APPLICATION OF DESIGN OF EXPERIMENT TECHNIQUES IN THE TWO-STEP PRETREATMENT PROSES FOR DIAMOND COATING

NORAFIFAH BINTI HUSIN

UNIVERSITI TEKNOLOGI MALAYSIA

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To mak, ayah, family and friends…

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ABSTRACT

Chemical vapor Deposition (CVD) is one of diamond synthesis process and it is widely applied to cutting tools to enhance wear resistance and increase tool life. One of the methods of ensuring adhesion of diamond on the substrate is through the roughening and the substrate surface and the removal of cobalt at the substrate surface. This method is applied during the pretreatment method, which is before the coating process. In this study, Murakami etchant was used to roughen the surface and this is followed by an acid treatment to remove cobalt at the Tungsten Carbide (WC-6% Co) surface. Concentration of both solutions has been varied. Design of Experiment Techniques has been used to determine the optimum combination of the factor that has been investigated. Result from this study shows the optimum condition for maximizing the surface roughness is 10.23g of potassium ferricyanide, 29.92g of potassium hydroxide and etching time in ultrasonic bath of 19.74 minutes while the optimum concentration for acid treatment is 60.36% for nitric acid, 10.29% of hydrogen peroxide and 89.43 seconds for etching time.

ABSTRAK

Chemical Vapor Deposition (CVD) merupakan salah satu proses pensintesisan intan dan banyak diaplikasikan kepada alat pemotong untuk meningkatkan daya ketahanan dan jangka hayat alat pemotong tersebut. Salah satu cara untuk meningkatkan daya ketahanan pensintesisan intan bagi sesuatu alat adalah dengan meningkatkan kekasaran permukaan dan menghapuskan kandungan kobalt yang terdapat pada permukaan bahan tersebut. Ini dilakukan semasa proses prarawatan iaitu sebelum proses penyalutan intan dilakukan. Di dalam kajian ini, larutan Murakami digunakan untuk membantu meningkatkan kekasaran permukaan dan diikuti dengan larutan asid untuk menghapuskan kandungan kobalt yang terdapat pada permukaan tungsten karbida (WC-6% Co). Kepekatan kedua-dua larutan ini diubah-ubah. Kaedah Reka Bentuk Ujikaji digunakan untuk menentukan kombinasi yang paling optimum terhadap bahan yang dikaji. Hasil kajian mendapati, kombinasi yang paling optimum untuk meningkatkan kekasaran permukaan adalah 10.23g kalium ferrisianida, 29.92g kalium hidroksida dan masa larutan yang diambil adalah 19.74 minit manakala kombinasi bagi kepekatan larutan asid adalah 60.36% bagi asid nitrik, 10.29% bagi asid hidrogen peroksida dan 89.43 saat masa yang diambil untuk proses larutan.

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A	-	first factor or input variable investigated for alkaline reagent – weight potassium ferricyanide
Adeq. precision	-	adequate precision
Adj. R^2	-	adjusted R^2
B	-	second factor or input variable investigated for alkaline reagent – weight of potassium hydroxide
C	-	third factor or input variable investigated for alkaline reagent - etching time
Cor. Total	-	totals of all information corrected for the mean
CV	-	coefficient of variation d.f. degrees of freedom
D	-	first factor or input variable investigated for oxidizing reagent - concentration of nitric acid
E	-	second factor or input variable investigated for oxidizing reagent – concentration of hydrogen peroxide
F	-	third factor or input variable investigated for oxidizing reagent – etching time
Pred. R^2	-	predicted R^2
Prob. > *F*	-	proportion of time or probability you would expect to get the stated F value
PRESS	-	predicted residual error sumof squares
R_y	-	surface roughness of the turned surface (μ)
R^2	-	coefficient of determination
S.D.	-	square root of the residual mean square

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CHAPTER 1

INTRODUCTION

1.1 Background of the Study

Diamond coatings, applied by the chemical vapor deposition (CVD) process, have unmatched advantages when applied to tungsten carbide cutting tools. Chemical vapor deposition diamond retains the properties of natural diamond. Its ultra-high hardness and elastic modulus result in exceptional dimensional stability and resistance to abrasive wear. Like natural diamond, CVD diamond has low coefficient of friction that results in low cutting forces and low power consumption, low frictional heating and resistance to built-up-edge when cutting. These corrosion-resistant coatings have long lives when they are used to cut corrosive polymers such as the phenolic resins used in composite materials, and they are resistant to the corrosive damage of cutting fluids. Diamond-coated tools excel at machining abrasive nonmetallic materials, nonferrous metals and abrasive nonferrous metals. However, the chemical instability that arises with diamond and metal alloys containing iron, nickel or cobalt limits its use when cutting ferrous alloys and super alloys.

The life of diamond-coated tools depends on the material being cut, the cutting speeds and feeds, and the part geometry. As a rule, graphite diamond-coated tools last 10 times to 20 times longer than bare tungsten carbide tools. This allows for lights-out-operation, and jobs can be machined completely with one tool. Wear and the need to recalibrate tools are reduced. In composite materials, it is not unusual to get longer life. With high-density fiberglass, carbon fiber and G10-FR4, diamond-coated-tool lives of 70 times that of bare carbide have been reported. Because of the long time required to coat tools with diamond, and the pretreatment procedures required to achieve good adhesion, diamond-coated tools are expensive.

Although diamond-coated tools cost about five times as much as good quality carbide tools, they can dramatically reduce overall cost of production because of their broad operating range and long life. For example, one automotive manufacturer machining high-density fiberglass raised productivity from 15 parts per standard $15 end mill to more than 750 parts with a $150 diamond-coated end mill. That saved the company more than $600,000 annually. As aerospace manufacturers increasingly make use of composite materials, engineers are learning that a combination of diamond coating and application-specific tool geometry provides the most efficient machining solution. Composite materials such as high-density fiberglass, carbon fiber and G10-FR4 are very abrasive. Without proper pretreatment for the cutting tool, the abrasiveness of these materials can lead to poor adhesion of the diamond film to the carbide (Jim Benes, 2008).

1.2 Background of Problem

Deposition of diamond films onto tungsten carbide is attractive since it can lead to potential improvements in the life and performance of cutting tools including rotary tools and inserts due to the excellent physical and chemical properties of the coatings. Chemical vapour deposition (CVD) of diamond coating has the potential to considerably prolong the lifetime of WC-Co dental cutting tools when applied to the machining of highly abrasive non-ferrous metallic alloys, borosilicate glass, human teeth and ceramic materials. However, deposition of adherent high quality diamond films onto substrates such as cemented carbides, stainless steel and various metal alloys have proved to be problematic due to the thermal expansion mismatch and the presence of cobalt binder, which provides additional toughness to the tool but causes poor adhesion and low nucleation density. There are a number of potential surface treatment methods which can be used to overcome these problems including chemical etching, ion implantation, interlayer coating and bias treatment. Various approaches have been used to suppress the influence of Co and to improve adhesion between the diamond coating and the tungsten carbide substrate (Sein et al, 2003).

Diamond coating process can be done in seconds compared to the conventional chemical vapor deposition process which takes hours. It is possible to coat the cutting edges of all types of tools that will last much longer and dull only after prolonged use. Valves, casings and blades of rotating machinery are subjected to wear during operations. Hence, diamond coating is applied to avoid these. Besides, longer-lasting tools, instruments, and wind shields are only a few of the available applications for diamond coating.

Important advances have been made in the diamond coating of metals, glass, ceramics and plastics, using various techniques, such as CVD, plasma- assisted vapor deposition, and ion-beam-enhanced deposition. Examples of diamond-coated products are: scratchproof windows such as those used in aircraft and missile sensors for protection against sandstorms; sunglasses; cutting tools such as inserts, drills, and end mills; wear faces of micrometers and calipers; surgical knives; razors; electronic and infrared heat seekers and sensors; light emitting diodes; diamond-coated speakers for stereo systems; turbine blades; and fuel-injection nozzles. Techniques have also been developed to produce free-standing diamond films in the order of 1 mm thick and up to 125 mm in diameter; these include smooth, optically clear diamond film, unlike the hazy gray diamond film formerly produced. The film is then laser cut to the desired shapes and brazed onto, for example, cutting tools.

The development of these techniques, combined with the important properties of diamond such as hardness, wear resistance, high thermal conductivity, and transparency to ultraviolet and microwave frequencies, have enabled the production of various aerospace and electronic parts and components.

1.3 Statement of Problem

Application of CVD diamond coating on tool can be adversely affected by its weak adhesive strength to the substrates. Therefore, the evaluation of the adhesive strength is of great importance to ensure the integrity of diamond coatings under severe machining conditions. In particular, adhesive strength is a critical issue for super hard diamond coatings on Co-cemented tungsten carbide (WC-Co). Surface treatments leading to both Co removal and roughening of the substrate can ensure adequate adhesion levels of the diamond coatings deposited onto WC-Co substrates (Kamiya et al., 2001).

Based on the literature survey, the pre-treatment method to overcome the problem that occurs in substrate before the coating process is being investigated. The purpose of performing the pretreatment is to improve the roughness of the substrate surface before the coating processes as well as to eliminate the cobalt in the substrate surface that can decrease the effectiveness of the coating layer.

Besides, no research has been made using Design of Experiment (DOE) technique to find the optimal setting in the pretreatment process. In machinability studies investigations, statistical design of experiments is used quite extensively. In general, machinability can be defined as an optimal combination of factors such as low cutting force, high material removal rate, good surface integrity, accurate and consistent workpiece geometrical characteristics, low tool wear rate and good curl or chip breakdown of chips (Noordin et al. 2004). So in this study, statistical design of experiment (DOE) techniques is used to determine the best optimum setting for the pretreatment method before the diamond coating process.

1.4 Objective of Study

The objectives of the research are:

i) To establish parameter relationship between surface roughness condition and diamond nucleation density

ii) To propose solution - By implementing two step pre-treatment methods for coating process improvement.

iii) To get the optimal solution by implementing the Design of Experiment Techniques (DOE).

1.5 Scope of Study

The scopes of this study are limited as follows:

i) Concentrate only on the pretreatment coating process.
ii) The substrate material is limited to WC with 6wt% of Co only
iii) Surface pretreatment parameters to be varied are limited to exposure time and weight of the element in Murakami solution and oxidizing agent that affects surface roughness and Co depletion.

1.6 Thesis Layout

This thesis consists of four chapters. Chapter 1 is the introduction to this study. Background of the study, statement of problem, objective of the study, scope of the study and thesis layout is presented.

Chapter 2 contains the literature review of the DOE techniques and pre-treatment method. In the pretreatment process the focus is on the two-step pretreatment method. Initially alkaline solution is being used this is and followed by etching the substrate using an oxidizing agent.

Chapter 3 is concerned with the research methodology for this study. In this chapter, the experimental steps were discussed in detail.

Chapter 4 is concerned with the analysis of the result using the Design Expert Software. In this chapter, the factors that will effect and give the high efficiency for the response being investigated is being discussed in detail.

Chapter 5 summarizes the work done in this project and some recommendations for future work are also made.