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Properties of boron-doped epitaxial diamond layers grown on (110) oriented single crystal substrates

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ABSTRACT

Boron doped diamond layers have been grown on (110) single crystal diamond substrates with B/C ratios up to 20 ppm in the gas phase. The surface of the diamond layers observed by scanning electron microscopy consists of (100) and (113) micro-facets. Fourier Transform Photocurrent Spectroscopy indicates substitutional boron incorporation. Electrical properties were measured using Hall effect from 150 to 1000 K. Secondary ion mass spectrometry analyses are consistent with the high incorporation of boron determined by electrical measurements. A maximum mobility of 528 $cm^2 V^{-1} s^{-1}$ was measured at room temperature for a charge carrier concentration of 1.1 10^{13} cm^{-3}. Finally, properties of boron doped (110) diamond layers are compared with layers on (100) and (111) orientated substrates.

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1. Introduction

Synthesis of high quality doped diamond epitaxial layers is important for electronic applications. Successful incorporation of phosphorous (n-type) and boron (p-type) in diamond with good electric properties is achieved by plasma enhanced chemical vapour deposition (PE-CVD) techniques [1]. Initial boron doped diamond layers grown using the CVD technique with diborane were reported by Fujimori et al. [2]. Substrate crystalline orientation plays a significant role in the growth and the properties of doped diamond layers. Boron doped homoepitaxial layers with electrical properties comparable with high quality natural IIb diamonds are obtained on (100) diamond substrates [3–5]. Efficient phosphorous doping of CVD diamond has also been successfully achieved since 1997 [7] but limited to layers grown on (111) substrates until 2005. Despite (001) oriented n-type homoepitaxial diamond layers show mobility comparable to the one obtained on (111) oriented substrates, they are more resistive due to high compensation [8]. This variation of electrical properties of doped diamond with crystalline orientation limits bipolar electronic applications of diamond. The pioneering work carried out by A. Lazea et al. on n-type doping on (110) oriented polycrystalline diamond substrates [9,10] has been an atypical endeavour to solve this issue. Y. Balasubramaniam et al. have recently reported epitaxial growth of n-type layers on (110) oriented substrate with electron Hall mobility as high as 370 $cm^2 V^{-1} s^{-1}$ at 300 K [11]. Several theoretical [11–14] and experimental studies [15] on epitaxial growth on (110) oriented diamond substrates have been reported. C. J. Chu et al. reported variable growth kinetics of intrinsic diamond layers with substrate crystalline orientation [16]. F. Silva et al. studied the effect of deposition conditions, including B/C ratio in the gas phase, on the growth rate as a function of substrate's crystalline orientation and developed a 3D geometrical growth model to describe the final morphology of millimetre thick CVD diamond layers [17–19]. Boron doped diamond layers have also been grown on (110) oriented substrate for electrochemical [20] or superconductivity [21] characterization. However, none of these papers include thorough electrical characterization of semiconducting boron doped diamond layers. Visser et al. reported interesting electrical characterization of both (100) and (110) oriented boron doped diamond layer in a low temperature range (<500 K) focusing on hopping conduction [22]. To fully determine the potential of diamond growth on (110) oriented crystals for electronic applications, it is necessary to determine growth conditions and properties of p-type diamond layers deposited on (110) oriented substrates. In this paper, we report on the electrical, optical and morphological properties of p-type diamond layers grown on (110) oriented single

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crystal substrates and we compare their electrical properties to that of (100) and (111) diamond epitaxial layers grown in the same reactor.

2. Experimental

Boron doped diamond layers were grown on (110) high pressure and high temperature Ib diamond substrate from Sumitomo Electric Industries, Ltd. These samples were obtained from a larger (100) oriented Ib diamond by laser cutting and polishing. The diamond substrates were cleaned in hot sulphuric acid with addition of potassium nitrate. Boron doped layers were grown in a homemade NIRIM reactor, which is described in Ref. [5]. Prior to growth the substrate was exposed to a pure hydrogen plasma for 10 min using the same pressure and microwave power as deposition conditions. Pressure, temperature, total gas flow rate, methane to hydrogen concentration and microwave (MW) power were kept constant during growth at 130 mbar, 1100 °C, 500 sccm, 0.1% and 500 W, respectively. Trimethyl-boron (TMB) mixed in hydrogen was used as a doping source. Its concentration in the gas phase was varied from zero up to 20 ppm using a two stage dilution gas system. The B/C ratio in the gas phase used during the growth of the different samples is reported in Table 1.

Thicknesses of grown boron doped films were calculated from the mass variation of the samples after deposition using a 1 μg resolution scale TGA 2950 thermogravimetric analyser. The average deposition rate was determined to be ~1.2 μm/h. All samples were characterized by scanning electron microscopy (SEM) using a Quanta 200F from FEI, by atomic force microscopy in tapping mode using a multimode scanning probe microscope a with a controller IIIa from Veeco ©, by Fourier-Transform Photocurrent Spectroscopy (FTPS) using a Nicolet 8700 Fourier-Transform Infra-Red spectrometer and by a home built Hall effect and secondary ion mass spectrometry (SIMS) using a Cameca IMS 4f equipment.

Fourier-Transform Photocurrent Spectroscopy (FTPS) measurements [23] were carried out at liquid nitrogen temperature in an Optistat from Oxford Instruments with a bias voltage of 100 or 200V_{DC} and an 5182 preamplifier from EG&G instruments. All spectra were corrected for frequency dependence of the sample and the DTGS detector. Ti/Al interdigitated contacts with a line spacing of 100 μm were patterned using lift-off technique and metals were deposited by sputtering technique on the oxidized surface of the doped layers.

Hall effect measurements were carried out between 150 K and 1000 K in two steps using the Van der Pauw method. Low temperature measurements ($T \leq 400$ K) were made in a Microstat^{He} from Oxford Instruments and high temperature measurements ($300 \leq T \leq 1000$ K) were made in a homemade oven. Ti/Pt/Au contacts were deposited by evaporation and annealed at 450 °C before measurements. A 6221 DC and AC current sources, two 2400 source metres and a 7152 low current matrix card from Keithley were used for Hall effect measurements.

Secondary-ion mass spectrometry (SIMS) using the Cameca IMS 4f equipment was performed on all samples to measure the depth distribution of boron with Cs^+ primary ions accelerated at 10 keV. The concentration of boron was quantified using implanted standards. Doped diamond samples were also analysed by cathodoluminescence (CL) spectroscopy to quantify the boron acceptor concentration N_a^{CL}. CL spectra of samples were obtained using a 10 keV electron beam on samples cooled down at 102 K in a tungsten-filament JEOL840 scanning electron microscope. Emitted light was collected by a parabolic mirror and injected with metal optics into a TRIAX550 monochromator equipped with a UV enhanced-silicon CCD camera.

3. Results and discussion

All doped layers have the same surface morphology (see Fig. 1). The surface is uniform on the whole surface without any non-epitaxial grains, in contrary to films grown on (100) oriented substrates [4]. However, the surface is rough and micro-facetted due to the unstable nature of (110) diamond surfaces as seen on microcrystalline [24] and epitaxial diamond [16] layers. The root mean square roughness measured by AFM is about 100 nm for an observed area of 100 μm^2. The SEM image in Fig. 1c shows the assignment of the different facets. The two directions that define mirror plans perpendicular to the {110} surface are [00-1] and [1–10]. Observed planes on the surface have to be of a different nature due to the 2-fold [110] axis of rotation in diamond. Planes of the surface that include the [00-1] direction have to be {100} and {010}. Other types of planes can be {111} or {113} which are observed on CVD diamond films [19]. Micro-facets have been assigned to be (100) and (113) micro-facets, as the angle between {111} planes is larger than the one of {113} and smaller than the angle between {100} planes. These facets are identical to the ones observed on the edge of thick single crystal grown by the PECVD method [17].

To determine the substitutional incorporation of boron, FTPS measurements have been carried out on all samples. Experimental results are shown in Fig. 2. The spectrum from the sample with low B concentration exhibits the peak due to the excitation state of boron in diamond at 347 meV. The photoionization spectrum of the B-doped diamond confirms the electronic transitions from ground to excited bound states from which free holes are thermally ionized into the valence band. The photoionization continuum in weakly B-doped diamond starts at around 370 meV and the oscillatory photoconductivity of 165 meV periodicity is observed at higher photon energies [25]. These are a clear signature of electrically active boron incorporation in diamond. As the boron content in the layer increases, the gap between the excitation peak and the photoionization threshold is less pronounced and the photoionization threshold shifts to lower energies. This is attributed to the overlapping of wave functions of the neighbouring boron atoms [26]. The same experiments were carried out on layers grown on (100) and (111) substrates. The spectrum of a (100) oriented diamond layer doped with B/C = 0.8 ppm in the gas phase (sample I24 of Ref. [4]) is also shown in Fig. 2 for comparison (dotted line). In this sample the peaks are observed at 347 meV and 363 meV with the 2 meV split due to spin orbital splitting of the valence band structure [27]. Despite the fact that the photoionization threshold and the oscillatory photocurrent are similar for different types of layers, the boron excitation peaks are more pronounced and the peak at 367 meV can be also observed on the (100) oriented diamond spectrum in the region below the photoionization threshold. On the other hand, photocurrent spectra could not be recorded using the continuous scan of the FTPS technique operating at kHz frequencies [28] instead they were collected at low frequency using the amplitude modulated step-scan FTPS technique [29]. This shows not only the slow photo-response of boron doped

Table 1
Summary of electrical properties of samples grown in this study as a function of their B/C ratio: $[p]^{300K}$ carrier concentration at 300 K, (μ^{300K}) carrier mobility at 300 K and (σ^{300K}) resistivity at 300 K measured by Hall effect; (E_a) activation energy, (N_a) acceptor concentration, (N_d) donor concentration, ($k = N_a/N_d$) compensation determined by fitting of Hall effect data, $[B]^{SIMS}$ boron concentration measured by SIMS, Na^{CL} boron acceptor concentration measured by cathodoluminescence.

Sample	B/C (ppm)	$[p]^{300K}$ (cm^{-3})	μ^{300K} ($cm^{-2}\,V^{-1}\,s^{-1}$)	σ^{300K} ($\Omega\,cm^{-1}$)	E_a (meV)	N_a (cm^{-3})	N_d (cm^{-3})	$k = N_a/N_d$ (%)	$[B]^{SIMS}$ (cm^{-3})	N_a^{CL} (cm^{-3})
K12	0	$1.1\ 10^{13}$	528	1070	360	10^{17}	$6.5\ 10^{16}$	65	$6\ 10^{16}$	$8.7\ 10^{16}$
K10	0.036	1.4210^{13}	434	1040	360	$3\ 10^{17}$	$8\ 10^{16}$	26	$1.8\ 10^{17}$	$1.2\ 10^{17}$
K13	0.3	$4.7\ 10^{13}$	430	312	331	$4.5\ 10^{17}$	10^{17}	22	$4\ 10^{17}$	$4.8\ 10^{17}$
K16	0.8	$2\ 10^{14}$	232	145	300	$2\ 10^{18}$	$4\ 10^{17}$	20	$2.3\ 10^{18}$	$2.2\ 10^{18}$
K04	2	$5.5\ 10^{14}$	148	73	260	$3\ 10^{18}$	10^{18}	33	$6.5\ 10^{18}$	–
K15	20	$7.8\ 10^{14}$	223	37	285	$4.5\ 10^{18}$	$4\ 10^{17}$	9	$3.4\ 10^{18}$	–

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