Diamond & Related Materials 69 (2016) 13–18

Contents lists available at ScienceDirect

Diamond & Related Materials

journal homepage: www.elsevier.com/locate/diamond

Structural, optical and mechanical properties of thin diamond and silicon carbide layers grown by low pressure microwave linear antenna plasma enhanced chemical vapour deposition

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ARTICLE INFO

Article history:
Received 3 March 2016
Received in revised form 1 June 2016
Accepted 24 June 2016
Available online 27 June 2016

Keywords:
Diamond
Silicon carbide
Adherence
Mechanical properties
Optical properties

ABSTRACT

In this work, we detail the properties of thin silicon carbide and polycrystalline diamond layers grown by microwave plasma enhanced chemical vapour deposition with linear antenna delivery. Structural, mechanical and optical properties are compared for their potential use as transparent hard coatings. Silicon carbide layers exhibit mechanical properties comparable to thin diamond layers but with a significantly higher adhesion and lower optical absorption coefficient over a wide spectral range.

1. Introduction

Silicon carbide (SiC), as diamond, possesses a large number of outstanding properties, such as high hardness, high thermal conductivity, large band gap, wide wavelength range of transparency, chemical inertness with variable electrical conductivity by doping, etc. Hence, SiC and diamond thin films hold a wide range of promising mechanical, optical, optoelectronic, and electrical applications as an inert, hard, conducting or insulating, and transparent coating [1,2]. Wide band gap thin layers would provide ideal transparent protective films on optical components. Additionally, when doped with boron, diamond thin films also hold promising electrochemical applications [3,4]. However, these layers must exhibit low surface roughness, high adhesion and wear resistance in addition to high transparency and chemical inertness for long term applications. In this work, we detail and compare structural, mechanical, adhesion and optical properties of thin silicon carbide and diamond layers grown by microwave plasma enhanced chemical vapour deposition with linear antenna delivery.

2. Experimental

Silicon carbide and diamond layers were deposited on {100} 20 mm^2 silicon wafers (ON Semiconductor Czech Republic, s.r.o) with an amorphous native oxide top layer and 10 mm^2 glass substrates (Corning Eagle XG) using a microwave plasma enhanced chemical vapour deposition system with linear antenna delivery (MW-LA-PECVD) [5,6]. Prior to growth all substrates were ultrasonically cleaned in acetone and isopropyl alcohol. Substrates for diamond layer growth were seeded with a nano-crystalline diamond dispersion (NanoAmando®B) via spin coating, whereas silicon carbide layers were grown on unseeded substrates. All layers were deposited in a hydrogen rich, methane and carbon dioxide gas mixtures at low pressure with the same microwave power. Deposition conditions of silicon carbide and diamond layers are reported in Table 1. As matter of fact, silicon carbide formation is not expected with the process gas mixture without a Si precursor. However, we have recently demonstrated the presence of Si by optical emission spectroscopy in the MW-LA-PECVD system at low CO_2 concentrations and

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http://dx.doi.org/10.1016/j.diamond.2016.06.014
0925-9635/

Table 1
Deposition conditions of silicon carbide (SiC) and diamond layers by microwave plasma enhanced chemical vapour deposition system with linear antenna delivery.

	Diamond	SiC
Microwave power	2 × 3 kW	2 × 3 kW
Methane concentration	5%	4.2%
Hydrogen concentration	92%%	95.75%
Carbon dioxide concentration	3%	0.05%
Process pressure	0.3 mbar	0.3 mbar
Substrate temperature	520 °C	520 °C
Deposition rate	50 nm/h	33 nm/h

the consequent formation of silicon carbide due to the assumed plasma etching of the quartz tubes which form the vacuum to air interface in this system [5]. Substrate temperatures were monitored during deposition using a Williamson Pro 92-38 infrared pyrometer and thermocouples mounted in the substrate table. Both temperature measurement techniques were in agreement. Temperatures are noted in Table 1.

Morphological and structural properties of deposited layers were characterised by a Tescan FERA 3 scanning electron microscope optimised for observation of thin film morphology and thickness measurement in cross-section. Ambient atomic force microscopy using a Dimension Icon (Bruker) in peak force tapping mode with Tap150AL-g tips optimised for surface topography and surface roughness was measured on a 3 × 3 mm^2 surface area.

The crystalline structure of deposited layers was characterised by X-ray diffraction methods using a PANalytical X'Pert PRO diffractometer with a Co anode ($\lambda = 0.1789$ nm) with different configurations. The diamond layers were measured with the line focus in a parallel beam geometry with a Goebel mirror in the primary beam and a parallel plate collimator (0.09°) in the diffracted beam by 2θ-scan with a fixed angle of incident equal to 2° whereas SiC layers were measured with line focus in Brag-Brentano geometry using divergent slits and linear detector (X'Celerator). The pole figures and ψ-scans of SiC were also measured with a point focus on ATC-3 texture cradle.

Raman spectra were recorded by a LabRAM HR (Horiba Jobin-Yvon) spectrometer coupled to an Olympus BX microscope with a 100× objective. He-Ne laser with 1.96 eV (633 nm) energy was used as excitation with the power at the sample of 8 mW. 600 grooves/mm grating and a confocal hole of 50 μm was used. Spectra were background-corrected using the spectrum of the glass substrate as the baseline.

The mechanical and tribological properties were measured by nanoindentation and scratch test techniques at room temperature using a fully calibrated NanoTest instrument (MicroMaterials). Nanoindentation experiments were performed with a sharp Berkovich indenter in a load controlled mode. Taking into account the thickness of the films, their roughness, ability to calibrate the indenter tip at shallow depths and the requirement of full plasticity beneath the indenter, two indentation loads of 1.0 and 1.5 mN [7] were chosen for determination of the stability of mechanical properties of the films with increasing depth and also to validate the used experimental setup. Progressive nanoscratch tests were performed in a 3 step procedure: 1-topography, 2-scratch and 3-topography for two maximum loads (300 mN and 500 mN). The initial and final topography measurements were performed at a load of 0.02 mN to avoid any wear. During the scratch procedure the initially constant topographic load of 0.02 mN was applied over the first 50 μm and then ramped to the maximum load at constant loading rate of 7.8 and 13 mN/s respectively. All scans were performed with a scan speed of 10 μm/s over a total scan length of 450 μm. Nano-wear tests, i.e. multi-pass scratch tests were performed at a constant load of 20 mN by alternating scratch and topography passes every two scratches to monitor surface degradation, 16 passes were carried out in total. All evaluations of scratch tests were performed on the basis of the indenter load-depth records and analysis of the residual scratch tracks. Laser scanning confocal microscope LEXT OLS 3100 (Olympus) was used for high-resolution imaging.

Index of refraction and absorption coefficient of deposited layers were measured from UV to near infrared (250 nm to 1700 nm) using photothermal deflection spectroscopy (PDS) [8] and ellipsometry. The optical transmittance, reflectance and absorptance spectra were measured simultaneously in the broad spectral range from ultraviolet to near infrared in a dual beam setup. The absorptance down to 10^{-4} was measured by the absolute photothermal deflection spectroscopy (PDS) normalised via the signal of the highly absorbing black coating [9]. Ellipsometry measurements were carried out using a VASE ellipsometer (Woollam) from 190 nm to 2400 nm. Spectra were

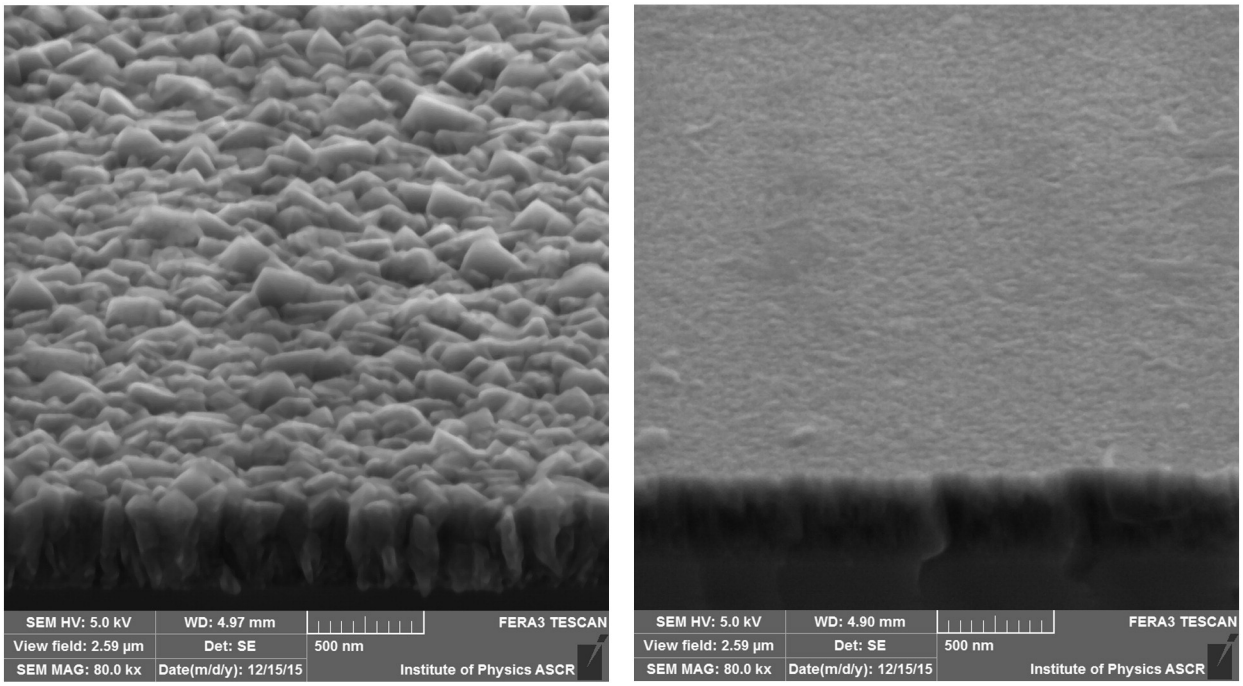

Fig. 1. Scanning electron microscopy images of diamond (left) and silicon carbide (right) layers.