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Research Paper

Optical Retro reflector Using Thin Diamond Film

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ABSTRACT:*Diamond is a unique material with robust mechanical properties, the electrical and the optical properties of the thin film diamond can be altered as they are deposited using microwave plasma enhanced chemical vapor deposition (MPCVD) method. The thin film diamond can be used both as optical window for certain wavelengths of light and as a reflector, based on the thickness and the deposition parameters of the thin film diamond. In this paper, we are illustrating a 1 µm thin film diamond that we were able to deposit, pattern and etch selectively using standard microfabrication techniques. This patterning of diamond film allows us to create 3D structures using single crystal diamond.*
KEYWORDS *–crystalline diamond, amorphous diamond, retroreflector, optical, MPCVD*

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I INTRODUCTION

Diamond is a unique material, it is the hardest material identified with five times better heat conductor than copper. Diamond has a low coefficient of friction similar to Teflon and also has the lowest work function values [1-3]. About the optical properties, diamond has wide optical transitivity from the UV to the long IR range. About the electrical properties, diamond is an intrinsic electrical insulator with resistivity on the range of 10^{14} to 10^{16} ohm-cm and it is dope able with either an n-type or a p-type impurity to reach resistivity as low as 0.1 ohm-cm [2,3]. Diamond has a wide band gap energy level of 5.45 eV and it is chemically inert and biocompatible. These unique combination of diamond properties allows its use in a wide range of industrial applications. The superior quality of diamond among other materials is well known in optical, mechanical, thermal and electronic applications [4-6]. For several optical applications there is a need for a reflective optical surface, there is also need for diamond as stronger mechanical material for voltage converter, circuit breaker and other microsystems [4-7]. These microsystems with diamond as thin film can benefit from the versatile properties of the diamond. The diamond film deposited by microwave plasma enhanced chemical vapor deposition (MPCVD) can be manipulated during nucleation and growth conditions to achieve specific characteristics. It is meaningless determining an absolute quality for a diamond film without defining the ultimate application. For instance, optical windows require high transparency in the range of wavelength considered [8-10]. The transparency is linked to the diamond's intrinsic quality (lowest amount of structural defects and impurities), as well as to roughness. The use of polycrystalline diamond film as a heat spreader also requires high intrinsic quality and a limited grain boundary network. However, mechanical applications do not need a very high crystallographic quality, but instead a high deformation resistance and sometimes a very low roughness. We in this paper present deposition of free standing thin film poly crystalline diamond that can be used for numerous application.

II THIN FILM DIAMOND

Diamond nucleation on non-diamond surfaces without pretreatment is usually very difficult and slow. Several studies reported in the literature [2-6,8], make clear the importance of control (and improvement) of nucleation and early stages of growth for application in which materials properties are sensitive to or directly dependent upon the morphology and the grain size. An increase in surface nucleation density should reduce morphological instabilities and associated surface roughness and further may improve the homogeneity of the film and reduce formation of voids at the substrate or coating interface, leading to better diamond-substrate adhesion and thermal transport properties [11]. Nucleation density has been increased from $<10^5$ cm^{-2} on untreated substrates up to 10^{11} cm^{-2} on scratched or biased substrates. But there is still much debate and

uncertainty about which mechanism truly governs the nucleation density. Diamond nucleation on non-diamond substrates is generally proposed to occur mostly on an intermediate layer of diamond-like amorphous carbon, metal carbides, or graphite formed at the substrate surface due to chemical interactions between activated gas species and the surface during the incubation period [12]. These possible nucleation layers are responsible to provide the nucleation sites for the diamond crystallite growth and they can change in thickness from several angstroms until few nanometers, which depend on the substrate and buffer layer material.

During MPCVD diamond deposition, the parameters that govern the gaseous phase interactions, plasma/surface interaction, and chemical surface characteristics need to be closely monitored and the relationship between them derived. During growth, the isolated crystallites grow and develop faceting due to the relative high rate of surface carbon diffusion from the surrounding substrate surface.Diamond grows epitaxially on both diamond and cubic boron nitride (cBN) single-crystal substrates under typical low pressure processing conditions, but it is difficult and expensive to obtain large area single crystal substrates of either material. Deposition of single crystal diamond on other substrates than diamond and cBN, while very desirable, has not yet been achieved [11-12]. Methods for obtaining large-area single crystals or highly oriented diamond film needs to be pursued especially for the development of large-scale electronic applications. In order to be able to successfully and reproducibly deposit single-crystal diamond films, it is first necessary to fully 1understand the mechanisms that govern the crystalline diamond film pretreatment, nucleation and growth. We were able to control of the different parameters involved on the diamond growth by MPCVD by associating the theoretical model with the experimental data. This understanding and control is extremely important since diamond is such a versatile material. It can, for example, be tailored from an insulator to a semiconductor.

III MPCVD DIAMOND PROPERTIES

These unique properties of diamond are explained by its covalent bonding between each nearest neighbor C atoms. The coordination number for carbon is determined by its characteristic sp3 hybridization bonding in which the four outer-shell electrons of the carbon atom are shared with adjacent atoms in equally spaced directions. Just for comparison the graphite structure, also composed only of carbon atoms, presents strong sp2 hybridization bond on the hexagonal rings but between the layers weak Van der Walls forces account for the graphite's friable nature and application as a useful 'dry" lubricant.

Figure 1.(a) Diamond structure and (b) graphite's layered structure [12].

Change on the growth parameters directly affects the diamond grain size from few nanometers (ultranano- and nanocrystalline) to few microns (microcrystalline). A better understanding of the difference between the grain sizes and diamond properties can be visualized on Table 2. Special note should be taken of the relationship of the grain size with the surface roughness, which is an important characteristic to be considered for specific applications. However, the change in the crystal size does not drastically change the Young's modulus or the hardness of the films. One characteristic that changes significantly with the grain size is the atomic bonding. Microcrystalline grains shows a 100% sp^3 bonding, while nano and ultranano crystal sizes there is a significant percentages of sp^2 (graphitic) bonding present.

Diamond was first successful synthesized by Bundy and co-workers in 1962 by high pressure and high temperature technique. Since then, other methods and techniques has being developed for the growth of diamond, among them hydrothermal, chemical vapor deposition (CVD) and physical vapor deposition. The CVD diamond deposition techniques can be divided into 4 categories: thermal activation, plasma activation, laser ablation, and combustion flames. Even though the methods vary, the growth conditions are very similar; 10 to 140 Torr pressure, 700 to 1100^{o}C substrate temperature and about 1% C/H ratio [12]. But these conditions are not unique for diamond deposition; developments have being made to deposit diamond at lower temperatures specially to accommodate the micro fabrication requirements. The focus was on the plasma activation CVD, specifically the microwave enhanced plasma chemical vapor deposition (MPCVD). The experimental deposition

was performed on a new MPCVD reactor from Seki Technotron Inc. with cooled and heated stages, voltage bias capability and a 5kW microwave power limit.

Table 2.Developed diamond property associated with the crystalline size

	Microcrystalline	**Nanocrystalline**	**Ultrananocrystalline**
Crystallinity	columnar	Mixed diamond and non-diamond	Mixed diamond and non-diamond
Grain size	~0.5-10μm	50-100nm	2-5nm
Surface roughness	400nm-1μm	20-100nm	7-40nm
Electronic bonding	sp^3	Up to 50% sp^2 (second phase)	2-5% sp^2 (in Grain Boundary)
Hydrogen content	<1%	<1%	<1%
Young's modulus (GPa)	900-1000	500-1000	980
Hardness (GPa)	95-100	95-100	97
Coeff. friction	0.4-0.7	0.1-0.7	0.03
Carrier concentration (cm^3)	1015-1019	Unknown	5x1019-2x1020
Electron mobility (cm^2/V.s)	200-1000	Unknown	5-10
Thermal conductivity (W/cm.^{o}C)	22	10-20	0.01-0.2
Optical transparency (% transmittance)	50-85	30-80	~10

IV EXPERIMENTAL RESULTS

For a successful diamond deposition, the initial nucleation step is critical and needs careful evaluation as it affects the adhesion, film properties, electronic interface states, and the type of substrate which can be used. Typical surface pretreatments applied with each respective nucleation density can be visualized on Table 3.Different approaches to improve the nucleation density have been reported as well as the usage of buffer layers to improve the substrate adhesion, orientation among other characteristics. Improvement of few orders of magnitude on the nucleation density by mixing other powders besides diamond to the ultrasonic slurry during the substrate pretreatment was seen experimentally. Using different ceramic interlayer containing SiC, SiN_x, SiCN, TiSiN and TiAlSiN to promote a higher nucleation density compared to as-polished Si substrate was also demonstrated. We observed on the same growth conditions the formation of nanocrystalline diamond film over the TiSiN and TiAlSiN layers and microcrystalline diamond films over the Si(C,N) and Si wafers., a textured growth of diamond film on single crystalline iridium buffer layer using bias as the nucleation step, showing 0.17^o for tilt and 0.38^o for twist. The importance and influence of the nucleation, substrate and buffer layers on the diamond film characteristics leads to a robust film thickness. The study done targetedultrasonicatednanodiamond slurries with and without addition of other powders; the influence of different slurries solutions on the nanodiamond agglomeration, and finally the influence of the bias enhanced nucleation. To help better understanding these factors, simulations based on density functional theory (DFT) modelled the diamond nucleation and defect formation on hetero-substrates and buffer layers. Those simulations compared with the experimental data identified the parameters that influence the diamond growth.

Table 3. Surface pretreatments conditions and nucleation density typical values

Pretreatment conditions	**Nucleation density (cm^{-2})**
No pretreatment	$10^3 – 10^5$
Surface abrasion	$10^6 – 10^{10}$
Ultrasonic scratching	$10^7 – 10^{11}$
Spinning diamond powder loaded photoresist	$10^6 – 10^8$
Spraying of diamond loaded fluids	$10^8 – 10^{11}$
Diamond powder loaded water	$10^8 – 10^{10}$
Biasing	$10^8 – 10^{11}$

Alongside, the optimization of the growth parameters such as substrate temperature, reactor pressure and reactant composition took place and interaction with the simulation was addressed. Optical emission spectroscopy (OES) was used to provide an opportunity to study the excitation processes of the plasma species and to establish the relationship linking electronic excited states to ground state species temperature and concentrations.

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Figure 2. Free standing polycrystalline diamond, ~50 mm diameter and ~230 μm thick.

A free standing diamond film was deposited on a Si (100) substrate using the Seki Technotron MPCVD system and the sample is shown on Figure 2. After the deposition the Si substrate was chemically etched away revealing the silicon side growth. The relatively low roughness (~59 nm) on the surface grown on the silicon substrate and a considerable amount of pores due to the island type diamond growth and relatively low nucleation density can be visualized as well. But even with the presence of pores, the surface is mirror like, as showed on Figure 2. Raman spectroscopy showed only one well defined peak, typical of a single crystal diamond (1332 cm^{-1}).

V CONCLUSION

We were able to deposit polycrystalline diamond film on a silicon wafer. The deposited film was of varying thickness for different applications, even with thicker film thickness the surface roughness was less than 60 nm and was of a mirror quality. Though the diamond film has pores, but the diamond film was still of optical quality and using a laser we were able to determine the reflectivity from the diamond surface. The MPCVD method of deposition of thin film diamond has several parameters that can be altered to determine the final property of the diamond film.

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