EFFECT OF SURFACE PRETREATMENTS ON THE DEPOSITION OF POLYCRYSTALLINE DIAMOND ON SILICON NITRIDE SUBSTRATES USING HOT FILAMENT CHEMICAL VAPOR DEPOSITION METHOD

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ABSTRACT

The deposition of diamond films on a silicon nitride (Si_3N_4) substrate is an attractive technique for industrial applications because of the excellent properties of diamond. Diamond possesses remarkable physical and mechanical properties such as chemical resistant, extreme hardness and highly wears resistant. Pretreatment of substrate is very important prior to diamond deposition to promote nucleation and adhesion between coating and substrate. Polycrystalline diamonds films have been deposited on silicon nitride substrate by Hot Filament Chemical Vapor Deposition (HF-CVD) method. The Si_3N_4 substrates have been subjected to various pretreatment methods prior to diamond deposition namely chemical etching and mechanical abrasion. The structure and morphology of diamond coating have been studied using X-ray Diffraction (XRD) and Scanning Electron Microscopy (SEM) while diamond film quality has been characterized using Raman spectroscopy. The adhesion of diamond films has been determined qualitatively by using Vickers hardness tester. It was found that the diamond films formed on chemical pretreated substrates has cauliflower morphology and low adhesive strength but also have low surface roughness. Substrates that pretreated with sand blasting have yield diamond film with well-facetted morphology with high crystallinity and better adhesion. However, the surface roughness of the diamond film deposited on substrates pretreated with blasting are also higher.

ABSTRAK

Endapan filem intan diatas substrat silikon nitrid (Si_3N_4) merupakan satu teknik yang menarik kepada applikasi industri kerana sifat intan yang cemerlang. Intan memiliki sifat fizikal dan mekanikal yang menakjubkan seperti kalis bahan kimia, kekerasan yang tinggi dan sangat kalis haus. Pra-penyediaan substrat sebelum endapan intan adalah sangat penting untuk menggalakkan pertubuhan nuklei dan meningkatkan rekatan diantara salutan dan substrat. Filem intan polihablur telah diendapkan diatas substrat silikon nitrid menggunakan kaedah endapan wap kimia filamen panas. Substrat Si_3N_4 telah melalui pelbagai kaedah pra-penyediaan sebelum endapan intan seperti punaran kimia dan lelasan mekanikal. Struktur dan bentuk salutan intan yang terhasil telah dikaji menggunakan pembelauan sinar X-ray (XRD) dan mikroskop electron imbasan (SEM) manakala kualiti filem intan telah dikaji menggunakan spektroskopi Raman. Rekatan filem intan telah dikaji secara kualitatif menggunakan ujian kekerasan Vickers. Hasil kajian menunjukkan filem intan yang terbentuk di atas substrate yang melalui pra-penyediaan kimia mempunyai morfologi *cauliflower* dan kekuatan rekatan yang rendah tetapi mempunyai kekasaran permukaan yang rendah. Substrat yang dibagas dengan pasir menghasilkan filem intan yang mempunyai segi permata dengan kehabluran yang tinggi dan kerekatan yang lebih bagus. Akan tetapi, kekarasan permukaan filem intan yang diendap diatas substrat yang dibagas dengan pasir juga lebih tinggi.

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CHAPTER 1

INTRODUCTION

1.1 Background of the research

Apart from their appeal as highly treasured gemstones, diamond possesses a remarkable physical properties such as extreme hardness, high thermal conductivities, excellent infrared transparency and remarkable semiconductor properties making diamond one of technologically valuable materials. However, it has proved very difficult to exploit these properties, due to the cost and scarcity of large natural diamond.

Many attempts have been made to synthesize diamond artificially using graphite as starting material. This proved very difficult, mainly because at room temperature and atmospheric pressure, graphite is the thermodynamically stable allotrope of carbon. Although the standard enthalpies of diamond and graphite are differ only by 2.9 kJ mol^{-1}, a large activation barrier separates the two phases preventing interconversion between them at room temperature [1]. To overcome these problem, high pressure high temperature (HPHT) growth techniques has been introduced by General Electric to produce industrial diamond. However, the drawback of HPHT method is that it still produces diamond in form of single crystal thus limiting the range of application it can be used.

This leads to the idea of producing diamond from gas phase at much lower pressure in which carbon atoms could be added one-at-a-time to an initial template, in such a way tetrahedrally bonded carbon network forms. It can be achieved by using chemical vapor deposition (CVD) method. CVD involves a gas phase chemical reaction occurring above solid surfaces, which causes deposition onto that surface. All CVD techniques for producing diamond film require a mean of activating gas-phase carbon-containing precursor molecules[2]. This gas phase activation is achieved typically by using one of these three basic methods:

- External heating (as in hot filament CVD)
- Plasma activation (as in Plasma assisted CVD)
- A combination of thermal and chemical activation (as in flame CVD)

The applications for which CVD diamond films can be used are closely related to the various extreme physical properties they exhibit. The extreme hardness and high wear resistance of the diamond makes it an ideal candidate for use as coating material in cutting tool.

1.2 Problem Statement

Diamond coating have a great application as wear resistant layers on tools. Such diamond-coated hard metal and ceramic inserts are used successfully in machining fiber-reinforced plastic, graphites and aluminium alloys. However, in order for CVD diamond to be used as coating for tools and wear parts, it has been shown that two problem must be overcome first The problems are the diffusion of atoms from the substrate to the diamond and diffusion of carbon atoms to the substrate, and also the adhesion and residual stress in interlayer of diamond coating [3]. Poor adhesion can be caused by many factors such as mismatch of coefficient of thermal expansion (CTE) between diamond and substrate, residual stress, impurities and others.

1.3 Objectives and Scopes of Study

The objectives of this study are to study the effect of substrate surface treatment on morphology, coating adhesion, surface roughness and residual stress of polycrystalline diamond coated on silicon nitride. Surface treatment of the substrate is very important in order to produce high quality diamond coating with high adhesion strength.

The scope of the research includes:

a) Polycrystalline diamond deposited on silicon nitride substrate using Hot Filament Chemical Vapor Deposition machine using 99% CH_4 gas as precursor.

b) Prior to diamond deposition, the substrate undergoes various surface pretreatment processes as following:

i. The surface was blasted with SiC 180 for 30 sec (mechanical pretreatment)
ii. The surface was grinded with 180 grit for 5 minutes followed by 600 grit for 5 minutes (mechanical pretreatment)
iii. The surface was etched with various chemical reagents (chemical etchings)

c) Characterization on microstructure, morphologies and mechanical properties using scanning electron microscopy, atomic force microscopy, Raman Spectroscopy, X-ray diffraction and Vickers hardness indenter

1.4 Significance of the Study

In an effort to enhance diamond nucleation and to control film growth morphology, the effect of surface conditions on nucleation processes will be investigated to select the optimum surface pretreatment method. The chemical properties and surface conditions of substrate materials critically influence surface nucleation processes of diamond in CVD. Thus, based on the output of this study, optimum surface treatment technique and deposition parameter will be determined to produce goof quality polycrystalline diamond coating.