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Successive current–voltage measurements of a thick isolated diamond film

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Abstract

Polycrystalline diamond films were deposited on p-type (100) silicon substrate using a methane/hydrogen gas mixture in a microwave plasma-assisted chemical vapor deposition system. After the back-etched process, the Al contacts were evaporated on both sides of a 150 μ m thick isolated diamond film for consecutive high-voltage measurements. It was found that the current–voltage (*I–V*) characteristics of the Al/diamond/Al structure exhibited two Schottky barrier diodes in a back-to-back configuration. Since the top diamond surface possessed better diamond quality than the bottom surface, the top Schottky diode with a breakdown voltage of 897 V and a lower breakdown voltage of -515 V for the bottom Schottky diode was observed for the first *I–V* measurement. However, the breakdown voltage was decreased by 37 and 140 V for the top and bottom Schottky diodes after the consecutive sixth repeated measurements. It was found that the oxygenated phenomenon was more prominent; in addition, the quality of the isolated diamond film was also degraded after the consecutive high-voltage measurements. It was indicated that decrease of the breakdown voltage was due to the oxidation layer and the non-diamond components on both surfaces of the isolated diamond film. © 2001 Elsevier Science B.V. All rights reserved.

Keywords: Plasma-assisted CVD; AFM; XPS

1. Introduction

Diamond with a wide energy gap (5.5 eV) has a unique combination of properties, such as high breakdown voltage, high thermal conductivity, and excellent radiation hardness etc. [1,2]. In the recent years, Si and GaAs was generally used as the semiconducting material, however, these materials have restricted applications in high voltage, high temperature, high radiation, and/or corrosive environments. To overcome these limitations, wide energy bandgap materials are preferred. Among them, diamond has the highest breakdown voltage, the highest thermal conductivity and radiation hardness which has potential as a semiconducting material.

It was known that the electrical breakdown is one of the main limitations of high-voltage device operation such as in high-power and high-voltage electronic devices. Several researches have been studied for the high-voltage characteristics of the diamond films [3–5]. It was known that the breakdown voltage was dependent on the surface morphology, film quality, and thickness [6]. However, successive current–voltage measurements were important for the stability of the electronic device. In this research, the successive

current–voltage measurements and the correlation to the physical characteristics of the isolated diamond film will be carefully studied.

2. Experimental details

Undoped diamond films were deposited on a 2 in. (100)p-type silicon wafer using a microwave plasma chemical vapor deposition system. Prior to deposition, the silicon substrate was pretreated by the photoresist in which 0.1 µm diamond powder is suspended in order to enhance the nucleation density. Typical deposition conditions were shown as follows: gas flow rates: CH₄/H₂: 20/600 sccm; pressure: 120 Torr; deposition temperature: 900 °C, deposition time: 200 h. The isolated diamond film was then obtained by etching the silicon substrate with the KOH solution (44% by weight to water) at $60 \,^{\circ}$ C. The Al contacts with 0.5 mm diameter were obtained on both sides of the isolated diamond film by vacuum thermal evaporation through a shadow mask. Following, the electrical properties of the Al/isolated diamond/Al structure were measured on a microprobe station with Source-Measure Unit Keithley 236 and 237 system in the voltage range of ± 1100 V. The step delay time between each electrical measurement is 30 s.

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The microstructure information and the surface roughness of the diamond film were observed via the atom force microscope (AFM). The XPS was then adapted to analyze the chemical components on both surfaces of the isolated diamond film. The quality of the diamond film was identified by Raman spectroscopy using the 514.5 nm line of Ar laser operated at a laser power of 125 mW and the beam size was approximately 10 μ m in diameter.

3. Results and discussion

In the scanning range of $50 \times 50 \,\mu\text{m}^2$, from Fig. 1(a) and (b), a root-mean-square roughness (R_q) of 1.67 μ m and 32.62 nm was respectively, observed for the top surface and bottom surface of the isolated diamond film. It was observed that the grain size is much larger for the top surface of the isolated diamond film than the bottom surface of the isolated diamond film, which means that the grain size of the diamond film increases with the diamond film thickness.

From Fig. 2(a) and (b), it was found that the FWHM of the characteristic diamond peaks at 1333.5 and 1331.4 cm⁻¹ were 5.9 and 8.1 cm⁻¹ respectively, for the top surface and

 $R_{g}=1.67 \, \mu m$



Fig. 1. AFM images of (a) the top and (b) the bottom surfaces of the isolated diamond film.



Fig. 2. Raman spectra of (a) the top and (b) the bottom surfaces of the isolated diamond film.

the bottom surface of the isolated diamond film. The increase on the Raman shifting of the characteristic diamond peak from 1331.4 to 1333.5 cm⁻¹ as the thickness of the diamond film increases indicates that the isolated diamond film is in compressive stress [7]. Note that the peak at $1500-1550 \text{ cm}^{-1}$ are attributed to amorphous carbon, and 1580 cm^{-1} is characteristic of graphite which was reported everywhere [8,9]. A broad shoulder centered at around 1550 cm^{-1} indicates the presence of amorphous carbon or disorder sp²-type carbon for the bottom surface of the isolated diamond film.

From the current-voltage (I-V) characteristic of the Al/diamond/Al structure, it was indicated that the equivalent circuit could be represented as two Schottky barrier diodes in a back-to-back configuration as shown in Fig. 3(a) [10]. The successive I-V characteristics of the Al/diamond/Al structure for the top Schottky diode and the bottom Schottky diode were shown in Fig. 3(b) and (c), respectively. The top Schottky diode with a breakdown voltage of 897 V and a lower breakdown voltage of -515 V for the bottom Schottky diode was observed for the first I-V measurement. A lower breakdown voltage for the bottom surface of the isolated diamond film indicated that the quality for the bottom surface of the isolated diamond film was poor. The poor quality for the bottom surface of the isolated diamond film was also confirmed by the Raman analysis as shown in Fig. 2(b). It was then suggested that the growth of polycrystalline diamond on the silicon substrate would introduce a high density of surface states at the diamond/silicon interface. This led to the broadening of the characteristic diamond peak at 1331.4 cm^{-1} (FWHM from 5.9 to



Fig. 3. (a) Equivalent circuit for the Al/diamond/Al structure. The I-V characteristics of the Al/diamond/Al structure obtained from the successive high-voltage measurements for (b) the top and (c) the bottom Schottky diodes.

 $8.1 \,\mathrm{cm}^{-1}$) and associated with the signal of non-diamond carbon phases between 1500 and $1600 \,\mathrm{cm}^{-1}$ for the bottom surface of the isolated diamond film. It was then suggested that the quality of the diamond film had strong influence on the breakdown phenomenon.

It was known that the breakdown voltage decreased and became stable after several times of measurement. It was found that the stable breakdown voltage was at 860 and 375 V respectively for the top and bottom Schottky diode after the consecutive sixth repeated measurements. Moreover, the breakdown voltage was decreased by 37 and 140 V for the top and bottom Schottky diodes after the consecutive sixth repeated measurements. From Fig. 4(a), the XPS spectrum for the top surface of the isolated diamond film before the consecutive high-voltage measurements was observed. It was found that the emission was mainly from the C_{1s} peak at 284.3 eV and O_{1s} peak at 532.8 eV. As shown in Fig. 4(b), the C_{1s} peak at 286.2 eV and O_{1s} peak at 532.5 eV was observed on the top surface of the isolated diamond film after the consecutive high-voltage measurements. It was known that C1s peak at 284.3 and 286.2 eV feature originates respectively, from the diamond and C-O bonding [11,12]. The ratio of the oxygen to carbon (O/C) signal



Fig. 4. XPS spectra of the isolated diamond film (a) before and (b) after the consecutive high-voltage measurements.

intensity was 0.36 and 2.05 before and after the consecutive high-voltage measurements, respectively. It was indicated that the oxygenated phenomenon increased considerably after the consecutive high-voltage measurements. Raman spectra of the Al/isolated diamond film before and after the consecutive high-voltage measurements were shown in Fig. 5(a) and (b). It was found that the FWHM increased from 6.2 to 7.1 cm^{-1} and non-diamond carbon phases (i.e. a broad shoulder center at around 1600 cm^{-1}) increased a lot after the consecutive high-voltage measurements. It was



Fig. 5. Raman spectra of the Al/isolated diamond film (a) before and (b) after the consecutive high-voltage measurements.

Guo et al. [13] reported that the hysteresis loop in the I-Vcharacteristic of Al/boron-doped polycrystalline diamond Schottky contact, and attributed to the deep levels of high density and high activation energy in the boron-doped polycrystalline diamond film. Large capture and emission time constants which respectively characterize the time required for majority carriers to reach a steady state value during charging and discharging processes. It was also known that the breakdown have been recognized as the main failure mechanisms in such ultra-thin oxide. Kiyota et al. [14] also reported that the mechanisms of the barrier formation on the as-grown surface are drastically changed by the oxidation. The chemisorbed oxidation layer acted as surface states between the Al contact and diamond film. The increased density of surface states gives rise to increased recombination/generation in the depletion region of the isolated diamond film. The time taken by majority carriers to reach the stable state increased. From the XPS analyses of Fig. 4, the O/C signal intensity was 0.36 and 2.05 before and after the consecutive high-voltage measurements, respectively. It was indicated that the oxygenated phenomenon increased considerably after the consecutive high-voltage measurements. Thus, successive current-voltage measurements were required for the breakdown to reach a steady state.

From our previous report, it was found that the oxygen signal for bottom surface of the isolated diamond film was stronger than the top surface of the isolated diamond film [15]. Moreover, as it is well known, there is a transition zone of a few hundreds of angstrom in the Si-diamond interface. Some impurities and defects cause a large number of electron and hole traps as well as interface states in this transition zone. Carriers trapping and relaxation were influenced by the non-diamond components and the oxidation layer existed on the bottom surface of the isolated diamond film. The 140 V decrease on the breakdown voltage for the bottom Schottky diode was larger than the 37 V for the top Schottky diode after the consecutive sixth repeated measurements. This phenomenon was attributed to the thicker oxidation layer and larger amount of the non-diamond components for the bottom surface of the isolated diamond film.

In this study, the successive current–voltage measurements of the Al/diamond/Al structure were first studied. The correlation between the surface property, the physical characteristics of the bulk diamond and the electrical conduction mechanism of the metal/diamond/metal has not been widely investigated. More detailed characterization and specific research in this area need to be carefully studied in the future research.

4. Conclusions

It was found that the *I*–*V* characteristic of the Al/diamond/ Al structure exhibited two Schottky diodes in a back-to-back configuration. Since the top diamond surface possessed better diamond quality than the bottom surface, the top Schottky diode with a breakdown voltage of 897 V and a lower breakdown voltage of -515 V for the bottom Schottky diode was observed for the first *I–V* measurement. However, the breakdown voltage decreased by 37 and 140 V for the top and bottom Schottky diodes after the consecutive sixth repeated measurements. It was found that the oxygenated phenomenon was more prominent; in addition, the quality of the isolated diamond film was also degraded after the consecutive high-voltage measurements. It was indicated that the 140 V decrease on the breakdown voltage for the bottom Schottky diode was attributed to the thicker oxidation layer and larger amount of the non-diamond components which existed on the bottom surface of the isolated diamond film.

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