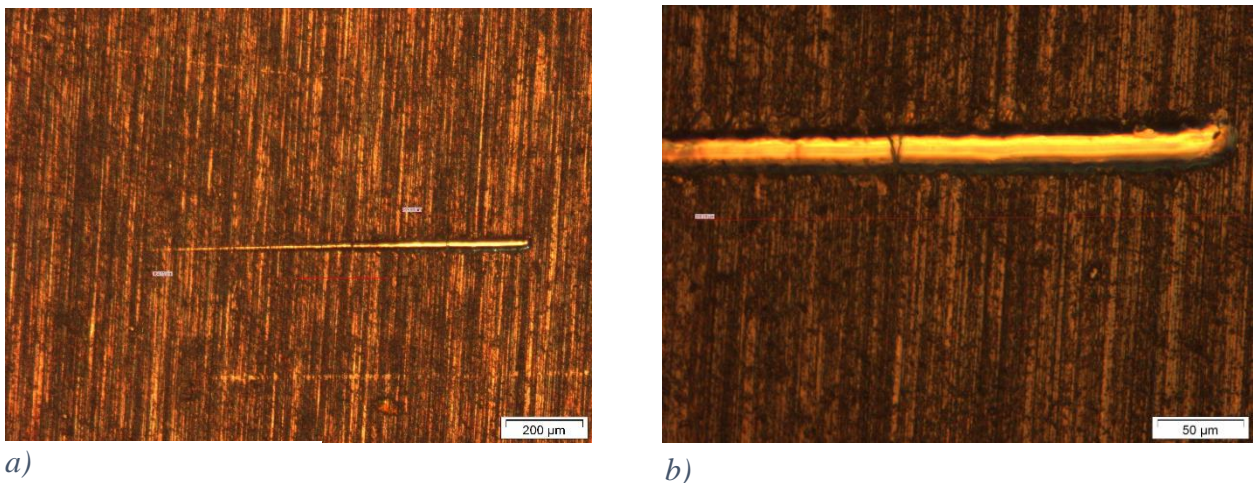
Table 10 shows the result of scratch test for the WC-Co with different cobalt content which is 6%, 9% and 12%. The thickness of the TiAlN coating is approximate 4 μm . For the force/load and distance, the values is fixed which is 10 000 mN and 1000 μm . This value is set to be fixed because from that value, the comparison can be made between this three materials. In this experiment, the critical load and critical distance is observe and compare. The higher the value, the higher the adhesion of the material and the coating.

From the table 8, the WC-Co 6% critical scratch load is 8494.60 mN and the critical distance is 823.10 μm . The value of critical scratch load is more than the other two which is WC-Co 9% is 6903.93 mN and for the WC-Co 12% is 6572.14 mN. As for critical distance, the value is also higher than the other two material which is for WC-Co 9% the value is 678.81 μm and for the WC-Co 12%, the value is 648.61 μm . From this result, it can be conclude that the higher the cobalt content in WC, the lower the adhesion strength of the material.

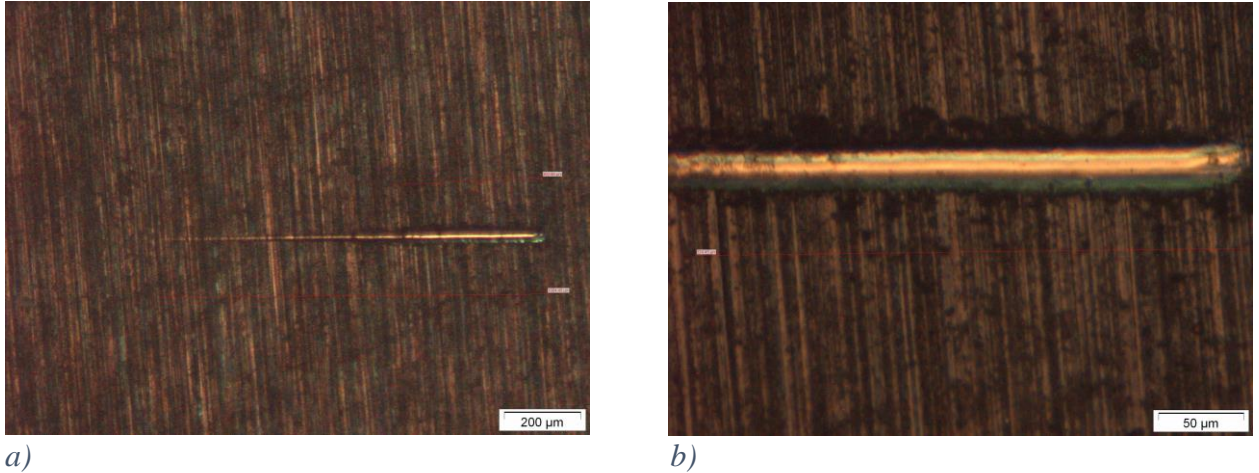
Below are the picture of scratch test on different type of cobalt which is 6%, 9% and 12% coated with TiAlN. On the picture, the distance of the scratch and the overall scratch distance is shown. This measurement is using an application that measure by a human. With that in mind, a parallax error might occur during measurement.



*Figure 14: Picture above shows the scratch on the WC-Co 6% material.
a) Shows the total length of the scratch test at 200 μm magnifier which is 1000 μm
b) Shows the critical scratch distance at 50 μm magnifier where the starting point the diamond indenter touch the surface of WC-Co at 836.3 μm*



*Figure 15: Picture above shows the scratch on the WC-Co 9% material.
a) Shows the total length of the scratch test at 200 μm magnifier which is 1000 μm
b) Shows the critical scratch distance at 50 μm magnifier where the starting point the diamond indenter touch the surface of WC-Co at 683.81 μm*



*Figure 16: Picture above shows the scratch on the WC-Co 12% material.
 a) Shows the total length of the scratch test at 200 μm magnifier which is 1000 μm
 b) Shows the critical scratch distance at 50 μm magnifier where the starting point the diamond indenter touch the surface of WC-Co at 675.55 μm*

From above result, the critical scratch value is shown which is at WC-Co 6%, the critical distance is at 836.3 μm . For WC-Co 9%, the critical distance is 683.81 μm and for WC-Co 12%, the critical distance is 675.55 μm . With this value in hand, it can be conclude that, the more the cobalt content in tungsten carbide, the lower the adhesion between coating and base material.

CHAPTER 5

CONCLUSION AND RECOMMENDATION

In conclusion, the hardness of the tungsten carbide are WC-Co 6% = 1612.5 HV, WC-Co 6% TiAlN = 2140.1 HV, WC-Co 9% = 1678.7 HV, WC-Co 9% TiAlN = 2049 HV, WC-Co 12% = 1740.7 HV, and WC-Co 12% TiAlN = 1679.7 HV. The value might be not accurate due to technical problem and parallax error. For the scratch test, the critical scratch load for WC-Co 6% TiAlN is 8494.60 mN while for WC-Co 9% TiAlN and WC-Co 12% TiAlN are 6903.93 mN and 6572.14 mN. As for the critical distance, WC-Co 6% TiAlN is 823.10 μm , WC-Co 9% TiAlN is 678.81 μm , and WC-Co 12% TiAlN is 648.61 μm . The result shows that the decreasing of the value for critical distance and scratch load which follow the assumption that is made in an early experiment where the higher the cobalt content, the lower the adhesion strength of the coating. While for the hardness, the more cobalt content, the higher the hardness of the material.

Some improvement has to be made in this study. Firstly, to get an accurate result, the material needs to be prepared accordingly. Before the coating is applied to the material, the surface of the material should be grinded and polished well. It is to ensure that the precision in result reading in the experiment and also to make sure that the adhesion between coating and base material becomes stronger. The roughness of the material indeed affects the hardness test result in this experiment. Because of this, the precision of the reading is lower when adding with the other error, which is parallax error and mechanical error. Besides that, the machine needs to be calibrated regularly by the professional to make sure that the machine still can be used by the student. Last but not least, the coating process needs to be done accordingly by the manufacturer. For this project, the manufacturer ignored the process detail and failed to give enough information regarding coating detail because it is confidential information. The result cannot be analysed carefully, and it affects the overall result in this study.

REFERENCES

- [1] M.S Raghuv eer, S.N Yoganand, K Jagannadham, R.L Lemaster and J Bailey, "Improved CVD diamond coatings on WC–Co tool substrates," *Wear*, vol. 253, no. 11-12, pp. 1194-1206, 2002.
- [2] S. W. Kallee, Friction stir welders for prefabricated components and panels, ALUMINIUM INTERNATIONAL TODAY, 2004, p. 1.
- [3] S.K.Selvam, and Dr. T.Parameshwaran Pillai, "Comparison Of Heavy Alloy Tool In Friction Stir Welding," *International Journal of Engineering Science and Technology (IJEST)*, vol. 4, no. 12, pp. 1-10, 2012.
- [4] W M Thomas, M J Russell, A Duncan & A Robelou, Friction stir welding – innovative variant techniques for the industry, Aluminium International Today, 2010.
- [5] Ma, R.S. Mishra and Z.Y., "Friction stir welding and processing," *Materials Science and Engineering*, vol. 50, pp. 1-78, 2005.
- [6] A. Baptista, F. Silva, J. Porteiro, J. Míguez, and G. Pinto, "Sputtering Physical Vapour Deposition (PVD) Coatings: A Critical Review on Process Improvement and Market Trend Demands," *Coatings*, vol. 8, no. 11, p. 402, 2018, vol. 8, no. 11, p. 402, 2018.
- [7] K. Chiteka, "Friction Stir Welding/Processing Tool Materials and Selection," *International Journal of Engineering Research & Technology (IJERT)*, vol. 2, no. 11, pp. 8-18, 2013.
- [8] R. Rai, A.De, H. K. D. H. Bhadeshia and T. DebRoy, "Review: friction stir welding tools," *Science and Technology of Welding and Joining*, vol. 16, no. 4, pp. 325-342, 2011.
- [9] Umar Patthi and Mokhtar Awang, "Friction Stir Welding on Aluminum 6092/sic/25p/t6 Metal Matrix Composite: its Microstructure Evolution and Mechanical Properties," *National Postgraduate Conference (NPC)*, pp. 1-6, 2011.
- [10] T. Prater, Alvin Strauss, George Cook, Brian Gibson, and Chase Cox, "A Comparative Evaluation of the Wear Resistance," *Journal of Materials Engineering and Performance*, vol. 22, no. 6, pp. 1807-1813, 2013.

- [11] Ashish BIST, J.S SAINI, Bikramjit SHARMA, "A review of tool wear prediction during friction stir welding of aluminium matrix composite," *Trans. Nonferrous Met. Soc. China*, vol. 26, pp. 2003-2018, 2015.
- [12] T. Prater, A. Strauss, G. Cook, B. Gibson and C. Cox, "A Comparative Evaluation of the Wear Resistance of Various Tool Materials in Friction Stir Welding of Metal Matrix Composites," *J. of Materi Eng and Perform*, vol. 22, no. 6, pp. 1807-1813, 2013.
- [13] R. Prado, L. Murr, D. Shindo and K. Soto, "Tool wear in the friction-stir welding of aluminum alloy 6061+20% Al₂O₃: a preliminary study," *Scripta Materialia*, vol. 45, no. 1, pp. 78-80, 2001.
- [14] Adesina, A. Y., Gasem, Z. M., & Al-Badour, F. A., "Characterization and evaluation of AlCrN coated FSW tool: A preliminary study," *Journal of Manufacturing Processes 25 (2017) 432–442*, vol. 25, pp. 432-442, 2017.
- [15] K. Bobzin, "High-performance coatings for cutting tools," *CIRP Journal of Manufacturing Science and Technology*, vol. 18, pp. 1–9, 2017, vol. 18, pp. 1-9, 2017.
- [16] W. A. Bryant, "The fundamentals of chemical vapour deposition," *Journal of Materials Science*, vol. 12, no. 7, pp. 1285–1306, 1977, vol. 12, no. 7, pp. 1285-1303, 1977.
- [17] S. Choudhary, S. Singhal, U. M. Srivastava, P. Jain, and V. Yadav, "Tungsten-Carbide Composite: A Review," *International Journal of Scientific & Engineering Research, Volume 3, Issue 9, September-2012*, vol. 3, no. 9, pp. 1–18, Nov. 2012. , vol. 3, no. 9, pp. 1-18, 2012.
- [18] B. Elsener, A. Rota, H. Böhni, "Impedance Study on the Corrosion of PVD and CVD Titanium Nitride Coatings," *Materials Science Forum*, vol. 44-45, pp. 29–38, 1991, vol. 44, pp. 29-38, 1991.
- [19] A. Fernández-Abia, J. Barreiro, J. Fernández-Larrinoa, L. L. D. Lacalle, A. Fernández-Valdivielso, and O. Pereira, "Behaviour of PVD Coatings in the Turning of Austenitic Stainless Steels," *Procedia Engineering*, vol. 63, pp. 133–141, 2013, vol. 63, pp. 133-141, 2013.
- [20] Sarin, M. Fitzsimmons and V. K., "Development of CVD WC–Co coatings," *Surface and Coatings Technology*, vol. 137, no. 2-3, pp. 158–163, 2001, vol. 137, no. 2-3, pp. 158-163, 137.

- [21] Lin, S. K. Khrais and Y., "Wear mechanisms and tool performance of TiAlN PVD coated inserts during machining of AISI 4140 steel," *Wear*, vol. 262, no. 1-2, pp. 64–69, 2007, vol. 262, no. 1-2, pp. 64-69, 2007.
- [22] T. Leyendecker, O. Lemmer, S. Esser, and J. Ebberink, "The development of the PVD coating TiAlN as a commercial coating for cutting tools," *Surface and Coatings Technology*, vol. 48, no. 2, pp. 175–178, 1991., vol. 48, no. 2, pp. 175-178, 1991.
- [23] B. Mann, V. Arya, A. Maiti, M. Rao, and P. Joshi, "Corrosion and erosion performance of HVOF/TiAlN PVD coatings and candidate materials for high pressure gate valve application," *Wear*, vol. 260, no. 1-2, pp. 75–82, 2006, vol. 260, no. 1-2, pp. 75-82, 2006.
- [24] A. Minevich, "Wear of cemented carbide cutting inserts with multilayer Ti-based PVD coatings," *Surface and Coatings Technology*, vol. 53, no. 2, pp. 161–170, 1992., vol. 53, no. 2, pp. 161-170, 1992.
- [25] D. Neves, A. E. Diniz, and M. S. F. Lima, "Microstructural analyses and wear behavior of the cemented carbide tools after laser surface treatment and PVD coating," *Applied Surface Science*, vol. 282, pp. 680–688, 2013, vol. 282, pp. 680-688, 2013.
- [26] H. Prengel, A. Santhanam, R. Penich, P. Jindal, and K. Wendt, "Advanced PVD-TiAlN coatings on carbide and cermet cutting tools," *Surface and Coatings Technology*, vol. 94-95, pp. 597–602, 1997, Vols. 94-95, pp. 597-602, 1997.
- [27] C. Ridges, M. Miles, Y. Hovanski, J. Peterson, and R. Steel, "Wear Testing of Friction Stir Spot Welding Tools for Joining of DP 980 Steel," *Friction Stir Welding and Processing VI*, pp. 97–103, 2011, pp. 97-103, 2011.
- [28] H. Saito, A. Iwabuchi, and T. Shimizu, "Effects of Co content and WC grain size on wear of WC cemented carbide," *Wear*, vol. 261, no. 2, pp. 126–132, 2006, vol. 261, no. 2, pp. 126-132, 2006.
- [29] H. Saito, A. Iwabuchi, and T. Shimizu, "Adherent diamond coatings on cemented carbide substrates with different cobalt contents," *Diamond and Related Materials*, vol. 10, no. 9-10, pp. 1700–1704, 2001, vol. 10, no. 9-10, pp. 1700-1704, 2001.
- [30] C. Yue, H. Feng, Z. Zhong, and H. Zhang, "Development and Application of Materials of Cutting Tools for Machining of Hard Materials," *Recent Patents on Materials Science*, vol. 8, no. 1, pp. 46–54, 2015, vol. 8, no. 1, pp. 46-54, 2015.