# APPLICATION OF DESIGN OF EXPERIMENT TECHNIQUE IN THE NICKEL-CHROMIUM ELECTROPLATING PROCESS ON WC-Co SUBSTRATE PRIOR HFCVD DIAMOND COATING

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**ESPECIALLY DEDICATED TO ….**

***My beloved fiancée***
Somaye

***My Father***
HassanAli

***My Brothers and Sisters….***

***My closed friends and relatives.....***

May Allah bless all people that I love and it's my honour to share this happiness with my love ones.

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# ABSTRACT

Cemented tungsten carbide (WC-Co) cutting tools are widely used in metal cutting industry. These tools wear rapidly when machining abrasive alloys and glass-epoxy composites. In order to enhance the overall machining effectiveness of WC-Co, the coating of diamond on WC-Co has been attractive by virtue of its excellent hardness and low coefficient of friction. Poor adhesion of diamond coating to WC-Co is however observed because of the weak interface bonding resulting from graphite film formation during low-pressure diamond deposition due to presence of the cobalt binder. The use of interlayer between WC-Co and diamond coating has proved to overcome and reduce the negative effect of cobalt binder. In the current study, the coating of nickel-chromium on the cemented carbide (WC-Co) substrate via electroplating process has been investigated in order to obtain a thin layer with a good adhesion. Nickel was first coated on WC-Co substrate in experiments/trials involving three variable parameters; bath temperature, current density and plating time. Subsequently, chromium was coated on the nickel surface in experiments/trials involving two variable parameters; bath temperature and current density. Two levels factorial design of experiments involving nickel and chromium coatings on WC-Co substrates enabled the identification of current density and plating time as the two important variable parameters affecting the thickness of nickel and chromium coating. Temperature did not appear to have a significant influence on the thickness of coating. However, it had notable effects on the adhesion of nickel and chromium coatings. The current density had a slight effect on the adhesion in comparison with the temperature. A current density of 4 Amp/dm$^2$, bath temperature of 58 ºC, and plating time of 26.31 minutes were identified as suitable electroplating parameter conditions for producing a thin, 6.3 µm, nickel layer with good adhesion on the WC-Co substrate whilst a current density of 12 Amp/dm$^2$, bath temperature of 42 ºC, and plating time of 15 minutes were identified as suitable electroplating parameter conditions for producing a thin, 2.4µm, chromium layer with good adhesion on the nickel coated WC-Co substrate.

# ABSTRAK

Alat pemotongan karbide secara meluasnya digunakan di dalam industri pemotongan logam. Alat ini haus dengan cepat ketika memotong aloi pelelas dan komposit kaca-epoxy. Untuk meningkatkan keberkesanan pemesinan WC-Co, salutan berlian pada karbide telah menarik perhatian disebabkan sifatnya yang keras dan pekali geseran yang rendah. Kelekatan salutan berlian yang lemah telah dikenalpasti disebabkan oleh kelekatan antara muka yang lemah yang mana akan menghasilkan pembentukan grafit semasa salutan berlian pada tekanan yang rendah disebabkan oleh kehadiran bahan pengikat kobalt. Penggunaan antarlapis antara WC-Co dan salutan berlian telah terbukti dapat mengurangkan bahan pengikat kobalt. Dalam kajian ini, salutan nikel-kromium pada karbide melalui proses elektroplating telah dilakukan untuk membolehkan lapisan berlian melekat dengan baik. Langkah pertama dalam eksperimen ini ialah menyalutkan nikel ke atas substrat dalam tiga pembolehubah parameter; suhu larutan, ketumpatan arus dan masa plating. Selepas itu, kromium akan disalutkan ke atas permukaan nikel dalam dua pembolehubah parameter; suhu larutan dan ketumpatan arus. Rancangan faktorial dua level salutan nikel dan kromium pada substrat WC-Co membolehkannya mengenalpasti ketumpatan arus dan masa plating sebagai dua pembolahubah parameter yang mempengaruhi ketebalan salutan nikel dan kromium. Suhu tidak menunjukkan perubahan signifikan kepada ketebalan salutan. Walaubagaimanapun, ia mempunyai kesan yang baik ke atas kekuatan melekat pada salutan nikel dan kromium. Ketumpatan arus telah memberi kesan yang sedikit kepada kekuatan melekat berbanding dengan suhu. Ketumpatan arus pada 4Amp/dm$^2$, suhu larutan sehingga 58ºC dan masa plating selama 26.31minit telah dikenal pasti sebagai parameter yang sesuai untuk elekto plating dalam menghasilkan lapisan nikel senipis 6.3µm yang mempunyai kekuatan melekat yang baik pada substrat WC-Co manakala ketumpatan arus pada 12Amp/dm$^2$, suhu larutan pada 42ºC dan masa plating selama 15 minit dikenalpasti sesuai untuk parameter elektroplating yang menghasilkan lapisan kromium senipis 2.4µm yang baik kekuatan melekatnya pada salutan nikel substrat WC-Co.

# TABLE OF CONTENTS

# LIST OF TABLES

# LIST OF FIGURES

# LIST OF SYMBOLS

| | | |
|---|---|---|
| A | - | First factor or input variable investigated – bath temperature (ºC) |
| Adeq. precision | - | Adequate precision |
| Adj. $R^2$ | - | Adjusted R-square |
| B | - | Second factor or input variable investigated – current density ($Amp/dm^2$) |
| C | - | Third factor or input variable investigated - plating time (min) |
| Cor. Total | - | Totals of all information corrected for the mean |
| CV | - | Coefficient of variation |
| d.f. | - | Degrees of freedom |
| Pred. $R^2$ | - | Predicted R-square |
| Prob > F | - | Proportion of time or probability you would expect to get the stated F value |
| PRESS | - | Predicted residual error sum of squares |
| R1 | - | Coating thickness response (µm) |
| R2 | - | Adhesion-test time response (second) |
| $R^2$ | - | Coefficient of determination |
| S.D. | - | Square root of the residual mean square |

# LIST OF APPENDICES

# CHAPTER 1

# INTRODUCTION

Diamond has always been a material of intense interest for scientists due to its very strong chemical bonding. The structure of diamond leads to special mechanical and elastic properties. The hardness, molar density, thermal conductivity, sound velocity, and elastic module of diamond are the highest of all known materials while its compressibility is the lowest of all materials. Diamond also has the largest Young's modulus among all materials. The dynamic friction coefficient of diamond is the lowest among the materials of interest. Diamond possesses the highest thermal conductivity ever known. By virtue of its excellent hardness and low coefficient of friction, diamond can be used as cutting tools (Lee *et al.*, 1999).

## 1.1 Background of the Study

Major problems in using natural diamond for many applications are its high cost and availability only in small size and quantity. In addition to overcome these problems, scientists have been trying to develop a synthetic route to diamond production that would produce diamond crystals comparable in quality to natural diamond. Initial efforts were focused on developing synthetic diamond by compressing carbon in a High-Temperature (1550–2250 $^{\circ}$C) and High-Pressure (50,000–100,000 atmosphere) cell, (HPHT). This technique requires massive equipment and is very expensive. Besides being expensive, these techniques can only produce bulk crystals or powders, which further limit the applications of the HPHT synthetic diamond (Liu and Dandy, 1995).

Because of such problems, scientist tried to use another technique for producing high-quality diamond and in the last twenty years, chemical-vapour deposition (CVD) technique was found. The CVD process permits use of near net-shape techniques to produce components that do not require extensive post deposition fabrication. Large-scale capability reduces CVD diamond cost and makes it a more attractive material for use in different applications.

The realization of deposition of diamond films on various kinds of substrates has great impact on the thin-film and coating technologies essential for improving surface quality in many industrial applications. The quality of the film and coating can, to some extent, determine the performance, and even the lifetime of a device. Within the last decade, a number of low-pressure diamond synthesis techniques have been developed. These techniques can be grouped into two major categories: thermally activated CVD and plasma activated CVD. A sound understanding of the nature of the CVD process, a better control of the diamond deposition to reduce or eliminate structural imperfections in the films to improve the quality of diamond films, and an increase of growth rates and deposition areas are some important areas requiring further investigation.

## 1.2 Background of Problem

The diamond coating applied should have good adhesion of the diamond film to the substrate in addition to increase the wear resistance, provide uniform coverage over flank and rake regions to ensure a stable machining process, and result in uniform grain size with low surface roughness to minimize the built-up edge effect. All of the above have to be achieved at an acceptable growth rate.

Cemented tungsten carbide (WC-Co) cutting tools are the workhorse of the metal cutting industry because of their high wear resistance and fracture toughness properties. However, these tools wear rapidly when machining abrasive high Al-Si alloys and glass-epoxy composites. The application of surface overlay coatings to enhance the overall machining effectiveness of WC-Co by reducing the tools downtime; increasing the cutting productivity and improving the quality of the machined surface are becoming more and more attractive (Polini and Barletta, 2008). Polycrystalline diamond (PCD) tools on a WC-Co substrate were generally used for this purpose. However, because of their high cost, many researcher and industrial users are instead using diamond films deposited using CVD techniques which are relatively inexpensive, and could be deposited on tools of any geometry. Compared to an uncoated cemented carbide tool, the CVD diamond coated tool shows much greater abrasive wear resistance which results in up to ten times longer tool life, and less build-up edge and lower cutting forces which yield a better surface finish on the workpiece materials (Lee, 1998).

However, one of the largest barriers to be overcomed is the poor adhesion of diamond film on the cemented carbides substrate. This is due to (i) the large thermal mismatch of the diamond film with the cemented carbide tool and (ii) weak interface bonding resulting from graphite film formation during low-pressure diamond deposition due to the presence of the cobalt binder. There are many reports, which claim to reduce the thermal mismatch and to limit the graphite formation by etching cobalt and/or depositing interlayer as diffusion barriers. The Co from the hard metal is relatively reactive under diamond deposition conditions while the WC is more or less inert. In fact, during the initial steps of high temperature diamond deposition process, the leakage of cobalt from the Co-cemented carbide catalyzes the formation of interfacial $sp^2$-carbon. This weak graphitic layer at the interface results in poor adhesion between the diamond coating and the substrate (Kamarajugadda, 1990).

## 1.3 Statement of Problem

Pre-treatment of the substrate, such as heat treatment and etching with chemical solutions, and use of interlayer play important role in determining the adhesion of the diamond coating onto hard metal and the final properties of coated parts. The negative activity of the cobalt and thermal mismatch must be reduced in order to get a good adhesion of the diamond coating on the hard-metal substrate.

Using the etching process to avoid the negative effects of Co lead to corrosion layer, deep pits of deficient Co on the substrate surface and increase the roughness of surface, which directly leads to the reduction of the mechanical properties and applied range of the cutting tools. For this reason, these problems, this study focuses on using interlayer as the cobalt diffusion barrier.

Many researchers have used physical vapour deposition (PVD) and CVD techniques for coating interlayer on WC-Co. The problems for using these techniques are their high capital and maintenance cost and coating interlayer on substrate in high temperature (around 800 °C especially in CVD technique) leads to the migration of cobalt to the interlayer material. Because of these problems, this study is applied a novel technique for coating interlayer on substrate which is much more cheaper than PVD and CVD technique and also coating process can be done at very low temperature (around 50-70°C) which is very useful for preventing the migration of cobalt to the interlayer material. This technique involves the electroplating process whose usefulness for the coating of material has been proved.

## 1.4 Objective of the Study

i. To analyze the thickness and adhesion of nickel coating on WC-Co substrate via the electroplating process prior to chromium electroplating

ii. To analyze the thickness and adhesion of chromium coating on nickel coated WC-Co via the electroplating process prior HFCVD diamond coating process

iii. To achieve the thickness of nickel-chromium coating below than 10μm

## 1.5 Scope of the Study

i. The substrate material is limited to tungsten carbide (WC) with 6wt% of Co.

ii. The coating materials are limited to nickel and chromium and the combined thickness must be below 10μm.

iii. DOE technique and Design Expert software, are used to analyze the thickness and adhesion of the coatings.

## 1.6 Thesis Layout

This thesis consists of five chapters. Chapter 1 is the introduction of this study. Backgrounds of the study, background of problem, statement of problem, objective of the study, scope of the study and thesis layout are presented in this chapter.

Chapter 2 contains the literature review of the DOE techniques, electroplating technique, and pre-treatment methods. In the electroplating process the focus is on the nickel and chromium electroplating process. Several solutions which have been used for etching the substrate surface and also several materials for interlayer are reviewed.

Chapter 3 is concerned with the research methodology for this study. In this chapter, the experimental steps from cutting the raw material until quantitative analyzing are discussed in detail.

Chapter 4 is concerned with the analysis of the result using the Design Expert Software. In this chapter, the optimum conditions for variable parameters of nickel and chromium electroplating processes have been found.

Chapter 5 summarizes the conclusions and results of experiments in this project and some recommendations for future work are also made.