UNIVERSITI TEKNIKAL MALAYSIA MELAKA

ELECTROCHEMICAL BEHAVIOR OF ELECTROLESS Ni-W-P ALLOY DEPOSIT ON CARBIDE SUBSTRATE IN 3.5 WT% SALT SOLUTION

This report submitted in accordance with requirement of the Universiti Teknikal Malaysia Melaka (UTeM) for the Bachelor Degree of Manufacturing Engineering (Engineering Materials) (Hons.)

by

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This report is submitted to the Faculty of Manufacturing Engineering of UTeM as a partial fulfillment to the requirements for the degree of Bachelor of Manufacturing Engineering (Engineering Materials) (Hons.). The member of the supervisory is as follow:

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(Dr. Muhammad Zaimi Bin Zainal Abidin)

ABSTRAK

Kajian ini lebih tertumpu kepada penyaduran logam keras seperti tungsten karbida (WC) yang kebiasaanya digunakan sebagai alat pemotong untuk meningkatkan rintangan haus, ketahanan dan sifat kekerasan untuk memperoleh permukaan bahan kerja yang berkualiti. Kaedah penyaduran nikel tanpa elektrik adalah salah satu teknik penyaduran yang sangat baik yang dapat menghasilkan kekerasan yang tinggi, ketebalan yang seragam walaupun pada bahagian-bahagian yang kompleks dan juga penghalang kepada kakisan dan haus. Pelbagai mandian penyaduran dengan nilai pH yang berbeza akan digunakan untuk mengkaji kesan mandian penyaduran pH terhadap komposisi penyaduran Ni-W-P tanpa elektrik. Analisis untuk ciri-ciri penyaduran akan dilakukan untuk menganalisa morfologi permukaan terhadap penyaduran aloi dengan menggunakan mikroskopi electron penskanan (SEM) manakala spektroskopi tenaga serakan sinar-x (EDX) dan belauan sinar-x (XRD) digunakan untuk mengkaji komposisi kandungan nikel, tungsten dan fosforus dalam penyaduran aloi ternari atas substrat WC. Kajian terhadap kekerasan penyaduran aloi ternari akan dinilai dengan cara Penguji Kekerasan Vickers. Tindakan kakisan akan dikaji dalam 3.5 wt. % larutan natrium klorida (NaCI) dengan menjalankan pengukuran electrokimia. Analisis EDX menunjukkan kandungan fosforus menurun secara beransur-ansur yang membuatkan kadar pemendapan meningkat selari dengan peningkatan nilai pH. Tambahan pula, ia juga menunjukkan peningkatan jumlah tungsten yang membuatkan kekerasan meningkat melalui ujian kekerasan. Tingkah laku elektrokimia menunjukkan bahawa substrat yang telah bersalut menunjukkan kadar kakisan yang lebih baik daripada keluli lembut walaupun lebih buruk daripada keluli tahan karat dan substrat yang bersalut juga tidak mempamerkan mana-mana kawasan pasif.

ABSTRACT

This study is focusing more on coating hard metal such as tungsten carbide (WC) that commonly utilized as cutting tools to improve its wear resistance, durability and hardness properties to obtain a proper workpiece surface quality. Electroless nickel (EN) is one of the excellent coating techniques that can provide high hardness, uniform thickness even in complex parts, as well as excellent corrosion and wear resistance. Various plating bath with different value of pH will be utilized to investigate the effect of plating bath pH on electroless Ni-W-P deposition composition. Analysis of coating properties will be done to analyze the surface morphology of the alloy coating by means of scanning electron microscopy (SEM) while energy-dispersive x-ray spectroscopy (EDX) and x-ray diffraction (XRD) are used to study the composition of nickel, tungsten and phosphorus contents in the ternary alloy coating on the WC substrates. The investigation of the hardness of the ternary alloy deposition will be evaluated by means of Vickers Hardness Tester. Corrosion behavior will be investigated in 3.5 wt. % sodium chloride (NaCI) solution by conducting electrochemical measurement. EDX analysis showed phosphorus content decreased gradually that made the rate of deposition increased as pH value increased. Furthermore, it also showed the amount of tungsten increased that result the increasing of hardness by means of hardness test. Electrochemical behavior showed that coated substrate resulting better corrosion rate than mild steel although poorer corrosion rate that stainless steel and also coated substrate did not exhibit any passive area.

DEDICATION

To everyone that contributes to this research and my friend that has been helping me all along

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TABLE OF CONTENT

Abstrak	i
Abstract	ii
Dedication	iii
Acknowledgement	iv
Table of Content	v
List of Tables	viii
List of Figures	ix
List of Abbreviations, Symbols and Nomenclatures	xi
CHAPTER 1: INTRODUCTION	**1**
1.1 Background of Study	1
1.2 Problem Statement	3
1.3 Objectives	4
1.4 Scope of Study	4
CHAPTER 2: LITERATURE REVIEW	**5**
2.1 Introduction of machine tools	5
2.1.1 Cutting tools	5
2.1.2 Requirement of cutting tools	7
2.1.3 Coated tools	7
2.2 Introduction of coating	8
2.2.1 Alloy coating	8
2.2.2 Coating for recycling	9
2.3 Method of alloy coating	10
2.3.1 Dry process	11
2.3.1.1 Physical Vapor Deposition (PVD)	11
2.3.1.2 Chemical Vapor Deposition (CVD)	12
2.3.2 Wet process	12

2.3.2.1 Electrodeposition 13
2.3.2.2 Electroless deposition 14
2.3.3 Comparison of alloy coating technique 16
2.4 Electroless nickel alloy deposition 16
2.4.1 Introduction 16
2.4.2 Basic reaction 17
2.4.3 Components required in the electroless nickel plating bath 20
2.4.3.1 The source of nickel ions 20
2.4.3.2 Reducing agent (sodium hypophosphite) 21
2.4.3.3 Complexing agent 21
2.4.3.4 Stabilizers 22
2.4.4 Effect of variables in the electroless nickel plating bath 23
2.4.4.1 Effect of temperature 23
2.4.4.2 Effect of pH 24
2.4.4.3 Effect of complexing agent 26
2.4.4.4 Effect of metal ion additive on electrochemical alloy properties 28
2.4.5 Properties of electroless nickel-based ternary alloy (Ni-W-P) deposition 32
2.4.5.1 Hardness 33
2.4.5.2 Wear 34
2.4.5.3 Conclusion 35

CHAPTER 3: METHODOLOGY 37
3.1 Process flow chart 37
3.2 Raw materials 39
3.2.1 Materials involved in electroless Ni-W-P plating bath 39
3.2.2 Materials involved in Electrochemical Measurement 39
3.2.3 Type of substrate 40
3.3 Sample preparation 41
3.4 Electroless Ni-W-P plating bath 42
3.5 Analysis of Ni-W-P alloy deposition on carbide substrate 44
3.5.1 X-ray diffraction (XRD) 44

3.5.2 Scanning electron microscopy (SEM) 46
3.5.3 Energy-dispersive X-ray Spectroscopy (EDX) 47
3.6 Hardness testing 48
3.7 Corrosion behaviour of electroless Ni-W-P alloy deposition 49

CHAPTER 4: RESULT AND DISCUSSION **53**
4.1 Observations of deposited sample 53
4.2 Rate of deposition of the ternary alloy coating 54
4.2.1 Weight of deposit 54
4.2.2 Surface area of deposit 55
4.2.2.1 Surface area of right circular cone 55
4.2.2.2 Surface area of shaded octagon 58
4.2.2.3 surface area of shaded circle 59
4.2.3 Density of deposit 60
4.3 Surface morphology by SEM and compositional analysis by EDX 63
4.3.1 Ni-P alloy deposition 63
4.3.2 Ni-W-P alloy deposition 65
4.4 X-ray diffraction analysis (XRD) 70
4.5 Microhardness Vickers of Ni-W-P alloy deposition 71
4.6 Electrochemical behaviour of electroless Ni-W-P alloy deposition 72

CHAPTER 5: CONCLUSION AND RECOMMENDATION
5.1 Conclusion 78
5.2 Recommendation 79

REFERENCES **81**

APPENDICES
Appendix A: Gantt chart PSM 1 & 2
Appendix B: Catalog of WC insert that used as substrate
Appendix C: SEM and EDX result
Appendix D: Turnitin result

LIST OF TABLES

2.1	The components and functions required in electroless plating bath	20
2.2	The type of complexing agent used in electroless plating baths	22
2.3	Type of electroless ternary alloy deposition and their enhancing properties	29
2.4	Chemical compositions of deposition alloys	33
3.1	Description of QPMT 10T335 PPEN WC insert	41
3.2	Bath composition and operating condition of the baths used	43
3.3	Technical specifications of XRD machine utilized	45
4.1	Value of the weight of deposit for various plating bath pH	54
4.2	The rate of deposition of various plating bath pH	61
4.3	Chemical composition of Ni and P in various plating bath pH	65
4.4	Chemical composition of Ni-W-P coatings determined by EDX Analysis	68
4.5	Microhardness values for various plating bath pH	71
4.6	Calculation value of electrochemical behaviour in 3.5 wt% NaCI solution	77

LIST OF FIGURES

2.1	Typical carbide inserts with various shapes	6
2.2	Comparison between the recycled drill and the new drill	10
2.3	Schematic illustrations of the migration of metal ions from a sacrificial anode to the cathode with formation of a metal deposit on the surface	13
2.4a	Schematic illustration of nonuniform coatings in electroplated parts	14
2.4b	Design guidelines for electroplating process	14
2.5	Principle of electroless deposition	15
2.5a	Oxidation of the reductant	15
2.5b	Reduction of metal ion, leading to formation of metal layer	15
2.5c	Graph describing rate of deposition	15
2.6	The elementary steps of the surface reaction in electroless Ni-P alloy deposits	19
2.7	Effect of temperature on the rate of deposition	24
2.8	Effect of pH on the rate of deposition	25
2.9	Effect of pH on phosphorus content	25
2.10	Effect of concentrations of organic acids on the rate of deposition	27
2.11	Variation in the hardness of Ni-Cu-P coatings as a function of applied loads	30
2.12	The relation between the red area percentages by the porosity test and the deposition thickness of the electroless Ni-Sn-P deposition and the compared Ni-P deposition	31
2.13	Hardness profile for electroless Ni-P and Ni-W-P alloys from acidic bath	33
2.14	Specific wear rates for electroless alloys	35
3.1	Workflow chart of electroless Ni-W-P alloy deposition	38
3.2	3.5 wt. % NaCI solution	40
3.3	QPMT 10T335 PPEN WC insert	41

3.4	Schematic illustrations of electroless nickel alloy deposition	43
3.5	PW3040/60 X'PERT PRO X-ray system (PANanalytical)	45
3.6	Scanning electron microscopy (SEM)	46
3.7	Energy-dispersive X-ray that attached with SEM	47
3.8	The HM-20 Vickers Hardness Tester	48
3.9	Schematic diagram of sample substrate mounted with epoxy resin and connected with insulated copper wire	49
3.9a	Represents as side view	49
3.9b	Represents as front view	49
3.10	Electrochemical analysis diagram	50
3.11	Corrosion process showing anodic and cathodic current composition	51
4.1	Picture of WC insert	53
4.1a	before electrroless deposition	53
4.1b	after electroless deposition	53
4.2	Rate of deposition in acidic bath	61
4.3	Rate of deposition in alkaline bath	62
4.4	SEM images and EDX analysis of acidic plating bath pH for electroless Ni-P alloy deposition	64
4.4a	pH 4.5	64
4.4b	pH 4.75	64
4.4c	pH 5.0	64
4.5	Chemical composition of Ni and P in electroless ni-P alloy deposition in the form of chart	65
4.6	SEM images and EDX analysis of acidic bath pH for electroless Ni-W-P alloy deposition	66
4.6a	4.5	66
4.6b	4.75	66
4.6c	5.0	66
4.7	SEM images and EDX analysis of alkaline bath pH for electroless Ni-W-P alloy deposition	67
4.7a	8.5	67
4.7b	9.0	67

4.7c	9.5	67
4.8	Chemical composition of acidic plating bath in electroless Ni-W-P alloy deposition	69
4.9	Chemical composition of alkaline plating bath in electroless Ni-W-P alloy deposition	69
4.10	XRD analysis of electroless Ni-W-P alloy deposition on WC Insert	70
4.11	Comparison of microhardness values of various plating bath pH	72
4.12	Polarization curve measurement of mild steel in 3.5 wt% NaCI solution	73
4.13	Polarization curve measurement of stainless steel 304 in 3.5 wt% NaCI solution	73
4.14	Polarization curve measurement of stainless steel 316L in 3.5 wt% NaCI solution	74
4.15	Polarization curve measurement of Ni-W-P substrate for acidic solution in 3.5 wt% NaCI solution	75
4.16	Polarization curve measurement of Ni-W-P substrate for alkaline solution in 3.5 wt% NaCI solution	75
4.17	Comparison of Ni-W-P substrate with mild steel and stainless steel in 3.5 wt% NaCI solution	76

LIST OF ABBREVIATIONS, SYMBOLS AND NOMENCLATURES

Al_2O_3	-	Alumina
ASTM	-	American Standard of Testing Material
CBD	-	Chemical Bath Deposition
CH_3COONH_4	-	Ammonium acetate
CNC	-	Computerized Numerical Control
Co	-	Cobalt
Co-C	-	Cobalt Carbide
Cu	-	Copper
CVD	-	Chemical Vapor Deposition
ED	-	Electrochemical Deposition
EDX	-	Energy-dispersive X-ray Spectroscopy
EN	-	Electroless Nickel
H^+	-	Hydrogen ion
HCI	-	Hydrochloric acid
H_2O	-	Water
$H_2PO_2^-$	-	Hypophosphite
$H_2PO_3^-$	-	Orthophosphite
HNO_3	-	Nitric acid
HV	-	Hardness Vickers
Mn	-	manganese
Mo	-	molybdenum
$Na_3C_6H_5PO_2.2H_2O$	-	Trisodium Citrate Dihydrate
NaCI	-	Sodium Chloride
$NaH_2PO_2.H_2O$	-	Sodium Hypophosphite Monohydrate
NaOH	-	Sodium Hydroxide
$NaWO_4.2H_2O$	-	Sodium Tungstate Dihydrate
NC	-	Numerical Control
Ni	-	Nickel

Ni-Cu-P	-	Nickel – Copper - Phosphorus
$Ni(H_2PO_2)_2$	-	Nickel salt of hypophosphorus acid
Ni-P	-	Nickel - Phosphorus
Ni-Sn-P	-	Nickel – Tin - Phosphorus
$NiSO_4.6H_2O$	-	Nickel Sulphate Hexahydrate
Ni-W-P	-	Nickel – Tungsten - Phosphorus
Ni-Zn-P	-	Nickel – Zinc – Phosphorus
P	-	Phosphorus
PVD	-	Physical Vapor Deposition
Rh	-	Rhodium
RT	-	room temperature
SEM	-	Scanning Electron Microscopy
SiC	-	Silicon Carbide
Sn	-	Tin
TiAlN	-	Titanium Aluminium Nitride
TiC	-	Titanium Carbide
TiCN	-	Titanium Carbide Nitride
TiN	-	Titanium Nitride
W	-	Tungsten
WC	-	Tungsten Carbide
WC-Co	-	Tungsten-Cobalt Carbide
XRD	-	X-ray Diffraction
Zn	-	Zinc

CHAPTER 1
INTRODUCTION

This chapter describes in general information about the study on coating process of carbide substrate. Furthermore, the problem statement, objective of the research and scope of this study are also highlighted in this section.

1.1 Background of study

Coating is an ideal technique to improve the limits of use of materials in terms of their performance capabilities, such as wear and corrosion resistance. Nowadays, several of coating methods available and rapid deposition technology development is growing fast due to high demand for the state-of-the-art surface modifications (Kwasny and Mikula, 2012). Furthermore, this study is focusing more on coating hard metal such as tungsten carbide that acts as substrate and commonly used as cutting tools. The main motive of coating on this type of material is to improve its mechanical properties in terms of wear resistance and durability and can work at higher service parameter such as temperature and load. Commonly, the tungsten carbide is utilized in wide application of cutting tools for machining operations such as drilling, metal cutting, milling and turning because of its incredible characteristics of high hardness and wear resistance. Hence, the need to increase its durability is by coating another layer of hard coating such as TiN and TiAlN. Moreover, the carbide that utilized for insert of cutting tools should be protected by hard coating. According to Mubarak *et al.* (2005) the term hard coatings can be described as "*the property of high hardness in the mechanical sense with good tribological properties*."

Common coating techniques use to deposit hard coating including Physical Vapor Deposition (PVD) and Chemical Vapor Deposition (PVD).PVD is a vaporization coating method that is done under high-temperature vacuum condition, gas-phase transport, and the condensation of atomized matter onto substrates maintained at lower temperatures (Reidel and Chen, 2012).While CVD is a method of depositing solid and form a thin film from gaseous phases material. There are several advantages and disadvantages for both types of hard coatings. For PVD, it can produce various type of coating; the coating thickness is small and accurate surface replication, high wear resistance and low frictional coefficient. One of the disadvantages of PVD are the specimen to be coated should be rotated during coating process to obtain a consistent coating thickness. This is because during processing, a solid piece of film material is heated and when it melted, it will evaporate in highly directional deposition. Hence, the specimen should be rotated to ensure the entire surfaces at the sharp edges will be coated properly and have consistent thickness. Furthermore, the manufacturing cost for this technique is also due to the need of high energy and shielding system requirements. On the other hand, the advantages of CVD method are uniform distribution of source over large areas and complex shape. This is because the materials distribute in multidirectional deposition. So, the entire surface can be coated properly. Furthermore, this method has high wear resistance and the production cost to coat is economical compared to PVD method. The drawback of CVD method is the contaminants. This method uses toxic metal that can affect the environment and worker.

Because of both types of coating above showed its own constraint plus the operating costs are too expensive, another method should be proposed in terms of cost, process time and product quality. The most suitable process for applying metallic coating thickness is electroless deposition process. This is because it enhances the quality of the product in terms of high hardness, uniform thickness even in intricate parts, high wear and corrosion resistance. Furthermore, it provides acceptable process times and obviously at low capital and operating costs (Palaniappa and Seshadri, 2008)**.**

Moreover, the ternary alloy coating will be utilized in this study. This ternary alloy coating is another alternative to binary Ni-P coating. According to Hong *et al.*

(2012), the third element is added to the binary Ni-P system to produce ternary alloy coating. For example, the ternary Ni-M-P alloy coatings. M usually represents the transition metal that added into the ternary alloy such as tungsten (W), cobalt (Co), Manganese (Mn), Rhenium (Rh) and Molybdenum (Mo). The third element that added is tungsten (W) and ternary alloy coating will become Ni-W-P. According to Sudagar *et al.* (2013), the choice of alloy coating will be considered on the applications and economics as well as influencing the properties of the coating. For example, W is used for the third element and this alloy increases the hardness, wear and corrosion resistance, suitable for this research that uses carbide substrate that focusing more on hardness.

1.2 Problem Statement

Tungsten carbide (WC) is coated to enhance the hardness of the material. So, the coating process will enhance the properties in terms of hardness resistance, wear resistance, and extending the tool life if utilizing for cutting tools. Besides that, many types of coating process have been researched and most of them required high capital and operating cost. This research propose another technique that can reduce the cost as well as increase the hardness, wear and uniform thickness because some of coating methods will give poor uniform thickness such as PVD method. Furthermore, the ternary alloy coating will be utilized in this research. The problems that will be faced are how to produce a substitute coating for used WC cutting tool insert, the possibilities to coat alloy coating using electroless deposition method, and how hard the coating can be from the composition of the alloy coating. Hence, the electroless method is the ideal technique to deposit alloy coating. Thus, in this research the ternary alloy coating on carbide substrate is generated by varying plating bath pH in order to determine the optimum composition for hardness and wear and to evaluate the electrochemical behavior of Ni alloy coating in 3.5 wt% NaCI solution.

Moreover, many variables should be controlled in order to obtain the optimum coating such as temperature, pH, and complexing agent. Besides that, there are several of plating bath pH used that is 4.5, 4.75, 5.0, 8.5, 9.0 and 9.5. Based on the theory stated by Mallory and Hajdu (1990), increasing pH will cause the rate of deposition to increase. Based on that theory, various plating bath pH has been selected in order to test experimentally whether the rate of deposition will increase as the pH value increase.

1.3 Objectives

This research was aimed to synthesize and characterize the ternary alloy coating (Ni-W-P) by electroless deposition method. This research is carried out under certain objectives as follows:-

i. To investigate the effect of plating bath pH on electroless Ni-W-P alloy deposition on carbide substrate surface and their mechanical properties.
ii. To evaluate the effect of Ni-W-P composition from various plating bath pH on electrochemical behavior in 3.5 wt% NaCI solution.

1.4 Scope of Study

The focus of the study is on the electrochemical behavior of electroless Ni alloy coating on the carbide substrate. The research will be conducted by doing electroless Ni alloy deposition on hard carbide substrate to replace current coating such as TiN and TiAlN. The deposition of the Ni alloy will be done at various plating bath condition such as bath composition and bath pH. The surface morphology and the composition of the alloy coating are studied using SEM-EDX and XRD. The electrochemical behavior of the alloy coating in 3.5 wt% NaCl solution is investigated using electrochemical measurement method such as Tafel Extrapolation Technique and Anodic Polarization Measurement. The hardness of the alloy coating is determined using Vickers hardness test.

CHAPTER 2

LITERATURE REVIEW

This chapter is about the review study from the previous research. The objective of this chapter is to analyze and summarize the previous study that related to this paper.

2.1 Introduction of machine tools

Machine tool utilizes as shaping or forming the metal or other materials of the parts. The techniques for machine tools to form the parts include lathes, drilling machines, grinding machine and milling machine. All machine tools have their own ways to hold the workpiece and at the same time they provide the guide movement of the parts of the machine. Nowadays, many machine tools can be controlled under automatic control such as numerical control (NC) machine and computerized numerical control (CNC) that shows the invention of machine tools from the simplest to the most complex.

2.1.1 Cutting tools

Main principle of cutting tools including single point lathe tools, multi-point milling tools, drills, reamers and taps. Based on this research, the substrate used is carbide substrate that is carbide based alloy such as tungsten carbide (WC), cobalt carbide (Co-C) and tungsten-cobalt-carbide (WC-Co). This type of materials will be utilized in this research. Furthermore, about 90% of all the tools utilized to cut the parts that made up of ferrous metal and non-ferrous metals are cemented carbide or coated cemented carbide tools (Minato, 2012). Moreover, the cemented carbide is widely used in industrial field such as machining in terms of cutting, grinding, drilling and milling because of its high hardness and high wear resistance (Zhu *et al.*, 2012). According to Society of Manufacturing Engineers, in most application, carbide cutting tools are widely used more than high-speed steels because they cut about 3 to 5 times faster compared to high-speed steels. Carbide is used in solid round tools or in the form of replaceable inserts. Insert is individual cutting tools with several cutting points and normally it is clamped on the tool holder (Galimberti, 1968). In addition, the carbide insert is an alternative technique to secure the usage of cutting tools because each insert has a number of cutting points such as a triangular insert has six number of cutting points, and square insert has eight numbers of cutting points. With the usage carbide insert for machining process, it will reduce the cost of using cutting tool. When the one edge of the insert is in wear condition, another edge is still available to make another machining process (Kalpakjian and Schmid, 2006). Figure 2.1 shows typical carbide inserts with the variety of shapes.

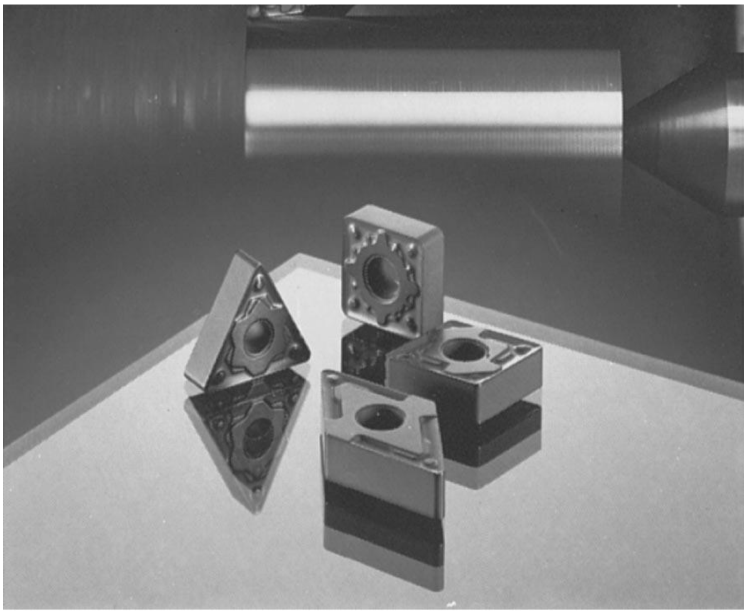

Figure 2.1: Typical carbide inserts with various shapes (Kalpakjian and Schmid, 2006)

2.1.2 Requirement of cutting tools

Most of the materials of the cutting tools belong to a non-ductile materials group. It is well known that the chipping of cutting edges and the carbide cutting tools that fracture are the most frequent types of tool failures (Zhao and Hong, 1992). The cutting tool can be classified as high temperatures, high contact stresses and rubbing along the tool-chip interface and along the machined surface. The requirements for an ideal cutting tool material that should have are high hardness and abrasive resistance, a sufficient toughness, and good mechanical properties such as wear resistance (Denkena *et al.*, 2013). This requirements show the vivid evidence that when making machining process, the tool does not undergo any plastic deformation. This will retain the cutting tool in terms of shape and sharpness. Moreover, the materials of the cutting tool must possess high hardness than the material it is being utilized to machine. According to Isakov (2001), the numbers of Vickers hardness for indexable inserts that made of cemented carbides in the range between 1000 HV and 1950 HV. In terms of wear resistance, the cutting tool is able to maintain sharpened edge throughout the machining process. Because of these demanding requirements, the development of various cutting tool materials in terms of mechanical, physical and chemical properties had been focused. The examples of desirable tool-material characteristics for a particular application are hardness and strength which show significant with respect to the mechanical properties (Kalpakjian and Schmid, 2006).

2.1.3 Coated tools

Since the 1960s, the continuous development of new alloys and engineered materials made them high strength and toughness but generally abrasive and chemically reactive with the cutting tool materials. These problems led to the significant development of coated tools. The cutting tool that has been coated have unique properties such as lower friction, higher adhesion, higher resistance to wear and cracking, higher hot hardness and impact resistance. Hot hardness is also referred as red hardness that is the ability of cutting tool to maintain sharp cutting edge.