UNIVERSITI TEKNIKAL MALAYSIA MELAKA

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EFFECT OF CHEMICAL SURFACE TREATMENT OF PVD TiN COATED WC ON THE COATING ADHESION

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EFFECT OF CHEMICAL SURFACE TREATMENT OF PVD TiN COATED WC ON THE COATING ADHESION

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DECLARATION

I declare that this thesis entitle "Effect of Chemical Surface Treatment of PVD TiN Coated WC on the Coating Adhesion" is the result of my own research except as cited in the references. The thesis has not been accepted for any degree and is not concurrently submitted in candidature of any other degree.

Signature : ....

Name : KHAIRUL IZANI BIN MOHD ZUKEE

Date : 24/09/2014

APPROVAL

I hereby declare that I have read this thesis and in my opinion this thesis is sufficient in terms of scope and quality for the award of Master of Science in Manufacturing Engineering.

Signature : ....

Supervisor Name : Md Nizam Abd Rahman

Date : 24/09/2014

DEDICATION

To my beloved parents Mohd Zukee bin Mohd Ragam and Nurlina.

Dedicated to my beloved brothers and sister.

Dedicated to all my family and my friends.

Thank you for your support and encouragement.

You all are everything for me.

May Allah bless all of us. InsyaAllah.

ABSTRACT

Substrate surface treatment prior to the coating deposition is very important in order to obtain good coating adhesion. There are various substrate treatment methods such as polishing, grinding, water peening, and chemical treatment carried out in order to modify the surface characteristics of the substrate. There are quite abundant of research have been done by previous researchers on the substrate surface treatments and one of the methods used was chemical treatment using alkaline and acid solution which was typically performed on tungsten carbide (WC) prior to the deposition of diamond coatings by chemical vapour deposition (CVD). However, study on the effect of substrate chemical treatment using alkaline and acid solution on WC prior to the PVD TiN coating deposition on the coating adhesion is lacking. Adhesion between deposited coatings and substrate is one of the most important success criteria of coating process. The aim of this research is to determine the effect of substrate chemical treatment method using ultrasonic cleaner on the adhesion of TiN PVD coated tungsten carbide. The input parameters evaluated were types of solutions and treatment time. While, the output responses evaluated were surface energy, cobalt content, surface roughness, and adhesion. The characterizations were carried out using sessile drop, SEM/EDX, AFM and Rockwell indenter. The experimental approach adopted was general factorial design. Minitab version 16 software was utilized to analyze the collected data. The chemical treatment using acid for 20 minutes was found to improve the coating adhesion due to formation of uniform peaks and valleys surface morphology. It was found that the coating adhesion was dependent on both surface morphology and surface roughness of the substrate. The cobalt content on the surface layer seems to affect the coating adhesion as well. It was concluded that the substrate surface characteristics were closely related to the performance of the TiN coated inserts.

ABSTRAK

Rawatan permukaan substrat sebelum proses pemendapan salutan adalah sangat penting untuk mendapatkan lekatan yang baik. Terdapat pelbagai kaedah rawatan substrat seperti penggilapan, pencanaian, penembakan air dan rawatan kimia yang dijalankan untuk mengubah ciri-ciri permukaan substrat sebelum proses pemendapan salutan. Terdapat banyak kajian yang dijalankan sebelum ini terhadap rawatan permukaan substrat dan salah satu kaedah yang digunakan adalah rawatan kimia menggunakan larutan alkali dan asid yang biasanya dilakukan pada tungsten karbida (WC) sebelum pemendapan salutan berlian menggunakan deposisi wap kimia (CVD). Walau bagaimanapun, kajian tentang kesan rawatan kimia terhadap substrat menggunakan larutan alkali dan asid pada substrat tungsten karbida sebelum deposisi wap fizikal (PVD) salutan titanium nitrida terhadap kekuatan salutan adalah kurang. Kekuatan lekatan antara salutan dan substrat merupakan salah satu kriteria penting dalam menentukan kualiti salutan tersebut. Tujuan kajian ini adalah untuk menentukan kesan kaedah rawatan kimia terhadap substrat tungsten karbida menggunakan pembersih ultrasonik terhadap kekuatan lekatan salutan titanium nitrida. Input parameter yang dinilai adalah jenis larutan dan tempoh rawatan. Manakala output parameter yang dinilai adalah tenaga permukaan, kandungan kobalt, kekasaran permukaan, dan kekuatan lekatan. Analisa pencirian telah dijalankan menggunakan titisan sessile, SEM/EDX, AFM, dan indentasi Rockwell. Pendekatan eksperimen yang digunakan adalah faktorial umum menggunakan perisian Minitab versi 16 untuk menganalisis data yang diperolehi. Rawatan kimia menggunakan asid selama 20 minit didapati meningkatkan lekatan salutan disebabkan pembentukan puncak dan lembah yang sekata. Lekatan salutan didapati bergantung kepada morfologi permukaan dan kekasaran permukaan substrat. Kandungan kobalt pada permukaan substrat juga didapati mempengaruhi kekuatan lekatan salutan. Sebagai kesimpulannya, ciri-ciri permukaan substrat mempengaruhi prestasi salutan titanium nitride ke atas tungsten karbida.

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LIST OF ABBREVIATIONS

AFM	-	Atomic force microscopy
Al_2O_3	-	Aluminium oxide
ANOVA	-	Analysis of variance
Ar	-	Argon
C	-	Carbon
Co	-	Cobalt
Cr	-	Chromium
CVD	-	Chemical vapour deposition
DC	-	Direct current
DI	-	Deionized water
DOE	-	Design of experiment
EDX	-	Energy dispersive X-ray
L_c	-	Critical load
N_2	-	Nitrogen
OFAT	-	One factor at a time
OM	-	Optical microscope
PVD	-	Physical vapour Deposition
R^2	-	Coefficient of determination
RF	-	Radio frequency
RSM	-	Response surface methodology
SEM	-	Scanning electron microscopy
TiAlN	-	Titanium aluminium nitride
Ti	-	Titanium
TiN	-	Titanium nitride
VDI	-	Verein Deutscher Ingenieure
W	-	Tungsten
WC	-	Tungsten Carbide

XRD	-	X-ray diffraction
γ	-	Surface energy
γ_s	-	Surface tension of solid
γ_l	-	Surface tension of liquid
γ_{sl}	-	Interfacial tension of solid/liquid
γ^p	-	Polar component of surface tension
γ^d	-	Dispersion component of surface tension

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CHAPTER 1

INTRODUCTION

1.1 Background

In today's modern manufacturing environment, there is a continually expanding interest for cutting tools within the last phases of manufacturing operations (Gregor *et al.*, 2006). Tool coatings technology had been exploited commercially since 1980s in cutting tool application due to its ability to improve the surface properties of the cutting tool while maintaining its bulk properties (Kim *et al.*, 2003). The application of thin film coatings on cutting tools is to prolong the cutting tool life by improving the surface properties of the tool (Tshinjan *et al.*, 2012; Haubner *et al.*, 2002; Dobrzański *et al.*, 2006). The improved performance of coated cutting tools has been proven and documented. Some of the published works that support this claim are Kim *et al.*, (2003); Nizam *et al.*, (2010); Viana and Machado, (2009); Chokwatvikul *et al.*, (2011). According to Czelle and Barimani (1995), coating on cutting tool, prevents the contact between the cutting tool base material and workpiece, thereby increasing the life of the tools. Research done by Dobrzański and Żukowska (2007) indicated that coated tool lasted twenty times longer than uncoated tool.

One of the common techniques used to deposit hard coatings on cutting tool is physical vapor deposition (PVD). The utilization of PVD coatings in tribological applications comes to be more across the broad. Titanium nitride (TiN) coatings is one of the most used and studied material due to its high hardness (Kim *et al.*, 2009; Jaya *et al.*, 2013), highly resistant to corrosion (Kim *et al.*, 2012), and exhibits low wear rate (Yang *et al.*, 2000; Jaya *et al.*, 2013).

One of the important success criteria of coating process is the adhesion between the deposited coatings and the cutting tools (Viana and Machado, 2009; Kakaš *et al.*, 2011; Sonoda *et al.*, 2001). The adhesion strength is influenced by the coating process parameters. In spite of optimized coating parameters, deposited PVD coatings failed because of insufficient surface properties of the substrate. In order to modify the surface properties, the substrate surface treatment is very important prior to coating deposition. There were various surface treatment methods studied by the previous researchers as reported in the literature including mechanical and chemical treatment.

Extensive research has been carried out by previous researchers on those surface treatment methods to determine their effect on the coating adhesion. In these studies, chemical treatment is mostly used for the removal of the Co content and the modification of the surface properties mainly for the deposition of diamond coatings using CVD deposition on WC (Norafifah *et al.*, 2013; Sarangi *et al.*, 2008; Lu *et al.*, 2006; Zhang *et al.*, 2000; Buck and Deuerler, 1998; Deuerler *et al.*, 1996). However, there is lack of study on the effect chemical treatment on the tungsten carbide (WC) prior to the deposition of TiN coating using PVD machine. In addressing this gap, this study look into the effect of various chemical treatment parameters (types of solution and treatment time) on the coating adhesion of PVD TiN coated WC substrate.

1.2 Problem Statement

Coatings must be firmly adhered to the substrate to perform its functions. Therefore, proper adhesion strength must be achieved. This will definitely enhanced the life of the coated material as the coating failures can be minimized. Substrate surface treatment is an important stage affecting the performance of coating adhesion (Carreras *et al.*, 2003). Coating adhesion on substrate can be influenced by various factors including the substrate surface treatment methods (Bouzakis *et al.*, 2010; Tönshoff and Mohlfeld, 1998; Ahn *et al.*, 2007; Huang *et al.*, 2011) and coating materials (Jakubeczyova *et al.*, 2011; Lugscheider and Bobzin, 2001; Kong *et al.*, 2008). There are quite abundant of research have been done by previous researchers on the substrate surface treatments and one of method have been used was chemical treatment using alkaline and acid solution which is typically performed on WC prior to the deposition of diamond coatings using chemical vapour deposition (CVD). However, study on the effect of substrate chemical treatment using alkaline and acid solution on WC prior to the PVD TiN coating deposition is still lacking. The relationship between coatings adhesion with other process parameters such as solutions (alkaline and acid) and time is unknown. The main aim of this research is to study the influence of those parameters on the coating adhesion of TiN coating deposited on the WC substrate using PVD.

1.3 Objectives of Study

The main aim of this research is to determine the effect of chemical surface treatment on coating adhesion. The specific objectives of this study are:

i. To study the effect of alkaline and acid solutions during chemical treatment on coating adhesion.
ii. To identify the effect of chemical treatment time on the coating adhesion.
iii. To explain the effect of chemical treatment with respect to surface energy, Co content and surface roughness.

1.4 Scopes of Study

This research focused primarily on the effect of chemical surface treatment on the coating adhesion using alkaline and acid solutions. The coating material and substrate materials used in this research were TiN and tungsten carbide (WC) respectively. In this research, the substrate went through the surface treatment process using ultrasonic cleaning machine. The types of solutions used and time were the independent process variables evaluated. Output responses evaluated were surface roughness, surface energy, cobalt contents, and coating adhesion. The collected data were analyzed using the Minitab version 16 software.

1.5 Significance of Research

Chemical surface treatment in modifying surface properties has generated interest in understanding the behavior of alkaline and acid solutions. Mechanical treatments during pretreatment on tungsten carbide are well documented. However, very little has reported on the effect of chemical treatment solutions during pretreatment prior to PVD coating. It is hoped that comprehensive study on this research will enrich knowledge and understanding of chemical surface treatment parameters. This study also expects that chemical surface treatment could improve surface properties of substrate which is potential to save considerable cost through the improvement of the tool life of the cutting tool due to low wear rate. Thus, through the improvement of tool life, the reduction in tooling cost is expected as well. Hence, the machining time for each process could be reduced as well and increased in productivity could be attained.

1.6 Thesis Layout

This thesis consists of five main chapters. It emphasizes on the effect of substrate surface treatment methods on the coating adhesion. First chapter describes about the background of study, problem statement, objectives, scope and the significance of the research. Chapter 2 explains about the comprehensive literature review related to the study which includes the fundamentals of the coating technologies, deposition process, coating adhesion, substrate surface treatments, characterization methods, and design of experiment. Chapter 3 focuses on detailed experimental procedures for this study. It includes the substrate chemical treatment method, coating deposition machine details, and characterization techniques used (e.g. SEM/EDX, Rockwell indenter, and AFM). Chapter 4 described the results of the experiment including discussions of the collected result. Conclusions of the research and recommendations are discussed in the last chapter.