

POLISHING OF POLYCRYSTALLINE

DIAMOND COMPOSITES

by

Yiqing CHEN

B.E. (Shanghai Jiaotong University, China)

MBA (UTS, Australia)

A thesis submitted in fulfilment of

the requirements for the degree of

Doctor of Philosophy

(School of Aerospace, Mechanical and Mechatronic Engineering)

at The University of Sydney

March 2007

Acknowledgements

I would like to thank many people who have made this research project possible. First of all, I would like to express my sincerest appreciation to my supervisor Professor Lianchi Zhang and co-supervisor Dr. Joseph Arsecularatne for their kind guidance, constant encouragement and every support throughout the course of the present study.

I would like to thank Ringwood Diamond Material Technologies Pty. Ltd. for providing the company's support and making all the PCD specimens for this research project.

Thanks are due to the technical officers, Mr. B. Oliver, Mr. G. Cumberland, Mr. T. Shearing, and many technical staff in the workshop and the laboratory for making the polishing machine and help in constructing the experiments. Help from members of staff, Mr. S. Bulcock, Mr. A. Sikorski and Dr. I. Kaplin at the Electron Microscope Unit (EMU), Dr. E. Carter from School of Chemistry, The University of Sydney, is greatly acknowledged.

Thanks are also due to Dr. T. Nguyen, Dr. I. Zarudi, Dr. K. Mylvaganam, Dr. T. Vodenitcharova, Dr. Y. Ali, Dr. K. Xiao and all of my colleagues at the school of AMME for enriching my stay at The University of Sydney.

I would like to thank the Australian Research Council for the financial support and offering an Australian Postgraduate Award (Industry) for this research project.

Finally, my deep thanks to my husband, my daughter and all my family members for their love, encouragement and support. To them I dedicate this thesis.

Declaration

I declare that this thesis contains no material which has been previously presented for the award of any other degree or diploma in any university or institution; and to the best of my knowledge, the material is original except where due reference is made in the text of the thesis.

Yiqing CHEN

Abstract

This thesis aims to establish a sound scientific methodology for the effective and efficient polishing of thermally stable PCD composites (consisting of diamond and SiC) for cutting tools applications. The surface roughness of industrial PCD cutting tools, 0.06 µm Ra is currently achieved by mechanical polishing which is time consuming and costly because it takes about three hours to polish a 12.7 mm diameter PCD surface. An alternative technique, dynamic friction polishing (DFP) which utilizes the thermo-chemical reactions between the PCD surfaces and a catalytic metal disk rotating at high peripheral speed has been comprehensively investigated for highly efficient abrasive-free polishing of PCD composites. A special polishing machine was designed and manufactured in-house to carry out the DFP of PCD composites efficiently and in a controllable manner according to the requirements of DFP.

The PCD polishing process and material removal mechanism were comprehensively investigated by using a combination of the various characterization techniques: optical microscopy, SEM and EDX, AFM, XRD, Raman spectroscopy, TEM, STEM and EELS, etc. A theoretical model was developed to predict temperature rise at the interface of the polishing disk and PCD asperities. On-line temperature measurements were carried out to determine subsurface temperatures for a range of polishing conditions. A method was also developed to extrapolate these measured temperatures to the PCD surface, which were compared with the theoretical results. The material removal mechanism was further explored by theoretical study of the interface reactions under these polishing conditions, with particular emphasis on temperature, contact with catalytic metals and polishing environment. Based on the experimental results and theoretical analyses, the material removal mechanism of dynamic friction polishing can be described as follows: conversion of diamond into non-diamond carbon takes place due to the frictional heating and the interaction of diamond with catalyst metal disk; then a part of the transformed material is detached from the PCD surface as it is weakly bonded; another part of the non-diamond carbon oxidizes and escapes as CO or CO₂ gas and the rest diffuses into the metal disk. Meanwhile, another component of PCD, SiC also chemically reacted and transformed to amorphous silicon oxide/carbide, which is then mechanically or chemically removed.¹

Finally an attempt was made to optimise the polishing process by investigating the effect of polishing parameters on material removal rate, surface characteristics and cracking /fracture of PCD to achieve the surface roughness requirement. It was found that combining dynamic friction polishing and mechanical abrasive polishing, a very high polishing rate and good quality surface could be obtained. The final surface roughness could be reduced to 50 nm Ra for two types of PCD specimens considered from pre-polishing value of 0.7 or 1.5 μ m Ra. The polishing time required was 18 minutes, a ten fold reduction compared with the mechanical abrasive polishing currently used in industry.

¹ A paper based on this research has been granted the Best Paper Award at 8th International Conference on Progress of Machining Technology organized by the Japan Society of Precision Engineering in Shimane, Japan in November 2006.

List of Symbols and Abbreviations

ΔG_T^0	mole Gibbs free energy difference at zero pressure
ħ	Planck's constant
2θ	the angle between the X-ray source and detector
A	nominal area of PCD specimen
A_p	area of the polishing plate swept by the sample
A_s	area of the polished sample's surface
A'	expected actual contact area
ADC	analog-to-digital converter
AFM	atomic force microscopy
AMP	amplified
С	specific heat
CCD	charge-coupled device
C(y)	carbon concentration at distance for interface y
C_o	initial (background) carbon concentration in the polishing plate
	miner (ouenground) encon concentration in the ponoming prace
C_1	interface carbon concentration
C_I C_s	interface carbon concentration carbon concentration at the surface of the polishing plate
C ₁ C _s D	interface carbon concentration carbon concentration at the surface of the polishing plate diffusion coefficient
C_1 C_s D $d_{(hkl)}$	interface carbon concentration carbon concentration at the surface of the polishing plate diffusion coefficient spacing between atomic planes within the sample
C_1 C_s D $d_{(hkl)}$ d	 interface carbon concentration carbon concentration at the surface of the polishing plate diffusion coefficient spacing between atomic planes within the sample specimen diameter

DFP	dynamic friction polishing
D_s	diameter of the shaft, in millimetres
E*	maximum potential energy
е	effective electron charge
E	Young's modulus
E_1	activation energies for transformation of diamond to graphite
E_{-1}	activation energies for transformation of graphite to diamond
E_a	the activation energy
EDM	electric discharge machining
EDX	energy dispersive X-ray analysis
EELS	electron energy loss spectroscopy
EMI	Electro Magnetic Interference
E_p	energy of the bulk Plasmon
erfc	error function
FET	field effect transistor
FWHM	full width at half maximum
$G_{T,P(products)}$	Gibbs free energy of reaction products
$G_{T,P(reactants)}$	Gibbs free energy of reactants
$G_{T,P}$	Gibbs free energy as a function of the pressure-temperature
h	average heat flux
JCPDS	Joint Committee on Powder Diffraction Standards
K	thermal conductivity
L	Load on PCD specimen
L_1	average force on an asperity

LCM	laser confocal Microscopy
LED	light emitting diode
т	effective electron mass
MCA	multi-channel analyzer
Ν	number of asperities
n	number of revolutions per minute (rpm)
n _c	valence electron density (number of valence electrons per unit volume)
Р	normal pressure on PCD specimen
<i>P</i> '	expected applied pressure
P_m	main motor power
PWM	pulse-width modulation
PCD	polycrystalline diamond
R	gas constant (=8.31 J/mol.K)
R_a	PCD asperity radius
R _d	radius of polishing disk
R _p	PCD specimen radius
SEM	scanning electron microscopy
SPM	scanning probe microscope
S_s	allowable torsion shear stress in N/mm ²
STEM	scanning transmission electron microscopy
Т	absolute temperature
t	time
TEM	transmission electron microscopy
T_r	temperature rise

V	sliding speed
VFD	variable frequency drive
XRD	X-Ray diffraction
у	distance from the interface in polishing disk
Z	distance from the polishing surface of PCD specimen
Z	height of asperity
$\Delta G_{T,P}$	Gibbs free energy difference
ΔV	the volume difference between graphite and diamond
\mathcal{E}_0	vacuum dielectric constant
η	density of asperities
Λ	wavelength of X-rays
μ	coefficient of friction
ν	Poisson's ratio
ρ	density of material
σ	standard deviation
χ	thermal diffusivity
ω	speed of rotation
$\Phi(z)$	distribution function of asperity heights

Contents

Acknowledgements	ii
Declaration	iii
Abstract	iv
List of Symbols and Abbreviations	vi
Chapter 1 Introduction	1
1.1 Background	1
1.2 Aim of this thesis	4
1.3 Thesis organization	5
Chapter 2 Overview of diamond polishing techniques	7
2.1 Introduction	7
2.2 Diamond and PCD polishing techniques	9
2.2.1 Mechanical polishing	9
2.2.2 Chemo-mechanical polishing	15
2.2.3 Thermo-chemical polishing	17
2.2.4 Dynamic friction polishing	24
2.2.5 High energy beam polishing	26
2.2.6 Electrical discharge machining	29
2.3. Material removal mechanisms	31
2.3.1 Micro-chipping	32

2.3.2 Phase transformation of diamond to non-diamond carbon	32
2.3.3 Diffusion of carbon into soluble metals	33
2.3.4 Chemical reactions	34
2.3.5 Evaporation and sputtering	35
2.3.6 Summary of polishing pathways	36
2.4 Comparison of various polishing techniques	37
3.5 Current understanding and existing gaps	42
Chapter 3 Characterization Methods	46
3.1 Introduction	46
3.2 Examination of surface roughness and topography	47
3.2.1 Optical microscopy	48
3.2.2 SEM and EDX	48
3.2.3 Surface roughness tester	52
3.2.4 Laser confocal microscopy	53
3.2.5 AFM	55
3.3 Examination of chemical and micro-structure	57
3.2.1 XRD analysis	57
3.3.2 Raman spectroscopy	59
3.3.3 TEM and electron diffraction	65
3.3.4 STEM and EELS	68
3.4 Temperature measurement	71
3.4.1 Principle of thermocouple	72
3.4.2 Thermocouple selection	74

3.4.3 Precautions and considerations for using thermocouples	76
3.5 Summary	77
Chapter 4 Design and manufacture of a polishing machine	79
4.1 Introduction	79
4.2 Design requirement and design plan	80
4.3 Design of polishing machine	82
4.3.1 Structure	82
4.3.2 Polishing disk	84
4.3.3 Motors and their controllers	86
4.4 Manufacture and assembly of the machine	90
4.5 Machine calibration	91
4.5.1 Load	91
4.5.2 Speed	94
4.6 Summary	95
Chapter 5 Experimental investigation of material removal mechanisms	96
5.1 Introduction	96

5.1 Introduction	96
5.2 PCD Material	98
5.3 Experiments	101
5.4 Surface generation	103
5.5 Composition and structural analysis of PCD specimens	105
5.5.1 SEM and attached EDX	105
5.5.2 XRD	107

5.5.3 Raman spectroscopy	108
5.6 Polishing debris analysis	112
5.6.1 SEM and EDX	112
5.6.2 XRD	115
5.6.3 Raman spectroscopy	116
5.6.4 TEM, electron diffraction, EDX and EELS analysis	118
5.7 Summary of the dynamic friction polishing process	130

Chapter 6 Surface temperature in polishing 1	32
6.1 Introduction 1	132
6.2 Modelling 1	133
6.2.1 Statistical model of PCD surface roughness 1	133
6.2.2 Temperature rise 1	136
6.3 Experimental measurement of surface topography 1	139
6.4 Predicted results and discussion 1	142
6.5 Comparison with literatures 1	147
6.6 On-line temperature measurement 1	49
6.6.1 Experimental set up 1	150
6.6.2 Measured results and discussion 1	152
6.6.3 Extrapolation of the measured temperature to the surface 1	155
6.7 Discussion	56
6.8 Conclusions 1	158

Chapter 7 Theoretical study of material removal mechanisms	159
7.1 Introduction	159
7.2 Reaction of carbon with transition metals	160
7.3 Structural transformation of carbon	163
7.4 Oxidation of carbon	170
7.5 Diffusion of carbon into the metal	175
7.6 Summary of polishing mechanism	177
Chapter 8 Parameter study – towards the optimization of polishing	181
8.1 Introduction	181
8.2 Experiments	182
8.2.1 PCD material	182
8.2.2 Polishing machine and experiments	183
8.2.3 Characterization of specimen surface and material removal	183
8.3 Effect of DFP parameters on material removal and cracks generated	184
8.3.1 Polishing time	184
8.3.2 Pressure	187
8.3.3 Sliding speed	190
8.3.4 Other factors – test sequence	193
8.4 Surface topography	195
8.5 Optimization of polishing for required surface finish	201
8.5.1 Polishing parameters for crack free surface	201
8.5.2 Optical surface finish	203
8.6 Conclusion	206

Chapter 9 Conclusions and Recommendation	209
9.1 Conclusions	209
9.2 Suggestions for future work	212
References	213
Appendix	236