NANOCRYSTALLINE DIAMOND DEPOSITED ON TUNGSTEN CARBIDE-COBALT SUBSTRATES USING HOT FILAMENT CHEMICAL VAPOUR DEPOSITION TECHNIQUE

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A thesis submitted in fulfilment of the
requirements for the award of the degree of
Doctor of Philosophy (Mechanical Engineering)

Faculty of Mechanical Engineering
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OCTOBER 2015

ABSTRACT

Diamond coatings on cutting tools provide the advantages of the properties of diamond in terms of high hardness, excellent wear resistance, and general chemical inertness. The main limitations of diamond coating are coating adhesion problems, high surface roughness and low production rate due to the use of coating equipment with small chamber. There is also a need to have a better understanding on the growth mechanism of diamond coatings on tungsten carbide with cobalt binder (WC-Co). Thus the motivation for this work is to obtain low surface roughness of diamond coatings while achieving good adhesion to substrates through the use of large chamber hot filament chemical vapour deposition (HFCVD) method. In this research, nanocrystalline diamond (NCD) coatings were deposited on WC-Co substrates using the HFCVD method. WC-Co was selected because it is used widely in the current tooling market. The cutting method was varied between precision cutting and electrical discharge machining EDM (Wire-Cut). It was found that precision cutting produce generally planar substrates and was the preferred method of cutting. To achieve good adhesion between the diamond coating and WC-Co substrates, the WC-Co substrates were pretreated before being deposited with diamond and some of these pretreatments parameters were varied. It was found that 20 minutes of Murakami agent treatment, 45-60 seconds of acid etching and <0.25 μm natural diamond seeding in ultrasonic bath were the best pretreatment method. The substrates were then deposited with diamond in the HFCVD chamber. Four batches of deposition were ran namely batch A, B, C and D. The overall results show that the deposited diamonds were nanocrystalline in size with cauliflower or ballas NCD morphology with various crystallite arrangements. Batch A produced generally four different types of morphologies. Type 1 was planar diamond coating morphology attributed to precision cutting effects. Type 2 was planar diamond coating morphology with micro features attributed to rough bench grinding. Type 4 was the extension of type 3 where EDM cut produced about a third tier morphology separating islands of diamond ballas aggregates. Three-tier ballas morphology improves the adhesion property where boundaries hinder failure path of the diamond coating. Batch B produced an obvious microcrystalline diamond layer under the NCD layer. Batch C produced a single layer of very thin NCD layer of only 1.7 microns. Batch D produced a layer NCD coating of about 4 microns thick by changing oxygen pulsing rate and time. X-ray diffraction (XRD) and grazing XRD showed that the diamond layer was in compression. Raman Spectrometer identified the presence of NCD. Atomic force microscope (AFM) showed the ultra-low roughness of the diamond coatings with *Ra* <200 nm. Nanoindentation revealed that the NCD coating has high hardness of 10-60 GPa and reduced modulus of 40 – 300 GPa. The adhesion strength is good as indicated from the indentation test. Electron microscopy results showed the ballas consist of elongated radial grains in accordance to the thickness of the NCD coating. Further magnifications revealed diamond twins that contributed to the properties and nano size of diamond crystallites. Transmission electron microscopy analyses also indicate that the NCD nucleated and grew on the tungsten carbide (100) planes in the ⟨111⟩ direction, forming (111) planes.

ABSTRAK

Salutan intan ke atas alat pemotong memberikan kelebihan sifat intan dari aspek kekerasan tinggi, rintangan haus yang sangat baik dan kelengaian kimia umum. Had utama salutan intan adalah masalah lekatan dengan substrat, kekasaran permukaan yang tinggi dan kadar pengeluaran yang rendah disebabkan oleh penggunaan peralatan salutan berkebuk kecil. Terdapat juga keperluan untuk memahami mekanisma pertumbuhan salutan intan ke atas substrat tungsten karbida dengan pengikat kobalt (WC-Co). Oleh itu motivasi untuk menjalankan kerja ini adalah untuk menghasilkan salutan intan berkekasaran rendah dengan kekuatan lekatan kepada substrat yang baik menggunakan alat pengendapan wap kimia filamen panas (HFCVD) berkebuk besar. Dalam kajian ini, salutan intan nanokristal (NCD) telah diendapkan ke atas substrat WC-Co menggunakan kaedah HFCVD. WC-Co dipilih kerana ianya diguna secara meluas dalam pasaran alat pemotong semasa. Substrat dipotong dengan kaedah pemotongan persis atau kaedah pemotongan nyah-cas elektrik (EDM) (wayar). Didapati pemotongan persis menghasilkan substrat yang rata dan dipilih sebagai kaedah pemotongan yang lebih sesuai. Untuk mendapatkan lekatan yang baik, substrat WC-Co telah dipra-rawat sebelum intan diendap dan parameter pra-rawatan dipelbagaikan. Didapati bahawa dengan rawatan Murakami ejen selama 20 minit, punaran asid selama 45-60 saat dan pembenihan dengan intan semulajadi bersaiz <0.25μm di dalam mandian ultrasonik adalah kaedah pra-rawatan terbaik. Selepas itu intan diendapkan ke atas substrat di dalam kebuk HFCVD. Sebanyak empat kelompok endapan telah dijalankan dan dinamakan kelompok A, B, C dan D. Keputusan secara keseluruhan menunjukkan bahawa intan yang diendapkan bersaiz nano dengan morfologi kubis bunga atau *ballas* NCD dengan pelbagai susunan kristalit. Kelompok A menghasilkan empat jenis morfologi. Jenis 1 adalah lapisan intan bermorfologi rata disebabkan oleh kesan pemotongan persis. Jenis 2 adalah seperti jenis 1 tetapi berciri mikro disebabkan oleh kesan kisaran kasar daripada pengisar meja. Jenis 4 yang merupakan lanjutan morfologi jenis 3 yang dihasilkan oleh kesan pemotongan EDM. Potongan EDM ini menghasilkan morfologi berperingkat tiga yang melibatkan pemisahan agregat-agregat *ballas*. Morphologi ini membaiki sifat lekatan dengan cara memberhentikan kemajuan laluan kegagalan di sempadan agregat *ballas*. Kelompok B menghasilkan lapisan intan mikrokristal di bawah lapisan NCD. Kelompok C menghasilkan lapisan tunggal NCD yang sangat nipis iaitu hanya 1.7 μm. Kelompok D menghasilkan lapisan salutan NCD yang tebalnya lebih kurang 4 mikron dengan memendekkan masa pengendapan dan menukar kadar denyutan dan masa oksigen. Pembelauan sinar-X (XRD) dan XRD geseran menunjukkan lapisan intan dalam keadaan mampatan. Spektrometer Raman mengenalpasti kehadiran NCD. Mikroskop daya atom menunjukkan kekasaran permukaan yang teramat rendah, *Ra* <200nm. Pelekuk nano menunjukkan salutan intan mempuyai kekerasan yang sangat tinggi, 10-60 GPa dan modulus terkurang 40 – 300 GPa. Kekuatan lekatan adalah baik sebagaimana ditunjukkan oleh ujian lekukan. Keputusan mikroskopi elektron menunjukkan intan bermorfologi *ballas* terdiri daripada bijian jejari memanjang selari dengan ketebalan salutan NCD. Pembesaran seterusnya mendedahkan kembar intan yang menyumbang kepada sifat dan saiz nano kristalit intan. Analisis mikroskop elektron transmisi juga menunjukkan bahawa intan NCD tumbuh dan membesar ke atas satah (100) tungsten karbida pada arah <111> dan membentuk satah (111).

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LIST OF ABBREVIATIONS

AFM	-	Atomic Force Microscope
BEN	-	Bias Enhanced Nucleation
CVD	-	Chemical Vapour Deposition
EDM	-	Electrical Discharge Machining
FIB	-	Focus Ion Beam Milling
HFCVD	-	Hot Filament Chemical Vapour Deposition
HPHT	-	High Pressure High Temperature
HRTEM	-	High Resolution Transmission Electron Microscope
MCD	-	Microcrystalline Diamonds
MEMS	-	Microelectromechanical Systems
MPCVD	-	Microwave Plasma Chemical Vapour Deposition
NCD	-	Nanocrystalline Diamonds
PVD	-	Physical Vapour Deposition
RMS	-	Root Mean Square
SAD	-	Selected Area Diffraction
SEM	-	Scanning Electron Microscopy
SIMS	-	Secondary Ion Mass Spectrometry
TEM	-	Transmission Electron Microscope
UNCD	-	Ultra Nanocrystalline Diamonds
XRD	-	X-Ray Diffraction
XRF	-	X-Ray Fluorescence

LIST OF SYMBOLS

'	-	minute (degree)
%	-	percent
°	-	degree
°C	-	degree Celsius
A	-	Ampere
Å	-	Angstrom
at.%	-	atomic percent
atm	-	atmosphere
bar	-	bar (10^5 Pascal)
cm	-	centimetre
eV	-	electron Volt
g	-	gram
GPa	-	Gigapascal
hr	-	hour
J	-	Joule
K	-	Kelvin
keV	-	kilo-electron Volt
kg	-	kilogram
kJ	-	kilojoule
kPa	-	kilopascal
kV	-	kilovolt
l	-	liter
m	-	meter
mbar	-	milli-bar
min	-	minute
ml	-	millilitre
mln/minute	-	milliliter normal per minute

mm	-	millimetre
kPa	-	kilopascal
MPa	-	Megapascal
N	-	Newton
nm	-	nanometer
Pa	-	Pascal
pm	-	picometer
s	-	second
V	-	Volt
vol.%	-	volume percent
W	-	Watt
wt.%	-	weight percent
α	-	growth parameter
Δa	-	slope angle
θ	-	Angle
θ_B	-	Bragg's angle
λ	-	wavelength
λc	-	Cut-off value
μm	-	micrometer

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CHAPTER 1

INTRODUCTION

1.1 Background of Research

Diamond is a wonder of nature and has properties exceeding other materials such as extreme hardness, high thermal conductivity and wear resistance; and low coefficient of friction and thermal expansion, which make it a very effective cutting material. Deposition of microcrystalline diamond (MCD) films via Chemical Vapour Deposition (CVD) method has been progressively improved over the past 30 years of research [1] but three main problems remains for tungsten carbide with cobalt binder (WC-Co) substrates, 1. Poor adhesion to the WC-Co substrate due to difference in coefficient of thermal expansion of diamond and WC-Co, with the cobalt binder phase is a graphite forming promoter, 2. Low diamond growth rates in compromise to quality crystalline grains, and 3. Surface roughness of the diamond thin films [2, 3]. High surface roughness will cause high tool wear and shorter tool life, in addition to affecting machining precision [4] and give poorer surface finish [5]. While various polishing methods for diamond has been introduced, diamond, being the hardest material on earth, makes its polishing process costly and time consuming. Thus, depositing low roughness films by reducing the diamond crystals to nano-size becomes important.

Nanocrystalline diamonds (NCD) has first made its appearance 14 years ago using Hot Filament Chemical Vapour Deposition (HFCVD) method [6]. NCD has superior properties in comparison to MCD of having roughness of a few orders of magnitude lower while retaining the excellent properties of diamond. Much research has been done on depositing NCD on various types of substrates. Many recent researches have contributed on HFCVD deposition parameters for NCD deposition [7].

Diamond coated tungsten carbide with cobalt binder (WC-Co) surpasses uncoated tool performance in machining of materials such as metal matrix composites, carbon fibers, Aluminium-Silicon Carbide (AlSiC), Aluminium-Silicon (AlSi), wood, odontological and micro-machining tools [3, 5, 8-14]. The performance of the WC-Co tool has been increased with the coating of MCD film [12].

1.2 Problem Statement

The two main problems related to diamond deposition which require further research are; 1. Poor adhesion to the WC-Co substrate due to difference in coefficient of thermal expansion of diamond and WC-Co, with the cobalt binder phase is a graphite forming promoter and 2. Surface roughness of the diamond thin films. Low roughness will reduce associated diamond surface wear [9], and increase cutting performance. While various polishing methods for diamond has been introduced, diamond, being the hardest material on earth, has makes its polishing process costly, time consuming and not practical for complex geometry. Thus, the importance of depositing low roughness films by depositing NCD films becomes significant [15].

HFCVD units are normally used for large area MCD coatings. While research units for HFCVD that coats NCD films have relatively small chambers [4, 16] and small filament to substrate distance in comparison with the commercial MCD units, large chamber deposition unit like CC800®/Dia low pressure HFCVD machine are at least 10 times more volume than small chambers that are mostly custom made [17]. Deposition parameters for NCD using large chamber HFCVD differs from that of small research chambers. Thus there is a need to establish deposition parameters for large chamber units. Previous research by Dayangku [18] shows that crystallite size of approximately 70 nm was successfully grown on different substrates using a large chamber HFCVD unit. Work by Izman and colleagues [19, 20] focused mainly on effects of pretreatment towards diamond deposition for large chamber HFCVD unit.

While Hassan and colleagues [21-23] focused on developing an interlayer for diamond deposition for large chamber HFCVD unit. Thus, there is also a need to understand fundamentals of diamond growth mechanism for large chamber HFCVD unit.

The mechanism of growth of NCD using Microwave Plasma Chemical Vapour deposition (MPCVD) method [24] has been well established. However NCD growth mechanism using HFCVD is still under investigations. The main reason underlining this is that the reactions that form NCD in MPCVD involves plasma formed in MPCVD that is not available in HFCVD. Thus much is not understood about the nucleation and growth mechanisms of NCD in a HFCVD reactor. Nevertheless HFCVD is able to deposit diamond films more uniformly than MPCVD as plasma is limited by the plasma ball.

Furthermore, while NCD deposition has been well studied on silicon substrate, and there are many studies for MCD deposition on WC-Co, the subject on NCD-WC-Co boundaries are limited [4]. Another field of research is to explore whether pre-treatment of NCD in large HFCVD units differ to that of NCD deposited in small units.

1.3 Objectives of Research

The objectives of the research are as follows:

1. To perform parametric study on the cutting and substrate pretreatments and to establish deposition parameters to produce strong adhesion of nanocrystalline diamond on tungsten carbide with cobalt binder surface for hot filament chemical vapour deposition method with large reaction chamber.
2. To characterise the deposited diamond coating by evaluating its chemical, morphological and mechanical properties.
3. To determine the characteristics of the bonding interface and growth mechanism of nanocrystalline diamond on tungsten carbide.

1.4 Scopes of the Research

The scopes of the research are as follows;

1. Tungsten carbide substrate sample pretreatment for nanocrystalline diamond deposition.
2. Deposition of thin coalesce nanocrystalline diamond film on tungsten carbide substrate.
3. Material characterisation, surface and cross-section morphology analysis of nanocrystalline diamond film using optical microscope, profilometer, atomic force microscope, scanning electron microscope, Raman spectrometer and X-Ray diffractometer.
4. Adhesion test analysis between diamond and tungsten carbide surface.
5. Characterisation of mechanical properties of nanocrystalline diamond film using nanoindentor.
6. Characterisation of bonding interface between nanocrystalline diamond and tungsten carbide using transmission electron microscope.

1.5 Significance of the Research

This research is expected to produce better and improved adhesion and mechanical properties of NCD coated on WC-Co cutting tool produced by large chamber HFCVD. This may be achieved by varying the deposition parameters and performing pretreatment on the substrate. Analysis of the results will give better understanding on the chemical, morphology and mechanical properties of the NCD that will affect the function of the film as well as better understanding of the NCD-WC interface. This will in turn give better understanding of the nucleation and growth mechanism of the film thus enabling progressive improvements for better diamond coating on cutting tools.

1.6 Organisation of Thesis

This thesis is organised five chapters. Chapter 1 briefly introduces the background of the research, objectives, scopes and significance of the research. Chapter 2 is the literature review. This chapter introduces the subject of study in detail. It deliberates the substrate, the coating, the technique and it is experimental parameters chosen, and the analyses required. This deliberation is based on the works of other researchers.

Chapter 3 is the detailed experimental and analysis methodology. The experiment set up is done in two major phases i.e. the substrate pretreatment phase and the diamond deposition phase. Description of diamond coating preparation for analyses was included.

Chapter 4 portrays the related analytical results to answer the objectives. The results were obtained from Optical Microscope, Scanning Electron Microscope (SEM) and Atomic Force Microscope (AFM) for topographical data. X-Ray Diffraction (XRD) and Raman Spectroscope produced data for chemical identification. Adhesion test was done to qualitatively measure the adhesion strength of the coating to substrate. Nanoindentation test was done to obtain nano-mechanical properties of the coating. Transmission Electron Microscope (TEM) was done to obtain atomic, chemical and morphology of the diamond to substrate bonding interface. All the data were analysed and discuss in depth to satisfy the objectives.

This thesis ends at Chapter 5. Chapter 5 concludes the findings, elaborates the main limitations of the findings and gives recommended work for future.

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